RINOPOLYCRETE - TOWARDS A CEMENT-FREE AND FULLY RECYCLED CONCRETE

PROJECTO FCT

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Recycled inorganic polymer concrete - Towards a cement-free and fully recycled concrete

(RInoPolyCrete)

Task 5 - Report 1

Production and test methods of paving blocks for the first stage of Task 5

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ACRONYMS

CEM	Portland cement
FA	Fly ash
MIBA	Municipal solid waste incinerator bottom ash (Portuguese source)
MIBA F	MIBA from the Finnish source
EAFS	Electric arc furnace slag
AAM	Alkali-activated materials
NaOH	Sodium hydroxide
SiO_2	Silicon oxide
Na_2O	Sodium oxide
Na_2SiO_3	Sodium silicate

1 Introduction

This report contains a description of the experimental campaign for the preparation of alkali activated paving blocks, from the stage of establishing the principles to produce the blocks, at a significant scale, to their characterization in the hardened state. This experimental campaign will first comprise a preliminary testing stage, in which several variables will be explored to obtain paving blocks with the highest possible compressive strength. In the first stage, the water/binder ratio for the highest density is to be determined; in the second stage, factors such as the number of layers to fill a mould, the manual process of compaction of these layers and the mechanical process of compaction of the blocks are to be explored. Once these variables are defined, the mixes will be properly formulated, produced and moulded as paving blocks within an extensive testing campaign on the hardened state. Finally, this document also contains a characterization of each of the tasks to be carried out, with the corresponding timeline, as well as the determination of quantities of each of the required materials to be used.

2 Materials

2.1 Cement

CEM I 42.5 R, according to EN 197-1 [1], will be used as reference binder.

2.2 Alkali-activated binders

Alkali activated fly ash (FA) will also be considered as a reference binder given the large amount of experience with its use in the literature. Additionally, three other different precursors will be alkali activated and used as binders for producing the paving blocks.

2.2.1 Fly ash

The FA was sourced from EDP - Gestão da Produção de Energia, S.A. at the Sines Power Plant and considered as the reference alkali-activated binder.

2.2.2 Municipal solid waste incinerator bottom ash

Municipal solid waste incinerator bottom ash (MIBA) from two sources will be evaluated. One of them is the MIBA already characterized in previous reports (in Task 2 and 3), whereas the second comes from a waste-to-energy power plant in Finland and still to be fully characterized.

2.2.3 Electric arc furnace slag

The electric arc furnace slag (EAFS) to be used in this campaign corresponds to the one previously used in Task 3.3. It is a by-product of steel manufacturing and was collected from the Siderurgia Nacional de Portugal, provided by HARSCO.

2.3 Alkaline activator

A commercial solution of sodium silicate will be used in the experimental campaign, which has a sodium oxide (Na_2O) content between 12.7-13.3% and a silicon oxide (SiO_2) content between 26.2-26.8%; water content 59.9-61.1% and relative density of 1.296-1.396 g/ml. Reactive grade sodium hydroxide pellets will also be used, with 98% purity and a density of 2.13 g/ml.

2.4 Aggregates

Two types of siliceous sand will be used as fine aggregate; 0/4 coarse sand and 0/1 fine sand. Additionally, a sand-gravel 0 (2-5.6mm) of calcareous nature will be used. The bulk density, water absorption and particle size distribution of these aggregates was already determined and presented in previous reports.

3 Preparation of the paving blocks

3.1 Preliminary experimental work - study variables

The variables to be studied were selected based on a previous review of the relevant litera-

ture. These correspond to those that have the greatest influence on the manufacture process and, consequently, on the resulting mechanical properties of the paving blocks. To explore these variables, two preliminary experimental stages of testing are included in the work plan.

3.1.1 First stage of the preliminary work

In this stage, the optimum mixing water content (water/binder ratio) for the highest density will be determined based on the Proctor compaction test, frequently used in soil-mechanics. This test will be performed only for the reference binders (cement and activated FA). For that, a range of water/binder ratios (0.20-0.40) will be added to the binders and aggregates and mixed in a mechanical paddle mixer. These mixes will then be subjected to the Proctor test (described in section 3.4.1), the results of which will allow plotting a graph of water/binder ratio vs. dry bulk density, so that the optimum water content can be selected based on the maximum dry density of the compacted material.

3.1.2 Second stage of the preliminary work

Once the optimum mixing water content has been established, in the second stage, the optimum compaction method of the paving blocks will have to be defined based on trial tests, so that homogeneous blocks can be manufactured. The method also has to be reproducible to guarantee the uniformity of the blocks. Three different variables will be considered: the number of layers to fill the mould; the manual procedure of compaction of each layer; and the procedure for the overall mechanical compaction of the block once filled, using a hydraulic press. The following tentative experimental programme will be carried out:

1) Number of layers: i) one single un-compacted layer to the edge of the mould assuming that a 20% compaction with the hydraulic press may be enough (i.e. from 100 mm to 80 mm); ii) one layer manually compacted (about half the height of the block) and one layer not compacted (between half the height of the block and the edge of the mould); iii) two layers manually compacted (about half the height of the block and between half the height of the block and the upper edge of the mould), followed by another (last and less thick) layer up to the edge of the mould not manually compacted.

2) Manual compaction: the number of strokes in each layer and its distribution across the surface will be evaluated in order to develop a procedure that is easily reproducible.

3) Mechanical compaction: for each of the previously manually compaction procedures, the blocks will be placed in a press and the possibility to achieve the desired mechanical compaction load (or stress/pressure) will be evaluated. For this, a strain gauge will be placed vertically in the wall of the mould and both strain and force (or stress) will be monitored. If only the material is being compacted, the load shall increase but the strain will either present positive (due to bending of the mould walls) or nearly null strain values. However, after compaction is completed, i.e. when the upper plate of the mould becomes in contact with the remaining mould, the mould will become compressed and strain will increase (in modulus) and present negative values (due to compression).

3.2 Formulation of the mixes

The contents of each of the constituents for the production of the blocks are calculated based on a weight ratio of 1:5 (binder:aggregates). The volumetric ratio of SiO_2/Na_2O is defined as 1.0 and the NaOH/precursor ratio as 10. The water/binder ratio will be determined as stated in 3.1.1. This ratio, determined for the mix with the reference alkali-activated binder (FA), will be used for the mixes with the other precursors. Table 1 presents the mixes formulation and the estimated quantities[1] for the preliminary and final stages, considering five mixes and 45 l of each mix (approximately 5 l for the preliminary testing stages and 40 l for the final ones). For the calculation of these quantities, no additional water was considered to compensate for the eventual absorption by the aggregates, as it is negligible.

3.3 Production process of the paving blocks

3.3.1 Mixes production

In the preliminary stage, small batches of up to 2 l will be made in a mortar mixer, following

[1] Since the final water/binder ratios to be used in the mixes are to be determined in the preliminary stage, these formulations and respective quantities are tentative.

the procedure of EN 196-1 [2], with exception of the amounts established in the standard and the mixing times. In the final stage, a batch of 40 l will be produced for each family (with each of the binders presented in section 2.1), so that at least 16 blocks can be moulded.

Table 1 - Formulation of the mixes and estimated total quantities of materials

Materials	Quantities (kg/m^3)					Estimated total (kg)
	Mix I	Mix II	Mix III	Mix IV	Mix V	
Cement	350					15.8
FA		350				15.8
MIBA			350			15.8
MIBA F				350		15.8
EAFS					350	15.8
Water	134	50	50	50	50	18.9
Fine sand 0/1	384	384	384	384	384	83.1
Coarse sand 0/4	831	831	831	831	831	184.4
Sand-Gravel 2/5.6	707	707	707	707	707	156.4
NaOH		31	31	31	31	5.6
Na_2SiO_3 solution		133	133	133	133	24.0

When using the mortar mixer, the mixes will be made as follows:

- Equipment:
  - Scale with an accuracy of 0.1 g;
  - Graduated vessels;
  - Trowel;
  - Mixer;
  - Metal tray.
- Procedure:
  - Weigh all the constituents of the mix;
  - Add the alkaline solution to the mixer bowl and then the precursor;
  - Turn on the mixer to mix at low speed and, after 30 seconds, gradually add the aggregates so that it takes 30 seconds. Mix all the constituents for 30 seconds in high speed;
  - Stop the mixer for 1 minute and 30 seconds, and scrape off the material remaining in the walls of the bowl with a plastic scraper;

- Resume the mixing process at high speed for 1 minute;
- Stop the mixer for another 30 seconds and scrape the material from the walls of the bowl;
- Continue the mixing process at high speed for 1 minute, and stop the mixer;
- After mixing, place the mix in a metal tray and clean the mixer thoroughly.

When using the concrete mixer, the mixes will be made as follows:

➢ Equipment:
  - Scale with an accuracy of 0.1 g;
  - Large vessels (plastic buckets);
  - Scoop trowels;
  - Concrete mixer;
  - Wheelbarrow.

➢ Procedure:
  - Weigh all the constituents of the mix;
  - Wet the mixer (inside and outside) with water and then drain the interior properly;
  - Start the mixer;
  - With the mixer rotating, add the alkaline solution to the mixer, then the water and the precursor;
  - After 1 minute, gradually incorporate the aggregates as rotation continues, from the largest to the smallest particle size (small gravel, coarse sand and fine sand);
  - Let it mix for approximately 3 minutes (tilt the mixer when necessary, so that all materials are properly mixed);
  - Stop the mixer and empty it into the wheelbarrow;
  - Clean the mixer thoroughly.

3.3.2 **Block moulding and demoulding process**

The block moulding process will be defined based on the information gathered in the literature review and on several trial mixes, in which different variables will be considered, as previously mentioned in section 3.1.2.

➢ Equipment:

- 200 × 100 × 80 mm prismatic moulds;
- Plastic rod;
- Trowel;
- Metal sheet;
- Wood extruder;
- Hydraulic press.

➢ Procedure:

- Coat the internal faces of the mould with a thin layer of mineral oil;
- Place the mould on top of a metal sheet;
- Using a trowel, fill the mould with a layer of material, corresponding to approximately half the volume of the specimen;
- Manually compact the first layer with 20 strokes using a plastic rod;
- Add a second layer of material up to the edge of the mould and manually compact with another 20 strokes;
- Carefully place the metal lid of the mould on the top surface;
- Place the set on the inferior plate of the hydraulic press and apply compression to the mould.

Block demoulding is made by extrusion after mechanical compaction, with the help of a wood extruder, making sure that the extruder and the mould are in-line. To that end, firmly push the block through the mould, by applying a force in the wood extruder perpendicular to the surface of the block. At least 16 blocks of each family will be produced and tested.

3.3.3 **Curing conditions**

Once the moulding process of the paving blocks is finished, the blocks are immediately extruded and subjected to thermal curing in an oven for a period of 24 hours at 70 ºC. After this stage, 8 blocks of each family will be subjected only to dry curing, being placed in a dry chamber at 20 ± 5 ºC and 60 ± 10 %RH, for 28 days. The other 8 blocks will be subjected to dry curing, under the same conditions as stated above, for 21 days, followed by 7 days in a carbonation chamber at 5% CO_2, 20 ± 5 ºC and 60 ± 10 %RH.

3.3.4 **Test methods**

Following the curing process, the specimens will be subjected to the tests defined in the standard that regulates paving blocks (EN 1338 [3]).

Table 2 - Test methods and number of specimens for each family of blocks and for each type of curing conditions

Test	Standard	Number of specimens
Visual aspects and shape and dimension	EN 1338 [3]	8
Slip/skid resistance		3
Tensile splitting strength		3
Abrasion resistance		3
Total water absorption		2
Freeze/thaw resistance		2
Thermal conductivity	-	3

These tests will be performed for each family of blocks and for each type of curing conditions in accordance with the organogram presented in Figure 1.

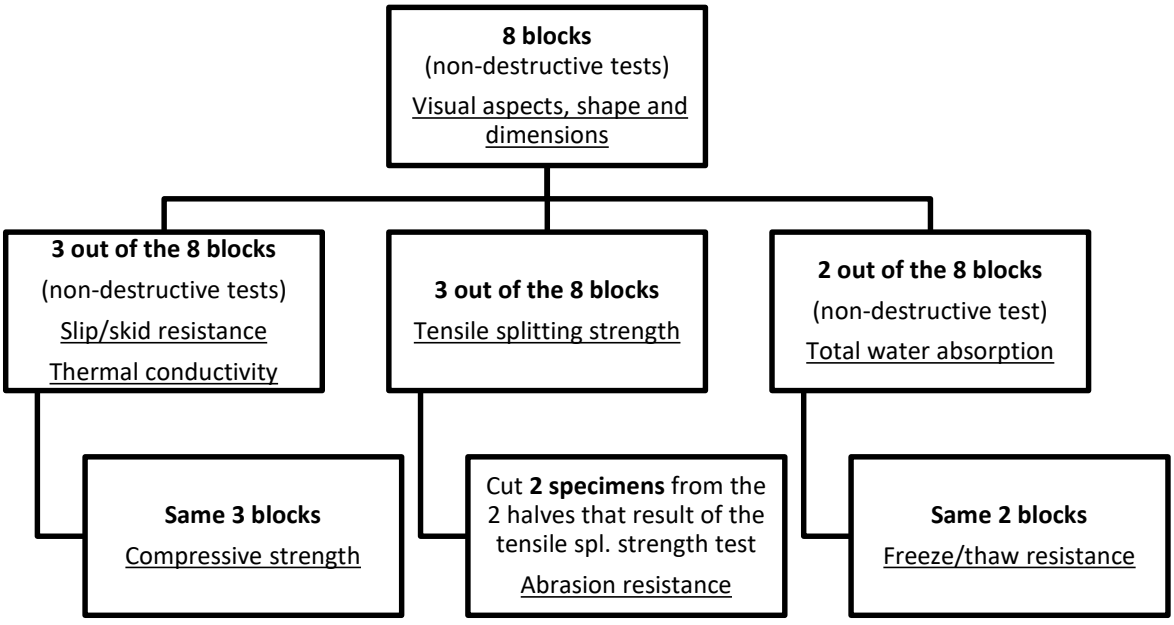


Figure 1 - Organogram with the testing sequence for each family of paving blocks and for each of the curing conditions

3.4 Preliminary tests

3.4.1 Proctor test

The Proctor compaction test will be carried out based on LNEC E 197 [4]. The test procedure will be properly adapted for the studied mixes, as the original test was developed for compacting soil. It is conducted for at least 4 different water contents (water/binder ratios) to obtain the optimum water content (w_{opt}), for which the value of the dry density is maximum, as described in 3.1.1. The test consists of introducing a mix in a cylindrical metal mould with a given volume in three layers and compacting each layer with a predefined number of blows using a drop hammer. The dry density of the compacted material is then obtained.

- Equipment:
  - Proctor compaction mould (small);
  - Proctor drop hammer;
  - Graduated vessels;
  - Mixer;
  - Trowel;
  - Palette knife.
- Procedure:
  - Weigh the mould without the collar (W_m);
  - Place the binder and aggregates in the mixer and gradually add water to reach one of the defined water/binder ratios (w);
  - Once the mixing process ends, remove the mix form the mixer bowl. With the mould on the floor, fill it in 3 layers. Compact each layer with 25 blows. The drops should be applied at a steady rate. The material should fill the mould and project above the collar by not more than 1 cm;
  - Carefully remove the collar and the material that extends above the mould with a palette knife;
  - Weigh the filled mould (W);
  - Extrude the compacted mix from the mould using a metallic extruder, making sure that the extruder and the mould are in-line;

- If possible, place the mix again in the mixer and add water to achieve higher water content and repeat the test.

➢ Calculation:

$$\gamma_d = \frac{W - W_m}{(1 + w) \times V}$$

- γ_d = dry density of the material (kg);
- W = weight of the mould filled with the mix (kg);
- W_m = weight of the mould (kg);
- w = water content of the material (%);
- V = volume of the mould (0.956 l).

Plot a graph with the water contents on the x-axis and the densities on the y-axis. The optimum water content is the one that corresponds to the highest dry density, as presented in Figure 2.

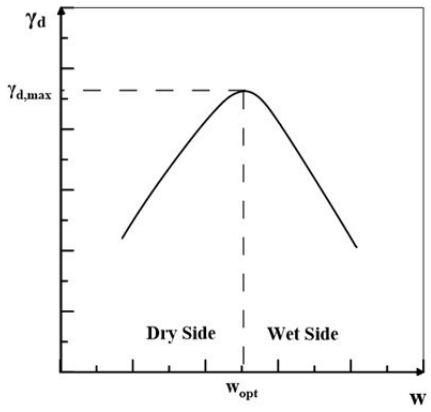


Figure 2 - Typical curve plotted from Proctor test results

3.5 Tests in the hardened state

3.5.1 Visual aspect, shape and dimensions

The visual aspect, shape and dimensions of the blocks will be inspected according to standard EN 1338 [3]. This test comprises the careful visual inspection of all blocks to verify their

homogeneity, uniformity in terms of texture and dimensions, the flatness of their faces and presence of flaws.

- Equipment:
  - Digital calliper;
  - Scale with precision of 0.01 g;
  - Digital camera.
- Procedure:
  - After curing, expose all specimens to natural daylight and inspect them in order to detect cracks, flaking or other flaws;
  - Verify the uniformity (if all blocks appear similar and have close properties in terms of texture and measurements) and homogeneity of the specimens. To that end, carefully weigh and measure each block in all three dimensions (in two different places for each dimension);
  - Observe the shape of their faces to verify if they are flat or curved;
  - Register this information together with a photographic record of the visual aspect of each of the specimens.

3.5.2 **Thermal conductivity**

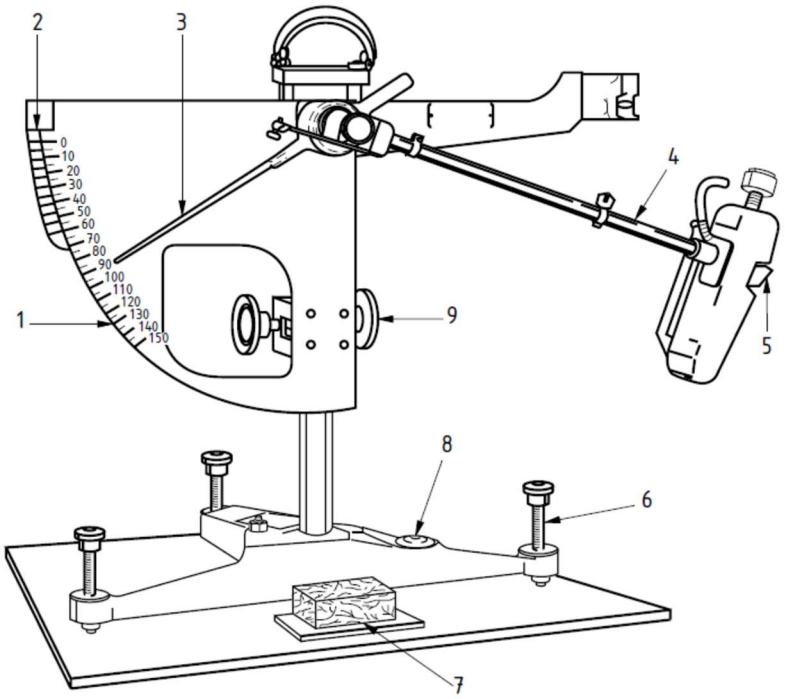
The determination of the thermal conductivity of the specimens will be made using the ISOMET 2114 device from Applied Precision.

3.5.3 **Slip/skid resistance**

The measurement of the unpolished slip resistance value (USRV) of the specimen will be made using a pendulum friction test equipment to evaluate the frictional properties of the specimen on the upper face, in accordance with EN 1338 [3]. In this test, the pendulum friction test equipment incorporates a spring loaded slider made of a standard rubber attached to the end of the pendulum. On swinging the pendulum the frictional force between the slider and test surface is measured by the reduction in length of the swing using a calibrated scale.

- Equipment:
  - Pendulum friction tester (like the one in Figure 3).
- Procedure:

- Immediately prior to testing, immerse the specimens in water at 20 ± 5 °C for at least 30 min;
- Adjust the friction in the pointer mechanism is so that when the pendulum arm and pointer are released from the right-hand horizontal position the pointer comes to rest at the zero position on the test scale;
- Place the test specimen is with its longer dimension lying in the track of the pendulum, and centrally with respect to the rubber slider and to the axis of the suspension of the pendulum. The track of the slider has to be parallel to the long axis of the specimen across the sliding distance;
- Adjust the height of the pendulum arm is so that in traversing the specimen the rubber slider is in contact with it over the whole width of the slider and over the specified swept length;



Key
1 C scale (126 mm sliding length)
2 F scale (76 mm sliding length)
3 Pointer
4 Pendulum
5 Rubber slider
6 Levelling screw
7 Test specimen holder
8 Spirit level
9 Vertical adjustment screw

Figure 3 - Pendulum friction tester [3]

- For testing, wet the surfaces of the specimen and the rubber slider with a generous amount of water, being careful not to disturb the slider from its set position. Then,

release the pendulum and pointer from the horizontal position and catch the pendulum arm on its return swing. Record the position of the pointer on the scale (the pendulum test value);

- Perform the latter operation five times, rewetting the specimen each time;
- Relocate the specimen after rotating through 180° and repeat the procedure.

➢ Calculation:

- The USRV is the mean of the pendulum test values obtained.

3.5.4 **Tensile splitting strength**

The determination of the tensile splitting strength will be carried out according to EN 1338 [3]. In this test, a compressive load is applied along two strips in contact with the longest length of the block (one placed at the bottom and another at the top of the block) at a continuous rate until failure occurs and the block splits in half.

➢ Equipment:

- Hydraulic press;
- Two packing pieces (15 mm wide 4-mm thick and at least 21-cm long), such as two strips of wood or GFRP.

➢ Procedure:

- Prior to the test, immerse the blocks in water at 20 ± 5 °C for 24 h, remove, wipe dry and test immediately;
- For testing, place the specimen is in the testing machine with the packing pieces on its upper face and bed face in contact with the bearers, ensuring that the packing pieces and the axes of the bearers are in line with the splitting section of the block (along the longest splitting section of the block - 20 cm, Figure 4);
- Apply the load smoothly and progressively at a rate which corresponds to an increase in stress of 0.05 ± 0.01 MPa/s. Record the failure load;

➢ Calculation:

$$S = l \times t$$

- S = area of the failure plane of the block (mm^2);
- l = mean of two measurements of the failure length, one at the top and one at the bottom of the block (mm);

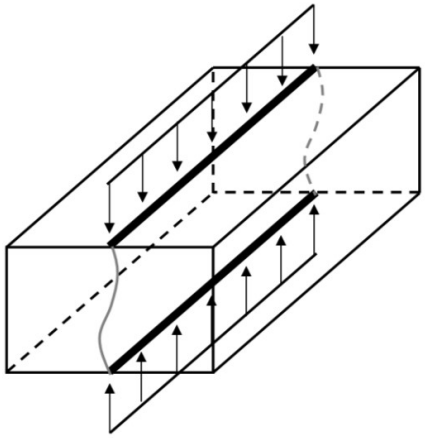


Figure 4 - Splitting tensile strength setup

- t = the thickness of the block at the failure plane and is the mean of three measurements: one in the middle and one at either end (mm).

$$T = 0.637 \times k \times \frac{P}{S}$$

- f_i = strength (MPa);
- k = correction factor for the block thickness, that is 1.3 for the tested blocks;
- P = failure load (N);
- S = area of the failure (mm^2).

3.5.5 **Abrasion resistance (Böhme test)**

The determination of the abrasion resistance of the blocks will be determined by the Böhme test, in accordance with standard EN 1338 [3]. In this test, square sheet samples taken from the blocks are placed on the Böhme disc abrader, on the test track of which standard abrasive is strewn, the disc being rotated and the specimens subjected to an abrasive load for a given number of cycles.

- Equipment:
  - Diamond saw;
  - Böhme disc equipment;
  - Digital calliper;
  - Scale with precision of 0.01 g.

- Materials:
  - Abrasive material (fused alumina/artificial corundum).
- Procedure:
  - Prior to the test, cut two square slabs from each block subjected to the splitting tensile strength, each with an edge length of 71 ± 1.5 mm;
  - Dry the specimens to constant mass at a temperature of 105 ± 5 °C;
  - Before the test, and after every four cycles, weigh the specimen;
  - Pour 20 g of standard abrasive on the test track. Clamp the specimen into the holder and, with the test contact face facing the track, load centrally with 294 ± 3 N;
  - Start the disc taking care that the abrasive on the track remains evenly distributed over an area defined by the width of the specimen;
  - Test the specimen for 16 cycles, each consisting of 22 revolutions;
  - After each cycle, clean both disc and contact face, and turn the specimen progressively through 90° and pour new abrasive on the track as described.
- Calculation:

$$\Delta V = \frac{\Delta m}{\rho_R}$$

  - ΔV = loss in volume after 16 cycles (mm^3);
  - Δm = loss in mass after 16 cycles (g);
  - ρ_R = density of the specimen (g/mm^3).

3.5.6 **Total water absorption**

The determination of the total water absorption will be carried out according to standard EN 1338 [3], by evaluating the mass loss, in percentage, after a specimen is soaked in water and then oven dried.

- Equipment:
  - Ventilated oven;
  - Flat based vessel to fully submerge several specimens;
  - Scale;
  - Cloth.

- Materials:
  - Water.
- Procedure:
  - Immerse the specimens in water in a flat based vessel, ensuring that a minimum of 20 mm water remains above them and that they are separated from each other by at least 15 mm. The minimum period of immersion is three days and/or until constant mass is reached (M_1);
  - Before weighing, wipe the specimens with a damp cloth;
  - Then, dry the blocks at a temperature of 105 ± 5 °C. For that, place each specimen inside the oven in such a way that the distance between each specimen is at least 15 mm. The minimum period of drying is be three days and/or until constant mass is reached;
  - Allow the specimens to cool to room temperature before they are weighed (M_2).
- Calculation:

$$W_a = \frac{M_1 - M_2}{M_2} \times 100\ \%$$

  - W_a = water absorption (%);
  - M_1 = initial mass of the specimen (g);
  - M_2 = final mass of the specimen (g);

3.5.7 **Freeze/thaw resistance test with de-icing salt**

The test for freeze/thaw resistance with de-icing salt, according to EN 1338 [3], will allow determining the amount of material that has scaled off a specimen after being subjected to 28 freeze/thaw cycles while its surface is covered with a 3% NaCl solution.

- Equipment:
  - Climatic chamber;
  - Rubber sheet (3 mm thick);
  - Thermal insulation (polystyrene 20 mm thick);
  - Polyethylene sheet;
  - Thermocouples;
  - Freezing chamber;

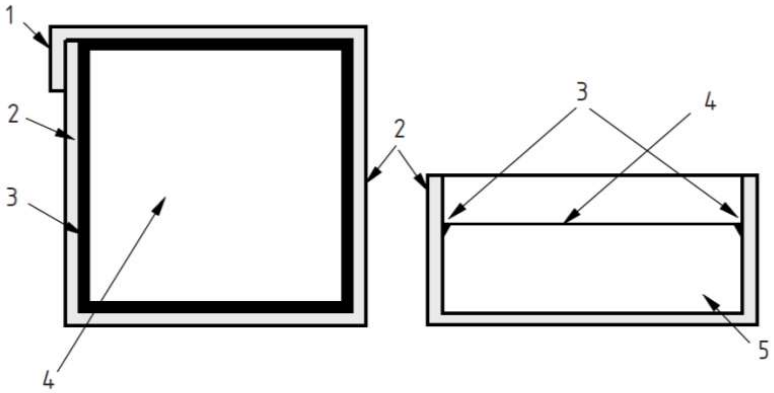
- Precision scale 0.01 g;
- Absorbent filter paper;
- Spray water bottle;
- Brush;
- Oven.

➢ Materials:

- Water;
- Adhesive (to glue rubber sheet to specimen);
- Silicon rubber (to provide a seal between rubber sheet and specimen);
- Sodium chloride (NaCl).

➢ Procedure:

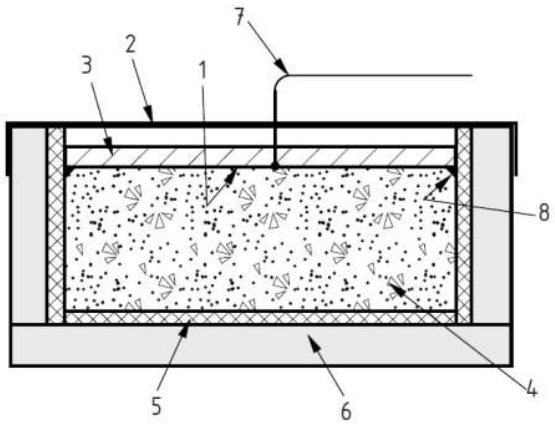
- Prior to the test, and after curing, leave the specimens for 168 h (7 days) in a climatic chamber at 20 ± 5 °C and 65 ± 10% RH. Then, insulate all faces of the specimen, except the test surface (top surface of the block), with a sealer and a rubber sheet, as depicted in Figure 5.
- Measure the tested area by making three measurements of its length and width to the nearest mm;
- To start the test, pour water on the test surface to a depth of 5 ± 2 mm, measured from the top surface of the specimen, and maintain for 72 h at 20 ± 5 °C;
- Prior to the freeze/thaw test, thermally insulate all faces of the specimen except the test surface with polystyrene (Figure 6). 5 min to 30 min before placing the specimens in the freezing chamber, replace the water layer with a 5 ± 2 mm layer of 3% NaCl in water. Cover the set with a horizontal polyethylene sheet to avoid water evaporation;



Key

1 Overlap	4 Test surface
2 Rubber sheet	5 Specimen
3 Sealant string	

Figure 5 - Schematic illustration of the position of the rubber sheet and sealant around the specimen (left: top view; right: cross-section view) [3]



Key

1 Test surface	5 Rubber sheet
2 Polyethylene sheet	6 Thermal insulation
3 Freezing medium (salted water)	7 Temperature measuring device
4 Specimen	8 Sealant string

Figure 6 - Schematic illustration of the position of the thermal insulation and of the thermocouple [3]

- Place the specimens in the freezing chamber horizontally and subject them to repeated freezing and thawing. The cycles are defined in the freezing chamber and

a thermocouple at the centre of one of the specimens (Figure 6) can be used to guarantee the temperature conditions of Figure 7.

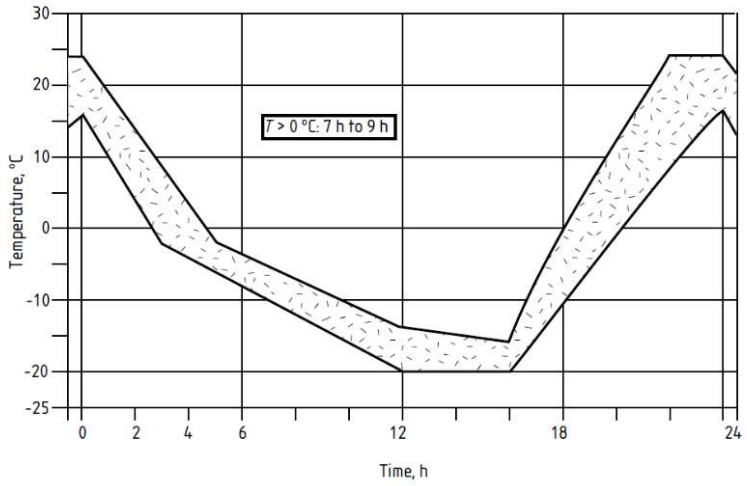


Figure 7 - Time-temperature cycle (centre of the surface of the specimens has to fall within the shaded area of the graph during the test cycle) [3]

- Add NaCl solution, if necessary, after 7 cycles and 14 cycles, during the thaw period, to keep a 5 ± 2 mm layer on top of the surface of the samples;
- After 28 cycles, for each specimen:

  a) Collect material, which has been scaled from the test surface by rinsing into the vessel using the spray bottle and brushing into the vessel until no further scaled material is removed;

  b) Carefully pour the liquid and scaled material through a filter paper. Then wash the material collected in the filter paper with a minimum of 1 L of water to remove any remaining NaCl. Dry the filter paper and collected material for at least 24 h at 105 ± 5 °C. Determine the dry mass of the scaled material, not considering the mass of the filter paper.

➢ Calculation:

$$L = \frac{M}{A}$$

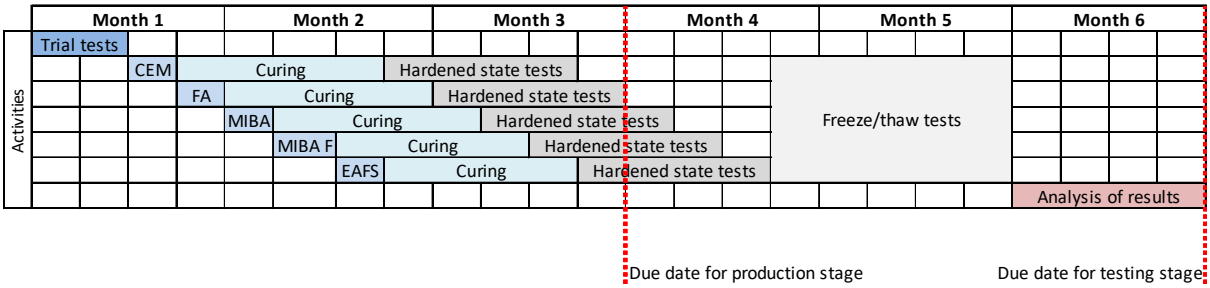
- L = mass loss per unit area of the specimen (kg/m^2);
- M = mass of the total quantity of material scaled after 28 cycles (kg);

- A = area of the test surface (m^2).

4 Timeline

Table 3 presents the schedule to follow for the development of each of the experimental stages.

Table 3 - Timeline



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