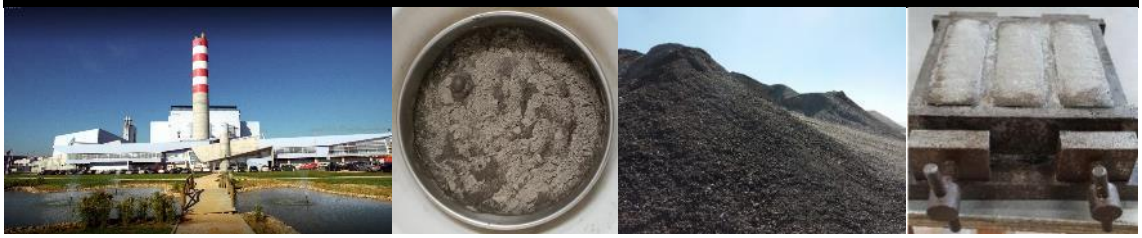
RINOPOLYCRETE - TOWARDS A CEMENT-FREE AND FULLY RECYCLED CONCRETE



PROJECTO FCT

PTDC/ECI-COM/29196/2017

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

(RInoPolyCrete)

Task 2 - Report 3

Test methods for the second stage of Task 2: Optimization of the alkali activator

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ACRONYMS

MIBA	Municipal solid waste incinerator bottom ash
AAM	Alkali-activated materials
NaOH	Sodium hydroxide
SiO_2	Silicon Oxide
Na_2O	Sodium Oxide
FA	Fly ash
WRA	Water reducing admixture
Na_2SiO_3	Sodium silicate

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1 Introduction

In this report, the experimental campaign to be carried out for the optimization of the preparation of alkali activated mortars is discriminated, from the stage of obtaining and preparing the precursor municipal incinerator bottom ash (MIBA), to fresh state and hardened tests made to the material obtained.

This experimental campaign will assess four experimental designs of one factor, in which the optimum value of the variable under study will be obtained taking into consideration the highest value of compressive strength. In the first design, the factor corresponds to the concentration of sodium hydroxide (NaOH); in the second design, the factor is the ratio silicon oxide/sodium oxide (SiO_2/Na_2O); in the third one, the curing temperature; and, the last experimental design studies curing time. Other response variables correspond to a series of properties in the fresh and hardened state that will be assessed.

The control mortars of this experimental campaign correspond to alkali activated mortars, the precursor of which is fly ash (FA), produced by Energias de Portugal, EDP - Energy production management, S.A. - at Sines Power Plant. This campaign runs parallel to the experimental campaign of MIBA.

Finally, in this document, each of the tasks to be carried out are duly characterized, with the corresponding start and end dates, as well as the determination of quantities to be used of each of the required materials.

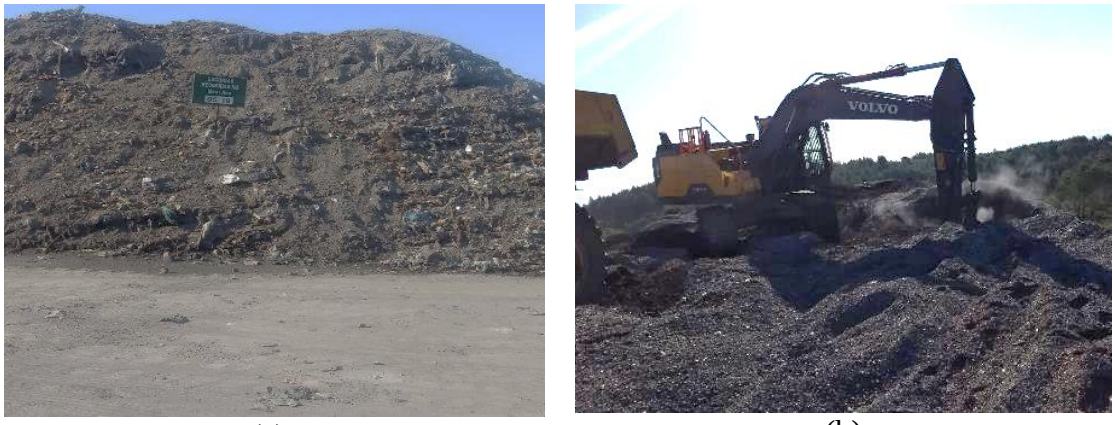
2 Materials

2.1 Municipal incinerator bottom ash

2.1.1 Collection and sampling

MIBA collection was carried out in the Valorsul facility. The collected sample for this experimental campaign corresponds to the residue generated for the month of January 2019. In addition, between 8 and 10 kilograms of MIBA from September, October and December, 2018, were also collected and will be used in an upcoming experimental campaign assessing the variability of MIBA it terms of its composition and influence on AAM's properties. A stationary

sampling of the pile was done with the use of a long-arm excavator, which made six perforations at different depths, distributed evenly throughout the slope (Figure 1).



(a) (b)

Figure 1 - MIBA stacks separated by generation month (a); MIBA sampling (b)

The material is transported using a tipper and deposited onto a flat surface in order to homogenize the MIBA, stirring it repeatedly (Figure 2). Finally, a representative sample was obtained from the stockpile, which was then transported to the laboratory in big bags.

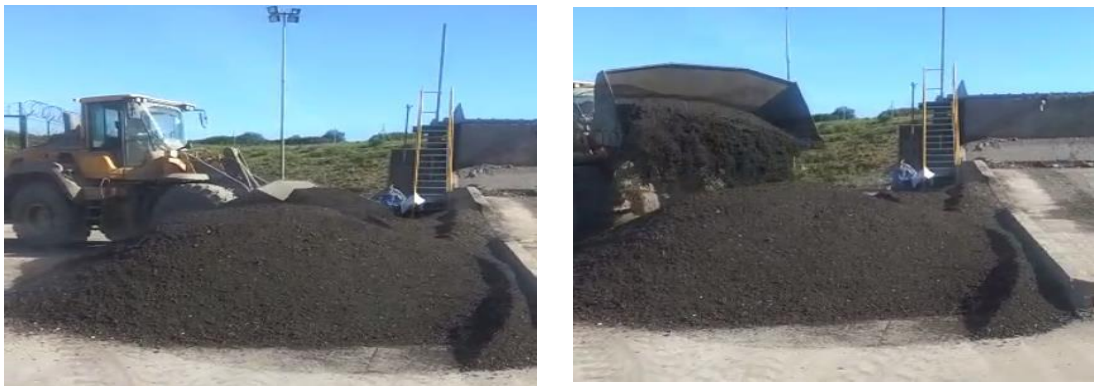


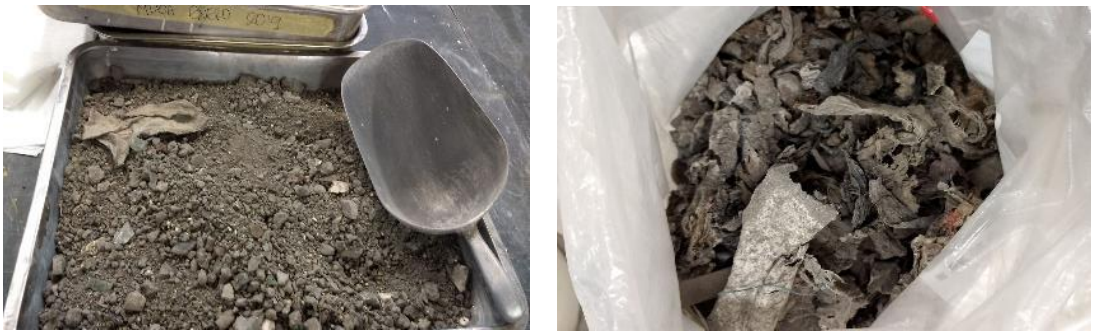
Figure 2 - Homogenization of MIBA

2.1.2 **Preparation and grinding**

MIBA preparation for grinding includes the following steps:

- Drying the material at 105 °C until constant mass is reached. The mass is considered constant when, in two consecutive weightings, one hour apart, it does not vary by more than 0.1% (Figure 3a).

- Manual cleaning of MIBA, in order to remove of plastic, wooden or metallic particles (Figure 3b).



(a) (b)

Figure 3 - Dry MIBA (a) and contaminants removed by manual cleaning (b)

The grinding stage is divided in three stages:

a. Grinding in the Los Angeles machine:

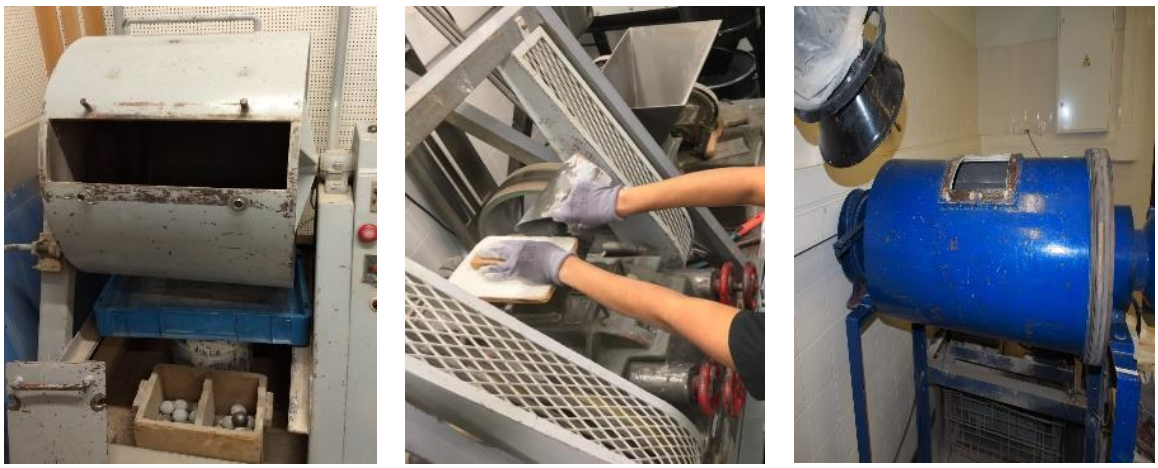
20 kilograms of material are initially ground in the Los Angeles machine, using an abrasive load of 12.30 kg of steel, equivalent to 31 steel balls for a time of 30 minutes (Figure 4a). The resulting MIBA is screened by using a 4 mm sieve, in order to separate the larger particles for later reduction in size.

b. Crushing in the cylinder mill:

A cylinder mill is used to reduce the size of particles larger than 4 mm, to be incorporated into the material previously pulverized in the Los Angeles machine (Figure 4b).

c. Crushing in the ball mill:

About 20 kilograms of MIBA are milled in a ball mill using an abrasive load of 56 kilograms of steel balls with diameters varying between ½ inch and 2 inches (1.27 cm and 5.08 cm, respectively), for a period of 2 hours, in order to produce a powdery product presenting an average particle size similar to that of cement (Figure 4c). Figure 5 shows MIBA at different grinding stages.



(a) (b) (c)

Figure 4 - Los Angeles machine (a); cylinder mill (b); ball mill (c)

(a) (b) (c)

Figure 5 - MIBA at different stages of grinding (a) MIBA upon entering the Los Angeles machine; (b) MIBA at the exit of the Los Angeles machine; MIBA at the exit of the ball mill machine (c)

2.2 Fine aggregate

Two types of siliceous sand are used as fine aggregate; 0/4 coarse sand and 0/1 fine sand. The bulk density, water absorption and particle size will be determined for each sand.

2.2.1 Particle size distribution

The particle size distribution will be carried out according to EN 933-1 [1]. The test consists of dividing and separating the material, sorting it by particle size in a decreasing way by means

of a series of sieves. The size of the opening in the sieves is selected in accordance with the nature of the sample and the required accuracy. There are two methods for the determination of the particle size distribution; the wet and dry methods. The former usually applies to very fine materials (e.g. ground MIBA), whereas the latter is used for aggregates.

- Equipment:
  - Sieves;
  - Ventilated oven with a temperature of 110 ± 5 °C;
  - Electric sieve machine;
  - Scale with an accuracy of ± 0.1% of the mass to be tested;
  - Trays.
- Procedure of the wet method:
  - Dry 300 grams of the aggregate at a temperature of (110 ± 5) °C to constant mass. Let cool, weigh and record as M1;
  - Place the portion to be tested in a container and add enough water to cover the portion of the test. Store for a period of 24 hours;
  - Shake the sample vigorously enough to result in complete separation and suspension of the fines;
  - Wet both sides of a sieve with a 0.063 mm opening diameter reserved for use in this test only and place a 2 mm protective sieve on top;
  - Pour the contents of the container into the top sieve. Continue washing until the water that passes the 0.063 mm test sieve is clean;
  - Dry the residue retained in the 0.063 mm sieve at (110 ± 5) ºC at constant mass. Let cool, weigh and record as M2.
- Procedure of the dry method:
  - Pour the dried material into the sieve column. The column comprises a series of sieves mounted and arranged, from top to bottom, in decreasing order of opening sizes with the bottom and the lid;
  - Shake mechanically the sieve arrangement. Remove the sieves one by one, beginning with the larger opening;
  - Weigh the material retained for the sieve with the largest opening size and record

its mass as R1, perform the same operation for the sieve immediately below and record the retained mass as R2, continue with the same operation for all sieves cease in the column, weigh the sieved material (Ri), and, if any, also the remainder in the pan and record its mass as P.

- Calculation:
  - Calculate the retained mass in each sieve as a percentage of the original dry mass M1;
  - Calculate the cumulative percentage of the original dry mass that passes each sieve through the 0.063 mm sieve;
  - Calculate the percentage of fines that pass the 0.063 mm sieve according to the following equation:

$$f = \frac{(M_1 - M_2) + P}{M_1} \times 100$$

Where:

M_1 - dry mass of the test portion, in kilograms;

M_2 - dry mass of the residue retained in the 0.063 mm sieve, in kilograms;

P - mass of the sieved material that remains at the bottom, in kilograms.

For dry sieving, the following formula will be used to calculate the amount of fines accumulated in the bottom sieve:

$$f = \frac{100P}{M_1}$$

2.2.2 **Bulk density and water absorption**

The density and water absorption will be carried out according to EN 1097-6 [2], wherein the density of the aggregate is calculated from the ratio of the mass of the particles and the volume occupied by them. The mass is determined by weighting the portion in the saturated and surface-dried state and again in the oven-dried state, while the volume is determined from the mass of water displaced by weighting using the pycnometer method. Water absorption is determined 24 hours after immersion of the aggregate by previously drying the surface.

- Equipment:

- Ventilated oven with a temperature of 110 ± 5 °C;
- Scale with a precision of 0.1% of the mass to be tested;
- Sieves;
- Thermometer;
- Trays;
- Pycnometer.

➢ Procedure:

- Insert the aggregate sample into the pycnometer in water at 22 ± 3 °C and remove the trapped air by gently shaking the pycnometer, then place the pycnometer in a water bath at 22 ± 3 °C and, 24 hours later, shake the pycnometer again to remove trapped air. Fill the pycnometer with water to the corresponding capacity, dry on the outside and determine the total mass of the pycnometer, the sample and the water;
- Remove the content in the pycnometer and dry the aggregate to constant mass at a temperature of 110 ± 5 °C;
- Fill the pycnometer with water at 22 ± 3 °C, cover and weigh again.

➢ Calculation:

$$\rho_a = \rho_w \times \frac{M_4}{M_4-(M_2-M_3)} [kg/m^3]$$

$$\rho_{rd} = \rho_w \times \frac{M_4}{M_1-(M_2-M_3)} [kg/m^3]$$

$$\rho_{ssd} = \rho_w \times \frac{M_1}{M_4-(M_2-M_3)} [kg/m^3]$$

Water absorption after immersion is calculated using the following formula:

$$WA_{24} = \frac{M_1-M_4}{M_4} \times 100 [\%]$$

Where:

ρ_a - apparent particle density (kg/dm^3);

ρ_{rd} - oven-dried particle density (kg/dm^3);

ρ_{ssd} - saturated and surface-dried particle density (kg/dm^3);

ρ_w - density of water at the test temperature (kg/dm^3);

WA_{24} - water absorption after immersion for 24 h (%);

M_1 - mass of the saturated and surface-dried aggregate (g);

M_2 - mass of the pycnometer containing the sample of saturated aggregate and water (g);

M_3 - mass of the pycnometer filled with water only (g);

M_4 - mass of the oven-dried test portion (g).

2.3 Alkaline activator

The alkaline activator corresponds to a solution that is prepared in the laboratory, which is made up of one or two solutes and a solvent. Once the solution is prepared, temperature is determined using a thermometer and the pH with the use of a digital pH meter. In addition to the solution prepared in the laboratory, a commercial solution of sodium silicate, will also be used in this experimental campaign, which has a sodium oxide (Na_2O) content between 12.7-13.3% and silicon oxide (SiO_2) between 26.2-26.8%; water content 61.1-59.9% and relative density of 1.296-1396 g/ml.

2.3.1 Solutes

The solutes used in the preparation of the alkaline activator correspond to sodium hydroxide and silica gel; both in solid state.

2.3.1.1 Sodium hydroxide

Reactive grade sodium hydroxide pellets, with 98% purity and a density of 2.13 g/ml.

2.3.1.2 Silica gel

Silica gel pellets, with a SiO_2 content of 98% and density of 2.19 g/ml.

2.3.2 Solvent

The water used in the preparation of the alkaline activator corresponds to that from the public network of EPAL company, which complies with the Directive 98/83/CE [3].

2.4 Water reducing admixture

SikaPlast-717 is a superplasticizer for commercial use, consisting of a combination of synthetic organic water-based dispersants, with a density of 1.21 ± 0.03 kg/dm^3 and a pH of 10 ± 1.

3 Preparation of the mortars

3.1 Study variables

The variables to be studied were selected according to the literature, corresponding to those that have the greatest influence on the mechanical properties of alkali activated materials (AAM). Table 1 shows the operationalization of the variables for each stage of the proposed experimental campaign. The response variable, by which the optimum level of each stage is determined, corresponds to the compressive strength. In this way, the best conditions are defined by means of a progressive approach based on the mechanical performance. When similar values are obtained in this mechanical property, a Post Hoc analysis is necessary.

Table 1 - Operationalization of the variables

Experimental campaign stages	Factor	Unit of measurement	Levels			
			1	2	3	4
1	NaOH Solution Concentration	Molarity (moles of sodium hydroxide / liter of solution)	4	6	8	10
2	SiO_2/Na_2O Relation	Dimensionless	0.5	0.5	1.0	1.5
3	Curing temperature	°C	50		80	
4	Curing time	Hour	24	48	72	

3.1.1 First experimental stage

In this stage, the optimum concentration of sodium hydroxide is determined based on the highest compressive strength. Table 2 shows the experimental design of a stage 1 factor.

Table 2 - Experimental design stage 1

NaOH (M)	Na_2O/SiO_2	Curing temperature	Curing time
4	-	80 °C	24 h
6			
8			
10			

3.1.2 Second experimental stage

Once the optimum concentration of sodium hydroxide has been established, in the second stage, the optimum ratio Na_2O/SiO_2 is determined, for which the highest compressive strength is obtained. Table 3 presents the experimental design of stage 2.

Table 3 - Experimental design stage 2

NaOH	Na_2O/SiO_2	Curing temperature	Curing time
Determined in stage 1	0.25	80°C	24 h
	0.5		
	1.0		
	1.5		

3.1.3 Third experimental stage

In the third stage, the optimum curing temperature is determined at which the highest compressive strength is obtained after establishing the optimum concentration of NaOH and Na_2O/SiO_2 ratio from stages 1 and 2. Table 4 presents the experimental design of stage 3.

Table 4 - Experimental design stage 3

NaOH	Na_2O/SiO_2	Curing temperature	Curing time
Determined in stage 1	**Determined in stage 2**	50 °C	24 h
		80 °C	

3.1.4 Fourth experimental stage

Once the optimal values of the NaOH concentration, the SiO_2/Na_2O ratio, and the curing temperature have been established, in stage 4, the optimum curing time will be established based on the materials' strength development. Table 5 presents the experimental design of stage 4.

Table 5 - Experimental design stage 4

NaOH	SiO_2/Na_2O	Curing temperature	Curing time
Determined in stage 1	**Determined in stage 2**	**Determined in stage 3**	24 h
			48 h
			72 h

3.2 Determination of contents

The contents of each of the constituents for the manufacture of AAM will be calculated based on volumetric ratios, using the densities of each of the raw materials. The binder/aggregate volumetric ratio (V_B/V_A = 0.33); the volumetric ratio of SiO_2/Na_2O (V_S/V_N = 0; 0.25; 0.5; 1.0; 1.5). The ratio water/binder (V_W/V_B) varies with the preparation of the alkaline activator, since one of the contains water. Two mass radios are made: water reducing admixture/precursor (WRA/B%: 2) and sodium hydroxide/precursor (NaOH/B%).

3.3 Procedure for the production and processing of mortars

The production and preparation of mortars is carried out in accordance with EN 196-1 [4], with the exception of the amounts established in the standard and the mixing time.

- Equipment:
  - Scale with an accuracy of 0.1 g;
  - Graduated volumetric material;
  - Mixer;
  - Bricklayer's trowel;
  - 40 x 40 x 160 mm prismatic moulds with shrinkage pins and no shrinkage pins;

- Compaction apparatus.

3.3.1 Mortar manufacturing

For the manufacture of mortars, the following procedure will be followed:

- Weigh all solid constituents of the mortar according to subchapter 0;
- Add to the NaOH solute to the solvent (pre-measured volume of water) and stir the solution with a glass stirrer;
- Add the alkaline solution in the mixer bowl and then the precursor;
- Alkali activated mortars, with MIBA as precursor, require a longer contact time with the alkaline solution, so that the hydroxyl ion (OH^-) can react with the metallic aluminium present in this type of waste, allowing the release of hydrogen gas while the mixture is in its fresh state. This process is carried out as follows:
  - Weigh an empty dry container;
  - Once the container has been tared, weigh the amount of MIBA according to sub-chapter 0 (determination of quantities);
  - Prepare the alkaline solution by dissolving the amount of NaOH solute in the water solvent according to subchapter 3.2 (determination of quantities), then quickly add this solution to the previously weighed MIBA;
  - Mix manually, adding half the amount of the superplasticizer and stir for 10 minutes, using a stirrer;
  - Cover the container with plastic wrap, making several holes at the top and let this mix sit for 24 hours;
  - After 24 hours, weigh again and compensate for weight loss with water;
  - Pour the mix of the alkaline solution and MIBA into the mixer bowl.
- Turn on the equipment at low speed, add the superplasticizer and, after 30 seconds, add the previously homogenized coarse and fine sand for 30 seconds, then mix for 30 seconds more in fast speed;
- Stop the mixer for 1 minute and 30 seconds, with a plastic paddle remove the mortar from the walls of the bowl;
- Continue mixing at fast speed for 1 minute;

- Stop the mixer for 30 seconds, with a plastic paddle remove the mortar from the walls of the bowl;
- Continue mixing at rapid speed for 1 minute, finally stop the mixer and with a plastic paddle remove the mortar from the walls of the bowl.

3.3.2 Mortar moulding

Mortar moulding is done using the following procedure:

- Place the mould firmly anchored to the shaking table;
- Using a trowel, add the first layer of the mortar in the mould, approximately half the volume of the specimen;
- Turn on the shaker table and compact the first layer with 60 strokes;
- Add the second layer of mortar in the mould and compact with 60 strokes;
- Carefully remove the mould and remove the excess mortar with a straightedge metal;
- Cover the mould with plastic wrap to avoid water loss.

3.3.3 Curing Conditions

Once the moulding of the mortars is finished, these remain for a period of 24 hours in ambient conditions and are subsequently stored in the oven at the temperature and time established in accordance with the experimental stage. At the end of this stage, the mortars are demoulded and placed in the curing conditions as shown in Table 6. The time the mortars must remain in the chamber is specified in each of the tests.

Table 6 - Curing conditions, test methods and number of specimens for each family of alkali activated materials

Test	Standard	Number of specimens	Curing conditions
Carbonation	EN 13295 [5]	6	28 days in dry chamber; place in carbonation chamber until testing age
Shrinkage	EN 1015-13 [6]	3	Specimens placed in dry chamber after demoulding until the end of the test.
Bulk density	EN 1015-10 [7]	3	Specimens placed in dry chamber until testing age
Ultrasound	EN 12504-4 [8]	9	
Elasticity module	EN 14146 [9]		
Flexural strength	EN 1015-11 [10]		
Compressive strength	EN 1015-11 [10]		
Water absorption by capillarity	EN 1015-18 [11]	3	
Water absorption by immersion	LNEC E-394 [12]	3	

3.4 Fresh state performance

3.4.1 Density

The determination of bulk density in the fresh state will be carried out according to EN 1015-6 [13]. The test consists of introducing and compacting the mortar in a container with a given volume and determine the ratio of mass and volume.

- Equipment:
  - 1 litre capacity vessel;
  - Scale;
  - Trowel;
  - Scoop;
  - Tamping rod;
- Procedure:
  - Weigh the empty vessel (m_{vessel});
  - Fill 1/3 of volume the vessel with mortar by means of the scoop press 10 times using the tamping rod; repeat this step twice until the entire container is filled;
  - Remove the excess with a trowel, weigh the full vessel and determine m_2;
- Calculation:

$$\rho = \frac{m_2 - m_{vessel}}{V_{vessel}}$$

The density of the mortar in the fresh state will then correspond to the difference in m_2 minus the mass of the empty vessel over the volume of the vessel.

3.4.2 Workability

The workability will be carried out according to EN 1015-3 [14]. The test consists of measuring the flow by means of the samples' diameter, which has been placed on a circular table through a defined mould and, given a number of vertical intact, allows the sample to fall freely the height initially given.

- Equipment:
  - Truncated conical mould;

- Mortar flow table (workability table);
- Tamper:
- Calliper;
- Bricklayer's trowel;
- Scoop;

➢ Procedure:

- Place the mould centred on the disc of the manual flow table and place the mortar in two layers. Each layer should be compacted 10 times with the tamper; make sure the mould filling is uniform;
- Remove excess mortar with the trowel, clean the area around the disk, after approximately 15 seconds. Remove the mould vertically and spread the mortar with the disc with 15 jolts at a constant frequency of approximately 1 per second;
- Measure the diameter of the mortar in two directions at right angles, using the calliper.

3.4.3 **Air content**

The determination of air content in fresh state will be carried out according to standard EN 1015-7 [15]. In this test, a volume of mortar is placed in a specific measuring vessel. Subsequently, water is added to the top of the mortar's surface and, by means of air applied under pressure or the use of a mixture of water + ethanol, it is forced into the mortar, displacing the air inside the pores. The water level drops and reflects the volume of water displaced from the mortar.

➢ Equipment:

- Sample container and cover assembly;
- Air content measuring;
- Tamper;
- Bricklayer's trowel;
- Palette knife;

➢ Procedure: air pressure method A.

- Calibrate the air content measurement equipment;
- Fill the bowl completely with mortar in four approximately equal layers, each layer being compacted by 10 short strokes with the tamper;

- Using the palette knife to skim off the excess mortar, leaving the mortar surface plane and level with the top edge of the bowl;
- Wipe the outside of the bowl clean and dry and clamp the cover securely in place on the bowl;
- Close the main air valve between the air chamber and the sample container;
- Fill the air space under the cover and above the mortar with water through valve A, keeping valve B open until all the air above the mortar surface is expelled;
- Adjust the air content indicator, to zero, by carefully opening the air valve;
- Close both valves A and B and open the valve between the air chamber and the sample container by 20 seconds;
- Measure the value of the air content.

The air content is made in two samples of mortar, the average of the two measurements being the value considered for the test.

3.5 Mortars testing in hardened state

3.5.1 Bulk density

The determination of dry bulk density of hardened mortar will be carried out according to standard: EN 1015-10 [7]. In this test, the apparent density in the hardened state of a given specimen is determined by the amount of mass in dry condition and the volume it takes.

- Equipment:
  - Scale with 0.1 g precision;
  - Oven at a temperature of 70 ± 5 °C or 60 ± 5 °C;
  - Calliper;
- Procedure:
  - Dry the specimen in an oven at a temperature of 105 ± 5 °C, until constant mass is reached. The mass is considered constant when in two consecutive measurements separated by two hours they do not differ by more than 0.2%.
- Calculation:

$$\rho = \frac{m_{s,dry}}{Vs}$$

- Vs = taken as the volume of the mould, which can be confirmed by measuring the dimensions of each test tube using a calliper.

3.5.2 **Dynamic modulus of elasticity**

The determination of the dynamic modulus of elasticity will be carried out according to standard EN 14146 [9]. This test is an indicator of the compaction of the mortar, it measures the frequency of the resonance of the prismatic specimens and from that value the dynamic modulus of elasticity is calculated.

- Equipment:
  - Frequency measuring device;
  - Computer with specific software;
- Procedure:
  - Enter the bulk density values in the software;
  - Place the specimen longitudinally in the frequency measuring device with one end connected to the emitting source and on the other end 1 mm from the receiver;
  - Turn on the variable frequency oscillator and observe the recorded graph;
  - The fundamental frequency is that which corresponds to the lowest frequency with which the maximum amplitude is obtained;
- Calculation:

$$E_{di} = \frac{4\ x\ L_i^2 x F_i^2 x \rho_i x 10^{-6}}{g}$$

  - E_{di} = dynamic elastic modulus of the test tube i (MPa);
  - L_i = length of the test piece i (mm);
  - F_i = longitudinal resonance frequency of test tube i (Hz;
  - ρ_i = specific weight of the specimen i (N/dm^3);
  - g = acceleration of gravity.

3.5.3 Ultrasound pulse velocity

The determination of ultrasound pulse velocity will be carried out according to standard EN 12504-4 [8]. This test measures the time in microseconds, which a given ultrasonic wave takes from the moment it is emitted until it is received after covering the distance between a sending unit and a receiving unit. In this test, it is possible to determine cracks or pores inside the samples, through abrupt variations in velocity. The velocity of the waves is much higher in solid continuous media than in gaseous media. The ultrasound pulse velocity test evaluates the compactness of a mortar as it can be related with the modulus of elasticity and mechanical strength.

- Equipment:
  - Scale;
  - Ultrasound device;
- Procedure:
  - The emitting transducer is placed at one end of the prismatic specimen and the transducer or receiver is placed at the opposite end;
  - Six readings are recorded (microseconds), corresponding to the time it takes for the wave to travel the specimen;
- Calculation:

$$v_i = \frac{d_i}{t_i}$$

  - v_i = propagation velocity in test tube i (mm/s);
  - d_i = distance between sender and receiver;
  - t_i = propagation time, average value for the 2 or 3 samples.

3.5.4 Shrinkage

The determination of shrinkage will be carried out according to standard EN 1015-13 [6]. This test measures the shrinkage of mortars, in a longitudinal direction over time.

- Equipment:
  - Shrinkage measuring equipment;
  - Scale with precision of 0.01 g;

- Procedure:
  - Once the specimens are demoulded, the first measurement of the specimens is carried out with the shrinkage measuring equipment, then once per day for 7 days, once every three days for 21 days, once per week until the end of the test.

3.5.5 Flexural strength

The determination of flexural strength of hardened mortar will be carried out according to the standard EN 1015-11 [10]. In this test, the flexural strength is determined by a three-point lead to a hardened mortar prism.

- Equipment:
  - Flexural and compression strength equipment;
- Procedure:
  - Place the specimen in the accessory for the determination of flexural strength;
  - Apply a force with a uniform load between 10 and 500 N/s, until failure between 30 and 90 seconds after the start of the test;
- Calculation:

$$fi = 1.5\frac{F_i}{b_i \; x \; d_i^2}$$

  - f_i = Flexural strength of test tube i (MPa);
  - F_i = Maximum force supported by test tube i (N);
  - b_i = length of the test piece i (mm);
  - d_i = specimen thickness i (mm).

3.5.6 Compressive strength

The determination of compressive strength will be carried out according to standard: EN 1015-11 [10]. In this test, the broken specimens from the previous test are placed in the accessory for the determination of the compressive strength, with both sides of the specimen with the contact surface and the load surface.

- Equipment:
  - Flexural and compression strength equipment;

- Procedure:
  - Apply a force with a uniform load between 50 and 500 N/s, until failure between 30 and 90 seconds after the start of the test;
- Calculation:

$$c_i = \frac{C_i}{A_i}$$

  - c_i = compressive strength of the test piece I;
  - C_i = Maximum force supported by test tube i (N);
  - A_i = section area compressed by the prism i (mm^2).

3.5.7 **Water absorption by capillarity**

The determination of water absorption by capillarity will be carried out according to standard: EN 1015-18 [11]. In this test, the water absorption coefficient is measured using prismatic mortar specimens, under the curing conditions specified to P_{atm}, after constant mass drying. One side of the specimen is immersed in 5 to 10 mm of water and the increase in mass is determined after specific periods of time.

- Equipment:
  - Tray with a minimum of 20 cm depth to submerge several specimens;
  - Scale;
  - Oven;
  - Bricklayer's trowel;
  - Absorbent filter paper;
  - Support sponge;
- Materials:
  - Water;
  - Sealing material such as paraffin or resin with a melting point higher than 60 °C;
- Procedure:
  - Dry to constant mass in an oven at 60 ± 5 °C;
  - Once the specimens have dried, the mortars are cut in half;
  - Place the mortar specimens on the trays with the aggregate-exposed face of 16 cm^2 submerged in water, at a depth of 5 mm to 10 mm for the duration of the test;

- Cover the trays to avoid evaporation and keep the water level constant;
- Activate the timer and after 10 minutes quickly remove the specimens one by one from the container;
- Remove excess water using absorbent paper, weigh and replace in container. Repeat the procedure at 30, 60, 90, 180, 300, 480 and 1400 minutes.

➢ Calculation:

$$C_i = \frac{M_{90i} - M_{10i}}{A_i \times (\sqrt{90} - \sqrt{10})}$$

- M_{10i} = mass of the specimen after moistening it for 10 minutes (g);
- M_{90i} = mass of the specimen after moistening it for 90 minutes (g);
- A_i = area of the semi prism at 28 days;
- C_i = water absorption coefficient ($\frac{kg}{m2\ x\ min^{0.5}}$).

3.5.8 Water absorption by immersion

The test for water absorption by immersion, according to LNEC E-394 [12], measures, in percentage, the amount of interconnected voids of a mortar.

➢ Equipment:

- Stove;
- Precision scale 0.01 g;
- Absorbent filter paper;
- Water vessel;

➢ Procedure:

- Place the specimens in a container;
- Add water until the specimen is immersed, in intervals of one hour, at 1/3 of its height, 2/3 of its height and in its entirety. The final water level should not exceed 20 mm of the top face of the specimen;
- It is considered that constant mass of the saturated mortars m_1 is reached, when the difference between the weights obtained in two consecutive weightings, in a 24-hour interval, is less than 0.1% of the average of the two measurements;
- Before each weighting, dry the surface of the specimen with an absorbent cloth or

a natural sponge, in order to remove surface water;

- After reaching constant mass, weight the mortars in water where m_2 will correspond to the hydrostatic mass after saturation;
- Remove the supply from the water and proceed to dry until constant mass in a ventilated oven at a temperature of 105 ± 5 °C (m_3);

➢ Calculation:

$$A = \frac{m_1 - m_3}{m_1 - m_2} \, x \, 100$$

- m_1 is the mass of the mortar saturated with air (g);
- m_2 is the hydrostatic mass of the saturated mortar (g);
- m_3 is the mass of the dried mortar (g).

3.5.9 Carbonation

The determination of resistance to carbonatation will be carried out according to standard EN 13295 [5]. In this test, mortar resistance against carbonation is measured in an accelerated environment where samples are exposed to an atmosphere containing 5 ± 0.1% CO_2 at a temperature of 23 ± 3 °C and relative humidity of 60 ± 5%.The carbonation depth is measured by applying a phenolphthalein indicator from a broken piece.

➢ Equipment:

- Carbonation chamber;
- Phenolphthalein solution;

➢ Procedure:

- The samples are placed inside the carbonation chamber until testing age;
- The depth of carbonation is measured on the split face of the prisms, using a phenolphthalein indicator. The depth of carbonation (mm) is the average depth of the four sides.

4 Schedule

Table 7 presents the schedule to follow for the development of each of the experimental stages.

Table 7 - Schedule

ACTIVITIES		MONTHS														
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Preparation and grinding MIBA		■	■													
Fine aggregate characterization			■													
First experimental stage	Mortar manufacturing and thermal curing			■												
	Fresh state tests (Density, workability and air content)			■												
	Bulk density			■	■	■	■	■	■	■						
	Dynamic modulus of elasticity			■	■	■	■	■	■	■						
	Ultrasound			■	■	■	■	■	■	■						
	Flexural strength			■	■	■	■	■	■	■						
	Compressive strength			■	■	■	■	■	■	■						
	Water absorption by capillarity			■	■	■	■	■	■	■						
	Water absorption by immersion			■	■	■	■	■	■	■						
	Carbonation			■	■	■	■	■	■	■						
Second experimental stage	Mortar manufacturing and thermal curing				■											
	Fresh state tests (Density, workability and air content)				■											
	Bulk density				■	■	■	■	■	■	■					
	Dynamic modulus of elasticity				■	■	■	■	■	■	■					
	Ultrasound				■	■	■	■	■	■	■					
	Flexural strength				■	■	■	■	■	■	■					
	Compressive strength				■	■	■	■	■	■	■					
	Water absorption by capillarity				■	■	■	■	■	■	■					
	Water absorption by immersion				■	■	■	■	■	■	■					
	Carbonation				■	■	■	■	■	■	■					
Third experimental stage	Mortar manufacturing and thermal curing					■										
	Fresh state tests (Density, workability and air content)					■										
	Bulk density					■	■	■	■	■	■					
	Dynamic modulus of elasticity					■	■	■	■	■	■					
	Ultrasound					■	■	■	■	■	■					
	Flexural strength					■	■	■	■	■	■					
	Compressive strength					■	■	■	■	■	■					
	Water absorption by capillarity					■	■	■	■	■	■					
	Water absorption by immersion					■	■	■	■	■	■					
	Carbonation					■	■	■	■	■	■					
Fourth experimental stage	Mortar manufacturing and thermal curing						■									
	Fresh state tests (Density, workability and air content)						■									
	Bulk density						■	■	■	■	■	■				
	Dynamic modulus of elasticity						■	■	■	■	■	■				
	Ultrasound						■	■	■	■	■	■				
	Flexural strength						■	■	■	■	■	■				
	Compressive strength						■	■	■	■	■	■				
	Water absorption by capillarity						■	■	■	■	■	■				
	Water absorption by immersion						■	■	■	■	■	■				
	Carbonation						■	■	■	■	■	■				
Repetitions													■	■	■	■

References

1. EN-933-1, *Tests for geometrical properties of aggregates - Part 1: Determination of particle size distribution. Sieving method*. 2012, Comité Européen de Normalisation (CEN): Brussels, Belgium. 22 p.

2. EN-1097-6, *Tests for mechanical and physical properties of aggregates - Part 6: Determination of particle density and water absorption*. 2013, Comité Européen de Normalisation (CEN): Brussels, Belgium. 54 p.

3. CEU, *Council Directive 98/83/EC of 3 November 1998 on the quality of water intended for human consumption.* Official Journal of the European Communities, 1998. **330**: pp. 32-54.

4. EN-196-1, *Methods of testing cement - Part 1: Determination of strength*. 2005, Comité Européen de Normalisation (CEN): Brussels, Belgium. 36 p.

5. EN-13295, *Products and systems for the protection and repair of concrete structures.Test methods. Determination of resistance to carbonation*. 2004, Comité Européen de Normalisation (CEN): Brussels, Belgium. 18 p.

6. EN-1015-13, *Methods of test for mortar for masonry - Part 13: Determination of dimensional stability of hardened mortars*. 1993, Comité Européen de Normalisation (CEN): Brussels, Belgium. 20 p.

7. EN-1015-10, *Methods of test for mortar for masonry - Part 10: Determination of dry bulk density of hardened mortar*. 1999, Comité Européen de Normalisation (CEN): Brussels, Belgium. 10 p.

8. EN-12504-4, *Testing concrete. Determination of ultrasonic pulse velocity*. 2004, Comité Européen de Normalisation (CEN): Brussels, Belgium. 18 p.

9. EN-14146, *Natural stone test methods. Determination of the dynamic elastic modulus of elasticity (by measuring the fundamental resonance frequency)*. 2004, Comité Européen de Normalisation (CEN): Brussels, Belgium. 18 p.

10. EN-1015-11, *Methods of test for mortar for masonry - Part 11: Determination of flexural and compressive strength of hardened mortar*. 1999, Comité Européen de Normalisation (CEN): Brussels, Belgium. 12 p.

11. EN-1015-18, *Methods of test for mortar for masonry - Part 18: Determination of water absorption coefficient due to capillary action of hardened mortar*. 2002, Comité Européen de Normalisation (CEN): Brussels, Belgium. 12 p.

12. LNEC-E394, *Concrete: determination of water absorption by immersion - testing at atmospheric pressure (in Portuguese)*. 1993, National Laboratory in Civil Engineering (LNEC - Laboratório Nacional de Engenharia Civil) Lisbon, Portugal. 2 p.

13. EN-1015-6, *Methods of test for mortar for masonry - Part 6: Determination of bulk density of fresh mortar*. 1999, Comité Européen de Normalisation (CEN): Brussels, Belgium. 8 p.

14. EN-1015-3, *Methods of test for mortar for masonry - Part 3: Determination of consistence of fresh mortar (by flow table)*. 1999, Comité Européen de Normalisation (CEN): Brussels, Belgium. 10 p.

15. EN-1015-7, *Methods of test for mortar for masonry - Part 7: Determination of air content of fresh mortar*. 1999, Comité Européen de Normalisation (CEN): Brussels, Belgium. 8 p.