

RINOPOLYCRETE - TOWARDS A CEMENT-FREE AND FULLY RECYCLED CONCRETE



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**Recycled inorganic polymer concrete - Towards a cement-free and fully recycled concrete
(RInoPolyCrete)**

Task 3 - Report 2

Test methods for the second stage of Task 3

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ACRONYMS

MIBA	Municipal solid waste incinerator bottom ash
AAM	Alkali-activated materials
NaOH	Sodium hydroxide
SiO ₂	Silicon oxide
Na ₂ O	Sodium oxide
FA	Fly ash
WRA	Water reducing admixture
Na ₂ SiO ₃	Sodium silicate

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1 Introduction

This report presents a description of the upcoming experimental campaign on the alkali activation of municipal solid waste incinerator bottom ash (MIBA) in the production of mortars. Existing literature on the subject has shown that mortars comprising MIBA as the sole precursor may exhibit reasonable compressive strength though below the requirements for structural concrete [1]. In parallel, activated fly ash (FA), which will also be used in this campaign as a control material, generally produces specimens with more adequate mechanical performance [2]. This difference is basically due to the fact that in MIBA the amorphous phases of the aluminosilicates are present in a lower amount when compared with those available in FA and thus MIBA has a lower reactivity [3-5]. The latter material also contains other deleterious materials, such as metallic aluminium or high organic carbon content due to the low efficiency of the combustion process [6].

This campaign has the purpose of finding an optimal mixture of MIBA and FA as precursors in the production of alkali activated materials (AAM), in order to improve the mechanical properties of the product. The viability of the precursor's composition MIBA + FA using the following mix proportions 75/25; 50/50; 25/75 will be assessed. Mechanical performance-based optimization of the alkali activator has been reached in a previous stage. Finally, each of the tasks expected to be carried out are duly characterized, from the stage of acquiring and preparing of MIBA and FA to fresh- and hardened-state tests of the monolithic material, as well as the methodology used for the analysis of the data obtained and expected results.

2 Materials

2.1 Municipal incinerator bottom ash

2.1.1 Collection and sampling

MIBA collection was carried out in Valorsul facility. The collected sample for this experimental campaign corresponds to the residue generated in 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 in 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).



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

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, separated by one hour, 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 and c).

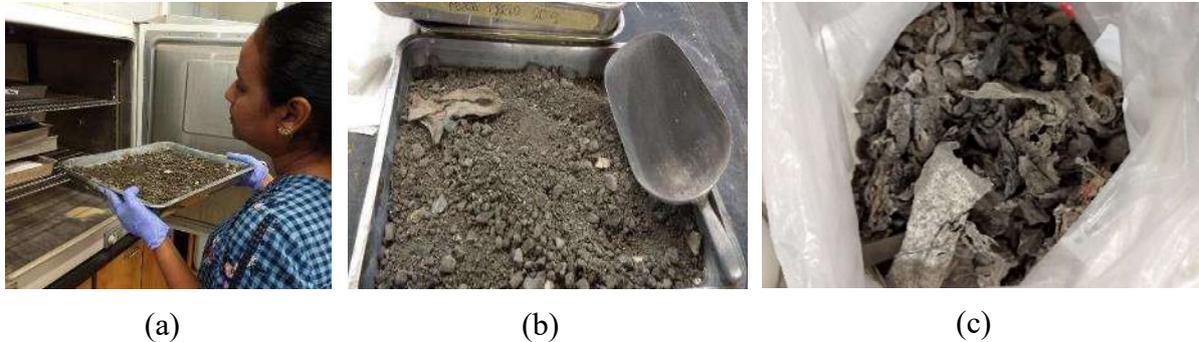


Figure 3 - (a) Drying process MIBA; (b) dry MIBA; (c) contaminants removed by manual cleaning

The grinding stage is divided into three stages:

a. Grinding in the Los Angeles machine:

20 kilograms of material are initially grinded 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 5). The resulting MIBA is screened by using a 4 mm sieve, in order to separate the larger particles for later reduction in size.



Figure 4 - (a) Grinding in the Los Angeles machine; (b, c) size separation through sieve N. 4

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.

c. Crushing in the ball mill:

About 20 kg 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 5). Figure 6 shows MIBA in the different grinding stages.



(a)

(b)

Figure 5 - (a) Cylinder mill; (b) ball mill



(a)

(b)

(c)

Figure 6 - MIBA at different stages of grinding: (a) MIBA upon entering the Los Angeles machine; (b) MIBA at the exit of the Los Angeles machine; (c) MIBA at the exit of the ball mill machine

2.2 Fly ash

FA, supplied by EDP - Gestão da Produção de Energia, S.A. at the Sines Production Centre factory. This precursor does not require preparation stages for its use.

2.3 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.

2.3.1 Particle size distribution

The particle size distribution will be carried out according to EN 933-1 [7]. 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 $\pm 0.1\%$ 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 the 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) °C 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 the sieve arrangement mechanically. 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 (R_i), 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:

M₁ is the dry mass of the test portion, in kilograms;

M₂ is the dry mass of the residue retained in the 0.063 mm sieve, in kilograms;

P is the 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:

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

2.3.2 Bulk density and water absorption

The density and water absorption will be carried out according to EN 1097-6 [8], 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 weighing 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 weighing in 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:

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

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

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

Water absorption after immersion is calculated using the following formula:

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

Where:

ρ_a - Apparent particle density (kg/dm³);

ρ_{rd} - Oven-dried particle density (kg/dm³);

ρ_{ssd} - Saturated and surface-dried particle density (kg/dm³);

ρ_w - Density of water at the test temperature (kg/dm³);

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).

2.4 Alkaline activator

The alkaline activator is a solution that is prepared in the laboratory and is made up of one or two solutes and a solvent. Once the solution is prepared, temperature is measured using a thermometer and the pH with the use of a digital pH meter.

2.4.1 Solute

The solutes used in the preparation of the alkaline activator correspond to sodium hydroxide and sodium silicate.

2.4.1.1 Sodium hydroxide

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

2.4.1.2 Sodium silicate

Reactive grade sodium silicate with a SiO₂/Na₂O ratio of 3.3 ± 0.1; 26.4 ± 1.5% silicon oxide, 8 ± 0.6% sodium oxide and 65.6 ± 2% water.

2.4.2 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 [9].

2.5 Water reducing admixture

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

3 Preparation of mortars

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. The response variable, by which the optimum mix design is determined, corresponds to the compressive strength. The first factor corresponds to the mass ratio of the FA/MIBA precursors, in which the 75% MIBA-25% FA, 50% MIBA-50% FA, 25% MIBA-75% FA will be evaluated. The ratios associated with the alkaline activator $\text{Na}_2\text{O} / \text{Binder}$ and $\text{SiO}_2 / \text{Na}_2\text{O}$ will be determined by a proportionality criterion, based on the optimal values found for the material activated by alkali with FA and MIBA as the only precursor; therefore, at this stage, three mix designs will be produced with values for the factors (i.e. $\text{Na}_2\text{O} / \text{Binder}$ and $\text{SiO}_2 / \text{Na}_2\text{O}$) previously fixed, temperature and curing time.

3.1 Determination of quantities

The amounts of each of the constituents for the manufacture of AAM will be calculated based on volumetric ratios, using the densities of the raw materials. The binder/aggregate volumetric ratio ($V_B/V_A = 0.33$); the volumetric ratio of $\text{SiO}_2/\text{Na}_2\text{O}$ will be determined by a proportionality criterion. Table 2 shows the total quantities required for the development of this experimental campaign.

Table 1 - Operationalization of the variables

Factor	Unit of measurement	Mix design		
		1	2	3
MIBA/FA	Dimensionless	75/25	50/50	25/75
Na ₂ O/binder	% in weigh	$0.75 \times \left(\frac{\text{Na}_2\text{O}}{\text{Binder}}\right)_{\text{MIBA_op}}$ $+ 0.25 \times \left(\frac{\text{Na}_2\text{O}}{\text{Binder}}\right)_{\text{FA_op}}$	$0.5 \times \left(\frac{\text{Na}_2\text{O}}{\text{Binder}}\right)_{\text{MIBA_op}}$ $+ 0.5 \times \left(\frac{\text{Na}_2\text{O}}{\text{Binder}}\right)_{\text{FA_op}}$	$0.25 \times \left(\frac{\text{Na}_2\text{O}}{\text{Binder}}\right)_{\text{MIBA_op}}$ $+ 0.75 \times \left(\frac{\text{Na}_2\text{O}}{\text{Binder}}\right)_{\text{FA_op}}$
SiO ₂ /Na ₂ O	Dimensionless	$0.75 \times \left(\frac{\text{SiO}_2}{\text{Na}_2\text{O}}\right)_{\text{MIBA_op}}$ $+ 0.25 \times \left(\frac{\text{SiO}_2}{\text{Na}_2\text{O}}\right)_{\text{FA_op}}$	$0.5 \times \left(\frac{\text{SiO}_2}{\text{Na}_2\text{O}}\right)_{\text{MIBA_op}}$ $+ 0.5 \times \left(\frac{\text{SiO}_2}{\text{Na}_2\text{O}}\right)_{\text{FA_op}}$	$0.25 \times \left(\frac{\text{SiO}_2}{\text{Na}_2\text{O}}\right)_{\text{MIBA_op}}$ $+ 0.75 \times \left(\frac{\text{SiO}_2}{\text{Na}_2\text{O}}\right)_{\text{FA_op}}$
Curing Temperature	°C	80		
Curing time	h	24		

Table 2 -Total amount per required material

Material	Amount (kg)
MIBA	29.6
Fly ash	28.3
Fine sand	49.8
Coarse Sand	115.55
Sodium hydroxide	3.3
Sodium silicate	26.9
WRA	1.15

The ratio water/binder (V_W/V_B) varies with the preparation of the alkaline activator, since one of the activators contains water. Two mass ratios are considered: water reducing admixture/precursor and Na₂O/precursor.

3.2 Procedure for the production and processing of mortars

The production and preparation of mortars is carried out in accordance with EN 196-1, 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;
- Compaction apparatus.

3.2.1 Mortar manufacturing

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

- Weigh all solid constituents of the mortar according to subchapter 2.2
- Add the NaOH to the solvent (pre-measured volume of water) and homogenize the solution with a glass stirrer;
- Add the alkaline solution in the mixer bowl and then the precursor MIBA
- Alkali activated mortars with MIBA as precursor require a longer contact time with the alkaline solution (Figure 7), 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 mix is fresh. 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 2.2 (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, 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.



Figure 7 - (a) Materials for the preparation of AAM; (b) reaction of MIBA and NaOH

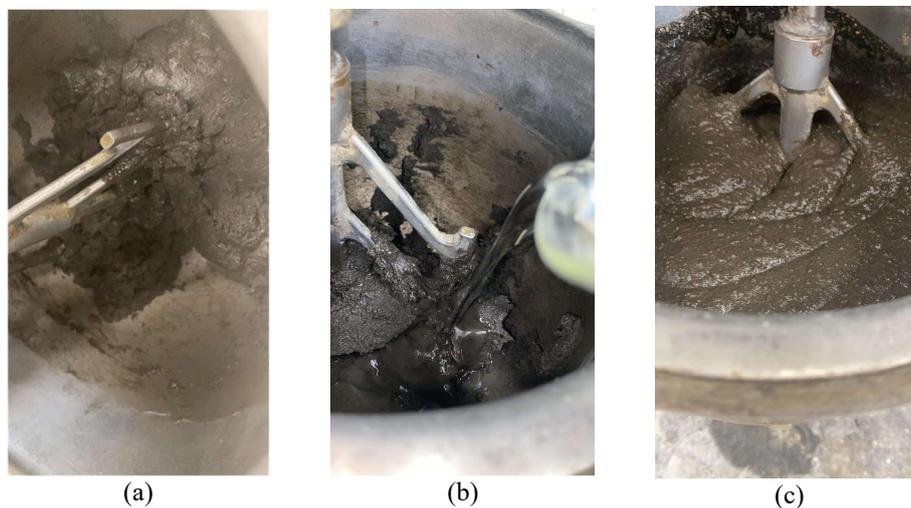


Figure 8 - AAM preparation: (a) MIBA mixture after 24 hours of rest; (b) addition of sodium silicate (c); AAM in fresh state with the added aggregate

- Turn on the equipment and mix for 5 minutes (Figure 8a);
- Add the FA and mix for 5 minutes more; with a plastic paddle, remove the mortar from the walls of the bowl;
- Add the sodium silicate and continue mixing for 5 more minutes (Figure 8b);
- Add the coarse and fine sand and mix for 5 minutes (Figure 8c);
- Finally, stop the mixer and with a plastic paddle remove the mortar from the walls of the bowl.

3.2.2 Mortar moulding

Mortar moulding is done using the following procedure:

- Line each of the mould parts with a plastic film (Figure 9);
- 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 shake 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.

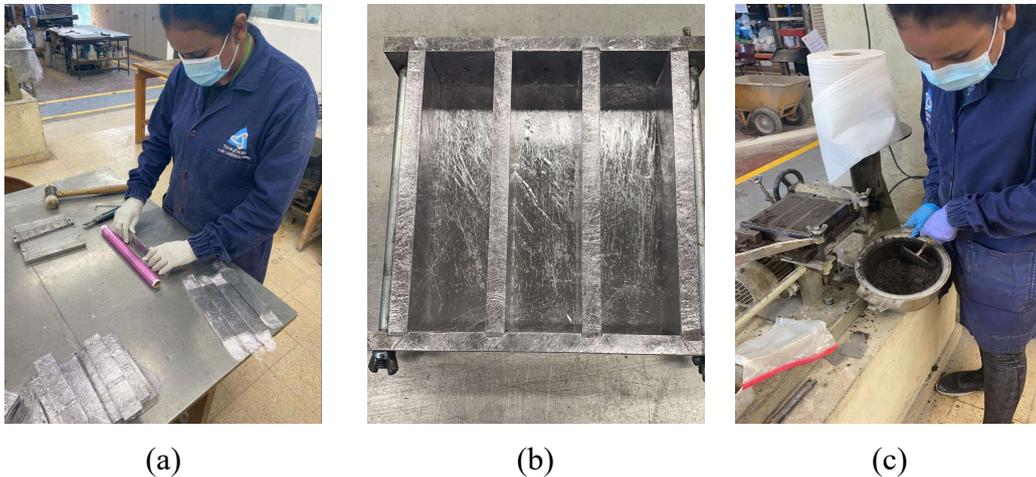


Figure 9 - (a) Preparation of the moulds; (b) moulds covered with plastic film; (c) mortar placement

3.2.3 Curing conditions

Once the moulding of the mortars is finished, these remain for a period of 24 hours in ambient conditions and subsequently introduced 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 3.

The time the mortars must remain in the chamber is specified in each of the tests.

Table 4 corresponds to the total number of specimens to be prepared to satisfy the proposed experimental campaign.

Table 3 - Curing conditions, test methods and number of specimens per family of alkali activated materials

Test	Standard	Number of specimens	Curing conditions
Carbonation	EN 13295 [11]	6	28 days in dry chamber; place in carbonation chamber until testing age.
Shrinkage	EN 1015-13 [12]	3	Specimens placed in dry chamber after demoulding until the end of the test.
Bulk density	EN 1015-10 [13]	3	
Ultrasound	EN 12504-4 [14]		
Elasticity module	EN 14146 [15]	9	
Flexural strength	EN 1015-11 [16]		
Compressive strength	EN 1015-11 [16]		
Water absorption by capillarity	EN 1015-18 [17]	3	
Water absorption by immersion	LNEC E-394 [18]	3	
Total specimens		27	

Table 4 - Total number of specimens to be prepared

MIBA/FA	Number of families by mix design	Number of moulds	Number of specimens
0.25/0.75	2	8	48
0.5/0.5	2	8	48
0.75/0.25	2	8	48
Total		48	144

3.3 Fresh state performance

3.3.1 Density

The determination of bulk density in the fresh state will be carried out according to EN 1015-6 [19]. The test consists in 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:

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

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.

3.3.2 Workability

The workability will be carried out according to EN 1015-3 [20]. The test consists in 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 of the height initially given (Figure 10).



(a)

(b)

Figure 10 - Workability step one (a) mortar slump (b)

➤ 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 the 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 approximately 1 per second;
 - Measure the diameter of the mortar in two directions at right angles, using the calliper.

3.3.3 Air content

The determination of air content in the fresh state will be carried out as per standard EN 1015-7 [21]. In this test, a volume of mortar is placed in a specific measuring vessel (Figure 11). Subsequently, water is introduced into 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;



Figure 11 - Equipment of air content

- Using the palette knife 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.

3.4 Mortars testing in hardened state

3.4.1 Bulk density

The determination of dry bulk density of hardened mortar will be carried out according to standard EN 1015-10 [13]. 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 occupies.

➤ Equipment:

- Scale with 0.1 g precision;
- Oven;
- Calliper.

➤ Procedure:

- Dry the specimen in an oven at a temperature of 105 ± 5 °C, until a 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:

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

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

3.4.2 Dynamic modulus of elasticity

The determination of the dynamic modulus of elasticity will be carried out according to standard EN 14146 [15]. 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 (Figure 12).

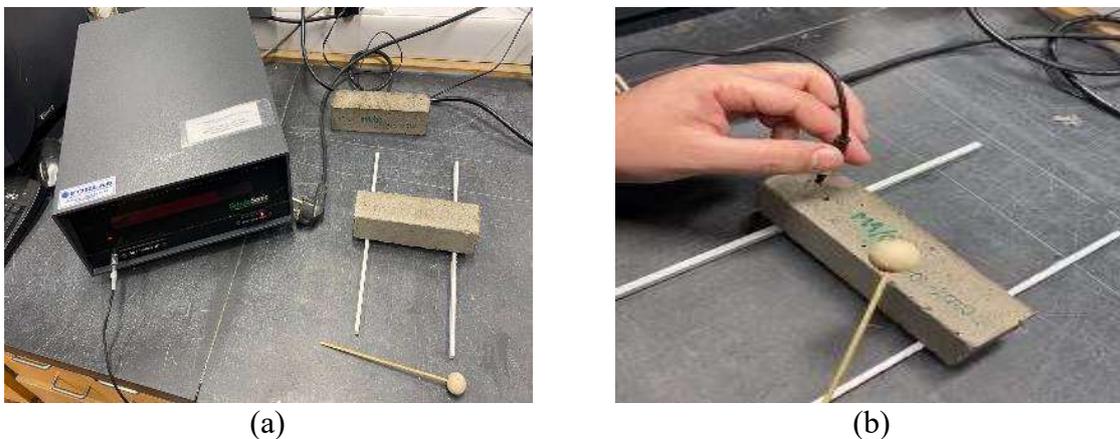


Figure 12 - Modulus of elasticity equipment (a) specimen (b)

➤ 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:

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

- E_{di} = dynamic elastic modulus of the test specimen i (MPa)
- L_i = length of the test specimen i (mm)
- F_i = longitudinal resonance frequency of test specimen i (Hz)
- ρ_i = specific weight of the specimen i (N/dm³).
- g = acceleration of gravity.

3.4.3 Ultrasound pulse velocity

The determination of the ultrasound pulse velocity will be carried out according to standard EN 12504-4 [14]. This test measures the time in microseconds that a given ultrasonic wave takes from the moment it is emitted, until it is received after covering the distance between an emitting unit and a receiving unit. In this test, it is possible to determine cracks or pores inside the samples, through abrupt variations in speed. The speed 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 to the modulus of elasticity and mechanical strength.

- Equipment:
 - Scale;
 - Ultrasound pulse velocity device (Figure 13).
- 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.

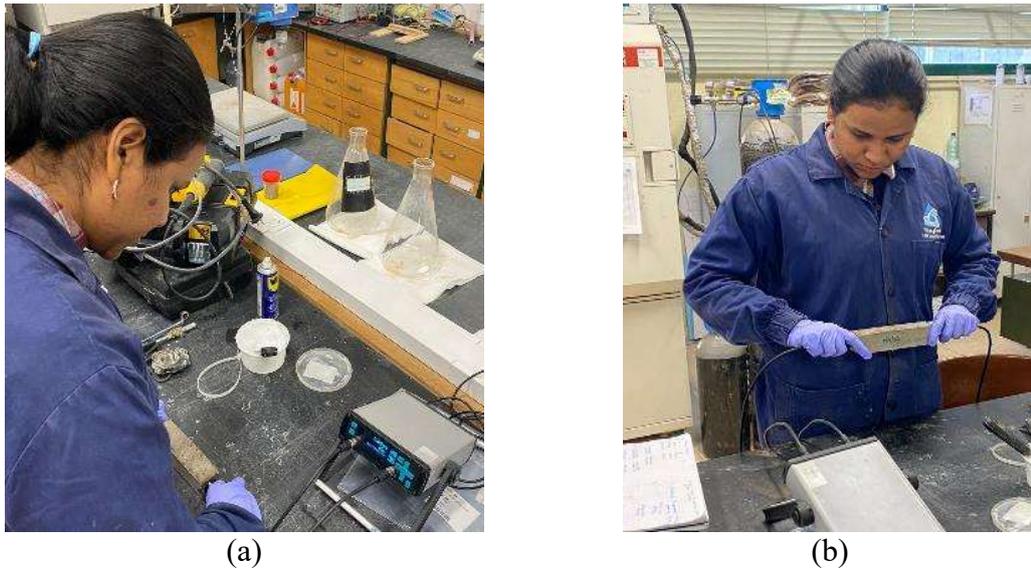


Figure 13 - (a) Ultrasound pulse velocity equipment; (b) measurement

➤ Calculation:

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

- v_i - Propagation velocity in test specimen i (mm/s);
- d_i - Distance between sender and receiver;
- t_i - Propagation time, average value of two or three samples.

3.4.4 Shrinkage

The determination of shrinkage will be carried out according to standard EN 1015-13 [12]. This test measures the shrinkage of mortars, in a longitudinal direction over time.

➤ Equipment:

- Shrinkage measuring equipment (Figure 14);
- Scale with precision of 0.01 g.

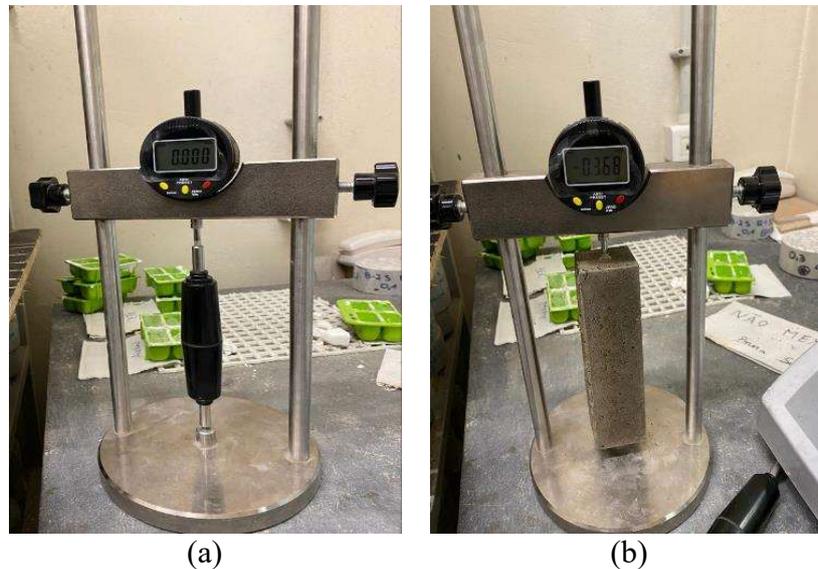


Figure 14 - (a) Shrinkage equipment; (b) test execution

➤ 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.

3.4.5 Flexural strength

The determination of flexural strength of hardened mortar will be carried out according to the standard EN 1015-11 [16]. In this test, the flexural strength is determined by a three-point load to a hardened mortar prism.

➤ Equipment:

- Flexural and compression strength equipment (Figure 15).

➤ 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 rupture between 30 and 90 seconds after the start of the test.

➤ Calculation:

$$f_i = 1.5 \frac{F_i \times l}{b_i \times d_i^2}$$

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

3.4.6 Compressive strength

The determination of compressive strength will be carried out according to EN 1015-11 [16]. 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.



Figure 15 - Equipment to determine flexural strength and compression strength of mortars

- Equipment:
 - Flexural and compression strength equipment (Figure 15).
- Procedure:
 - Apply a force with a uniform load between 50 and 500 N/s, until rupture 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 specimen i .
- C_i - Maximum force supported by test specimen i (N)
- A_i - Section area compressed by the prism i (mm^2)

3.4.7 Water absorption by capillarity

The determination of water absorption by capillarity will be carried out according to EN 1015-18 [17]. 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;
- Absorbent filter paper;
- Supports.

➤ Materials:

- Water;
- Sealing material such as paraffin or resin with a melting point higher than $60\text{ }^\circ\text{C}$.

➤ Procedure:

- Dry to constant mass in an oven at $60 \pm 5\text{ }^\circ\text{C}$;
- 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 place 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 90 minutes (g);
- A_i - Area of the semi prism at 28 days;
- C_i - Water absorption coefficient ($\frac{kg}{m^2 \times min^{0.5}}$).

3.4.8 Water absorption by immersion

The test for water absorption by immersion, according to LNEC E-394, 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 weighing, dry the surface of the specimen with an absorbent cloth or a natural sponge, in order to remove surface water;
- After reaching constant mass, weigh 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:

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

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

3.4.9 Carbonation

The determination of resistance to carbonation will be carried out according to standard EN 13295 [11]. In this test, mortar resistance against carbonation is measured in an accelerated environment where samples are exposed to an atmosphere containing $5 \pm 0.1\%$ CO_2 at a temperature of 23 ± 3 °C and relative humidity of $60 \pm 5\%$. The carbonation depth is measured by applying a phenolphthalein indicator in 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 4 sides.

4 Schedule

Table 5 presents the schedule to follow for the development the experimental campaign.

5 Expected results

In order to satisfy the objective proposed in this experimental campaign, the data will be analysed through the MINITAB statistical software, in which an analysis of variance, ANOVA, will be developed for each of the experimental stages. This will allow determining the selected level that statistically yields the best results in terms of the compressive strength response variable, thereby fulfilling the objectives of this experimental campaign. In the same way for each one of the tests in the fresh and hardened state, the respective analyses and comparison of the results of other authors will be carried out.

Table 5 - Schedule experimental campaign

Task N.º	Task denomination											
		1	2	3	4	5	6	7	8	9	10	
1	Collection and sampling of MIBA											Task completed
2	Preparation and grinding of MIBA											Task completed
3	Preparation of Mortars 25% MIBA - 75 % FA											
4	Preparation of Mortars 50% MIBA - 50 % FA											
5	Preparation of Mortars 75% MIBA - 25% FA											
6	Performance assessment of AAM											
7	Durability assessment of AAM											
8	Possible repetitions											
9	Analysis of the results											

The mechanical properties of alkali activated materials are expected to improve with increasing proportion of FA in the precursor, as this material does not have the issue of metallic aluminium, as it does in MIBA. This would result in a more stable mix in the fresh state, contributing to the formation of the reaction products responsible for the structure of the alkaline materials already in the hardened state.

The compressive strength of alkali-activated mortars is expected to increase as the concentration of sodium silicate increases ($\text{SiO}_2/\text{Na}_2\text{O}$), and once the optimum value is exceeded, the mechanical properties are likely to be affected. This behaviour is to be expected, since exceeding the optimum concentration of sodium silicate could present fresh mixes with low densities, because the amount of sodium hydroxide added to the MIBA is low, and therefore the release of hydrogen is delayed until mixing with the silicate.

Regarding the durability performance, a decline is expected due to the porosity presented by

the specimens when gaseous hydrogen is released from the reaction of the metallic aluminium present in alkali activated materials with higher MIBA ratios.

Finally, a material that satisfies the minimum specifications required to scale its production as concrete is expected to be developed.

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