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Use of recycled by-products as cement mortar binders and aggregates to lower the carbon footprint.
Ús de subproductes reciclats com a matriu i agregats en morters de ciment per reduir la petjada de carboni.

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In my view, all that is necessary for faith is the believe that by doing our best we shall succeed in our aims.

Rosalind Franklin

I would like to give a special thanks to my parents who have been there all through my college journey.

To my Grandma, for opening me the science window.

I'm feeling particularly grateful for the opportunity given to me through this internship program to conduct my research about one of my many passions surrounding chemistry: the issue of alternatives to raw materials. And how can we, as individuals and companies, move forward to work together to ensure effective, sustainable change.

The success of this work is mainly due to Dr. David Gonzalo who introduced me the delights of the construction industry and taught me the first steps on how to investigate.

I would also like to give thanks to my Saint Gobain laboratory team for always keeping a warm and friendly atmosphere around them.

Thanks to Dr. Daniel Sainz for providing me with the tools necessary in order to adequately research, portray, and present my findings.

REPORT

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1. SUMMARY

The increasing generation of waste materials means alternatives need to be found for their final disposal. The reuse of industrial by-products in mortar production offers many benefits such as economic and environmental. To give a specific use to these by-products many tests may be driven to ensure an effective replacement and that such replacement will not affect to the final properties of the mortar.

Such replacements in a cement mortar base can be done by substituting an inert aggregate or by replacing the reactive binder. Most common and spread replacements of the mortar binders have been done by several industries using FCC (fluid catalyst cracking) coming from the fluidized bed of petroleum refining.

Also, until now fly ashes have been widely used in cementitious binders in addition to Portland cement. However, the gradual closure of coal-fired plants raises a problem of future availability of fly ashes. Thus, biomass ashes may be considered a more sustainable alternative to fly ashes.

Keywords: waste materials, mortar, cement binders, aggregates, FCC, biomass ashes.

2. RESUM

El constant augment de residus generats per les indústries, obliga a buscar alternatives per a la seva eliminació final. La reutilització de subproductes industrials en la producció de morters ofereix un gran ventall de beneficis no només econòmics sinó també mediambientals. Per tal de donar un ús específic a aquests subproductes cal realitzar diversos anàlisis de caracterització d'aquestes primeres matèries amb l'objectiu d'assegurar uns substitució efectiva i que dita substitució no afecti a les propietats finals del morter.

Tals substitucions en una base de morter de ciment poden realitzar-se substituint un àrid inert o bé es pot fer la substitució del lligand reactiu, és a dir, el ciment. Les substitucions més comunes i esteses del lligand actiu d'un morter han estat realitzades per diferents indústries utilitzant FCC (Fluid Catalyst Cracking) procedent del llit fluïditzat del refinament del petroli.

De la mateixa manera, fins ara s'ha estat utilitzant àmpliament les cendres volants en lligands de morter com a substitut del ciment Portland. No obstant, el tancament progressiu de les centrals de carbó planteja un problema de la futura disponibilitat d'aquestes cendres. Per això, les cendres de biomassa poden considerar-se una alternativa més sostenible que les cendres volants.

Paraules clau: subproductes industrials, morter, lligand de ciment, àrid inert, FCC, cendres de biomassa.

3. INTRODUCTION

This project is the result of my internship at the R&D department of Saint Gobain Weber on my last year of the chemistry degree. It has been possible thanks to an agreement between the University of Barcelona and the research department of Saint Gobain. Throughout this year, I have been introduced to the cement industry and I have been working to reach sustainable goals in the frame of the *Wasterials Project. Wasterials* is born to replace the raw materials present in the Saint Gobain products for by-products coming from external industries that otherwise, these by-products, would be thrown away. The main goal of Wasterials is to reduce the CO_2 footprint from the Saint Gobain company and reaching carbon neutrality by 2050 from all the Saint Gobain companies. This work is just a little contribution to this big, ambitious and ecofriendly project.

3.1. SAINT GOBAIN

Saint Gobain is a French multinational corporation founded in 1665 in Paris. It started being a glass production company and it now produces a wide variety of construction products and high-performance materials.

The company was founded by Jean-Baptiste Colbert the economy minister of Louis XIV of France in order to boost French artisans. Its first project was to develop the technologies in order to reform *La Galerie des glaces* at Versaille's palace. From there, Saint Gobain is now the main manufacturer of the glass industry in the world and has also set a variety of companies into the construction field. (1)

Nowadays, the company has 200,000 workers worldwide.

Saint Gobain is divided into seven affiliate companies each of them having a different role into the construction field.

These companies are: *Saint Gobain Isover* offering thermal insulation solutions, *Saint Gobain Ach* offereing soundproofing solutions, *Saint Gobain Eurocoustic* an expert in the suspended ceilings market, *Saint Gobain Ecohphon* fabricating acoustic panels, *Saint Gobain*

Placo leader in the fabrication of laminated gypsum boards, *Saint Gobain Pam* giving iron pipe solutions for water and sewage networks and finally *Saint Gobain Weber* world leader of premixed mortars, solutions for facades, tile fixing and flooring.

3.2. CHEMISTRY OF THE CEMENTS

3.2.1 Definition of the cement

Cements are defined as hydraulic conglomerates that, once mixed with water, form pastes that set and harden due to hydration reactions of their constituents, giving rise to hydrated products that are mechanically resistant and stable to both air and water. The raw materials used are basically limestone, clay and gypsum which are mainly composed of silicates and aluminates.

3.2.2 Fabrication of the cement

Cement is fabricated by heating limestone with small quantities of materials carrying silica and alumina (such as clay) to about 1500 ºC in a rotatory kiln; this process is known as calcination.

The resultant hard, sintered "clinker" is then grounded together with a small amount of gypsum into a fine powder to produce the so-called: "Ordinary Portland Cement" OPC. The most commonly used type of cement.

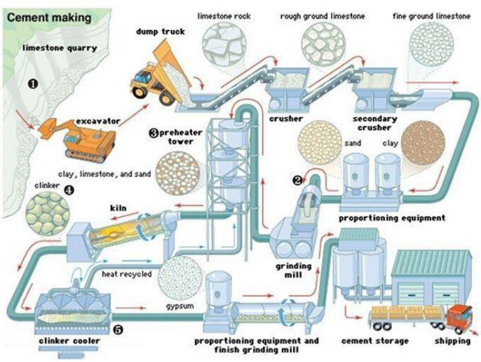

Figure 1: *Process of the fabrication of the cement.* [3]

Clinker composition	
Compound	Amount (%)
CaO	64
SiO_2	20
Al_2O_3	6
Fe_2O_3	3
Other	7

Table 1: *Clinker compositon.* [4]

3.2.3 Types of cement

There's a wide variety of classification for the cement attending parameters such as: chemical composition, mechanical and physical requirements.

The UNE EN-197-1 regulation classifies and guarantees the quality of their specifications. In the following tables there's the classification of the cement attending their chemical composition and their physical requirements.

Regarding their chemical composition. Classification of cements attending its chemical composition:

- Portland Cement: Hydraulic cement it's the result of a mixture of clinker and gypsum.
- Siderurgical Cement: Product obtained from a mixture of clinker, blast furnace slag and gypsum.
- Portland Pozzolan cement: Hydraulic cement it has a combination of Portland Cement (94-65%) and pozzolans (6-35%):
- High-Alumina Cement: Product obtained from the milling of a clinker obtained from the calcination of the bauxite and aluminum oxide.

3.2.4 Chemical composition of cement

There are four products mainly present in the clinker. Its proportion will define the type of cement. Cement chemists use the following abbreviations to describe the compounds present in the clinker.

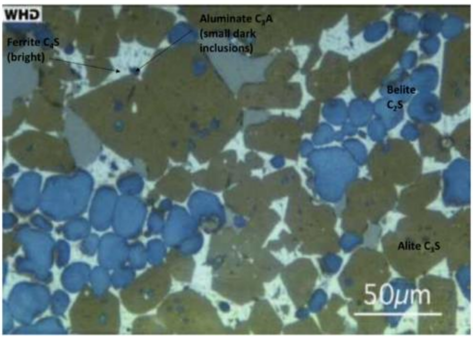

Figure 2: *Polished section of a clinker nodule.* [9]

Abbreviation	Compound
A	Al_2O_3
C	CaO
F	Fe_2O_3
H	H_2O
M	MgO
S	SiO_2
Ŝ	SO_3

Table 2: *Abbreviations on cement chemistry.* [8]

Abbreviation	Chemical compound		Common name	% in clinker	Comments
C_3S	3 $CaO.SiO_2$	Tricalcium silicate	Alite	50-70	Hydrates and hardens quickly it gives the early strength. Higher heat of hydration.
C_2S	2 $CaO.SiO_2$	Dicalcium silicate	Belite	15-30	Hydrates and hardens slower. Gives off less heat. High late strength.
C_3A	3 $CaO.Al_2O_3$	Tricalcium aluminate	Aluminate	5-15	Very high heat of hydration. Some contribution to early strength.
C_4AF	4 $CaO.Al_2O_3.Fe_2O_3$	Tetracalcium aluminoferrite	Ferrite	5-20	Little contribution to the strength. Lowers clinkering temperature and controls de color of the cement.

***Table 3:** Abbreviations on clinker composition* [(8)].

3.2.5 Hydration of the cement

In the presence of water, all the previous described compounds, hydrate to form new compounds that are the infrastructure of the hardened cement paste. [(8)]

2 (3 $CaO.SiO_2$) + 11 H_2O $\longrightarrow$ 3 $CaO.2SiO_2.8H_2O$ **(C-S-H)** +3 ($CaO.H_2O$)

3 $CaO.SiO_2$ + 9 H_2O $\longrightarrow$ 3 $CaO.2SiO_2.8H_2O$ **(C-S-H)** + $CaO.H_2O$

3 $CaO.Al_2O_3$ + 3 ($CaO.SO_3.2H_2O$) gypsum + 26 H_2O $\longrightarrow$ 6($CaO.Al_2O_3.3SO_3.32H_2O$)

3 $CaO.Al_2O_3$ + 6$CaO.Al_2O_3.3SO_3.32H_2O$ + 4 H_2O $\longrightarrow$ 3(4$CaO.Al_2O3$ $SO_3.12H_2O$)

3 $CaO.Al_2O_3$ +$CaO.H_2O$ + 12 H_2O $\longrightarrow$ 4$CaO.Al_2O_3.13H_2O$

Portlandite ($Ca(OH)_2$) is one of the hydration products of the clinker phases. It has a large, hexagonal prism morphology and occupies the 15-25% of the solid volume in the hydrated cement paste it has a much lower surface area than C-S-H and does not contribute much to strength. It is the responsible for keeping the pore solution alkaline (pH= 12-13).

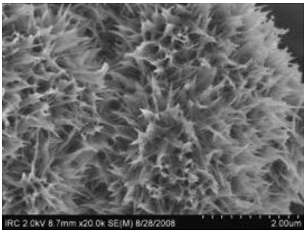

Figure 3: *SEM image of C-S-H.* [9]

Figure 4: *SEM image of Portlandite.* [9]

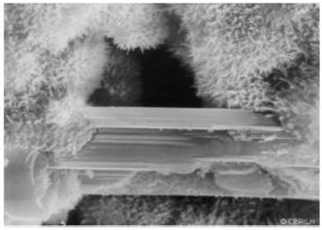

Figure 5: *SEM image of Portlandite and C-S-H.* [9]

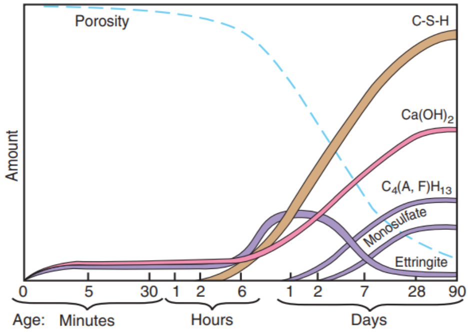

Figure 6: *Hydration rates of the clinker phases.* [8]

3.2.6 Ternary phase diagrams

The CaO-Al_2O_3-SiO_2 system serves to describe the composition of ceramic materials. In its SiO_2 poor part the phase relations and transformations in Portland and aluminate cements; in its SiO_2 rich/intermediate CaO part earthenware pottery including bricks and stoneware; and its Al_2O_3-rich part ceramics with refractory properties. These diagrams, help situating the materials an attending on the region of the triangle they are, the materials will show different properties.

Hydraulicity is the capacity of cements to set and harden when reacting with water. As the CaO amount increases, hydraulicity increases. Therefore, limestone and Portland cement are more hydraulic than fly ashes.

Pozzolanicity is the capacity of a material to react with calcium hydroxide in order to form hydraulic compounds similar to those generated at the cement hydration. As the SiO_2, increases the pozzolanicity increases also.

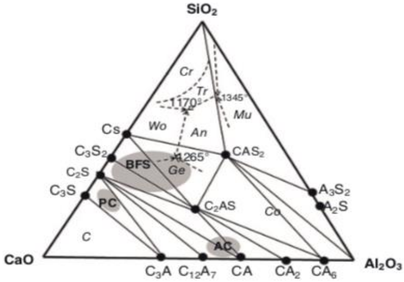

Figure 7: *Ternary phase diagram. PC (Portland Cement); BFS (Blast Furnace Slag); AC (Aluminate Cement).* [(6)]

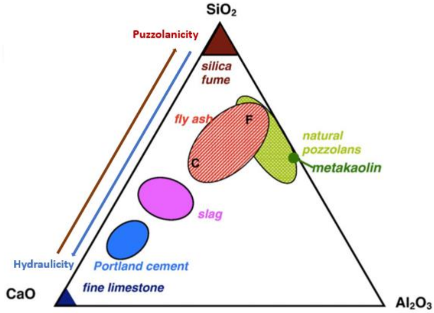

Figure 8: *Ternary phase diagram.* [(9)]

3.3. MORTARS

Mortar is a mass consisting of a material with binding capacity, an aggregate and water. Mortars usually contain a mixture of cement and sand in a ratio of (1:3). In addition, additives (substances in a dosage of less than 5%) are normally added to the mixture in order to modify certain properties of the material. Thus, by varying the components and proportions, mortars with different properties and characteristics can be obtained.

3.3.1 Mortar classification

Mortars are classified attending the characteristics of its individual components, the proportion in the formulation and the final use that is going to be given to the material. Mortars are classified attending the UNE-EN 998-1 regulation which classifies the mortars attending its function, its binder capacity and its properties or application field.

Mortar classification attending its function	
Industrial mortar	Unites the different elements of a factory (bricks, rocks…)
Coating mortar	Provides finish and protection.
Decoration mortars	Used for ornamental wall finishing.
Special mortars	Tile adhesive mortars, waterproof mortars repair mortars…

Table 4: *Mortar classification attending its function.* [10]

Mortar classification attending its binding capacity	
Mud mortar	Contains clay soils and water.
Cement mortar	Mixture of cement, sand and water.
Gypsum mortar	High temperature gypsum mixture.
Bastard mortar	Binder based on a mixture of lime and gypsum or lime and cement, sand and water.
Lime mortar	Mixture of lime, sand and water.

Table 5: *Mortar classification attending its binding capacity.* [10]

3.3.2 Components of a mortar

3.3.2.1 Binder

A binder is a material which reacts to give consistency to the compounds. The main tasks of the binder are to form a firm bond within the background and the tile and that's called adhesion; and to bind aggregates together (cohesion).

There are hydraulic binders: a material which sets and hardens when mixed with a convenient amount of water. Some hydraulic binders are: cement and hydraulic lime.

There are aerial binders: A material which sets and hardens when exposed to air (CO_2). Some aerial binders are gypsum and aerial lime.

Property	Mechanical resistance	Setting time	Workability	Adherence
↑	Cement	Gypsum	Aerial lime	Aerial lime
	Hydraulic lime	Cement	Gypsum	Gypsum
	Aerial lime	Hydraulic lime	Hydraulic lime	Hydraulic lime
	Gypsum	Aerial lime	Cement	Cement

Table 6: *Binder classification attending its physical properties.* [2]

3.3.2.2 Aggregates

Aggregates are granulated inorganic materials which interact with the binder and add strength to the mortar. They provide packing density, flexural strength and durability.

The aggregate is the component with a higher proportion in the mortar formula. Hence the type and quality of the aggregate must be chosen properly attending the chemical composition and the physical properties of the aggregate. The aggregate can be siliceous, calcareous, clay or ceramic and there's a wide classification of the aggregates that can be used in a mortar at regulation UNE-EN 13139.

3.3.2.3 Water

The effect of the water will have on the mortar it depends upon the type of ligand. The mortar mixing water gives the workability, the plasticity and the homogeneity needed. Nevertheless, if excessive water is added it can be harmful for the mortar. When the water evaporates, pinholes can be seen and the mechanical resistance of the cement is decreased.

Any clean, salt less water can be used for preparing mortars. If any high concentration of salt is present in the water, white crystals in the surface of the mortar can be seen and so efflorescence takes place.

3.3.2.4 Chemical additives

Chemical additives are substances added in a proportion below the 5%. They give the unique properties to the mortar.

Additive classification
Plasticizers: incorporated to increase the flexibility, softness and fluidity of the mortar. Most used plasticizers are: polycarboxylates and melamines.
Defoamers: used to reduce the entrainment of air when mixing the mortar with water. Good defoarmers are: polyalkylimide glycol and polysiloxane.
Accelerators: used to speed the rate of setting and/ or the early strength of tile adhesives. The most commonly used accelerators are: Calcium chloride, calcium formiate or lithium carbonate. The typical dosage for these additives is 0.5%.
Water retention agents: used to retain water in the mortar for a longer period of time so there is enough water for the cement to hydrate. The most used water retention agents are chemically modified methyl cellulose.
Retarders: used to slow the rate of the cement hydration and prolong the opening time of the tile adhesive. Common retarders are sodium salts of fruit acids α-hydroxycarboxylic acids (citric or tartaric acids). The usual dosification for these retarders is not more than 0.25%.

Table 7: *Additive classification attending UNE-EN 934-2 regulation.* (12)

3.3.2.5 Mortar properties

The most important properties for the mortars can be divided into the ones belonging at the mortar in the fresh state (workability and deformation under small stress) and those belonging into the harden state (mechanical resistance).

- Fresh mortar properties: adherence, density, consistency, workability period, capacity for water reduction, occluded air.
- Dry mortar properties: adherence, density, shrinkage, porosity, water vapor permeability, water permeability, mechanical resistance, thermal behavior, capillary water absorption.

3.4. MORTAR CODIFICATION

Regarding to tile adhesive mortars, the regulation UNE-EN 12004 regulates and guarantees de quality of their specifications. This codification has a letter at the beginning (C, D or R) a number (1 or 2) and a letter at the end (F, T or E). So, for example a C1TE tile adhesive is one that is a cementitious adhesive, with adherence after testing the tile adhesive in water, in heat and the initial time giving an adherence of 0,5 N/mm2. T indicates that it has no slipping and E indicating a prolonged open time.

Figure 9: *Mortar codification regarding UNE-EN 12004.* (13)

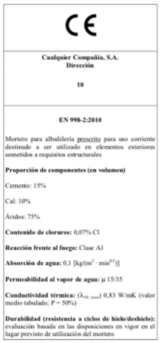

Figure 10*: Example of the tag mortars should have according to the CE mark.* (15)

CE mark

Cement is one of the principal construction products. It is considered a critical product because affects directly to the security of buildings, roads, bridges and every element of construction where cement is used. Therefore, in the European Union all the fabricants of cement products need to be accredited with the CE mark. The manufacturer or his authorized representative is responsible for fixing the CE marking and must be in conformity with the European Directive 93/68/EC and must be shown on the packaging, on the label or the accompanying commercial documentation. (15)

3.5. SUSTAINABLE DEVELOPMENT

Industrial by-products are wastes from industries that generate throughout their industrial processes and afterwards are thrown away and are no longer used.

The reuse of these industrial by-products in mortar production offers many benefits such as: environmental (by diminution of natural resource mining, prevention of disposal problems, energy saving and reduction of carbon dioxide emissions) and economic, because industrial by-products may be low-cost materials and they can be used to replace higher-cost materials. (21)

In the images below three different by-products are going to be replaced in the mortars: siliceous sand is going to be replaced by sand coming from PAM industries, grey cement for the FCC (Fluid Catalyst Cracking) from BP oil industries and white cement for biomass ashes coming from SAICA group.

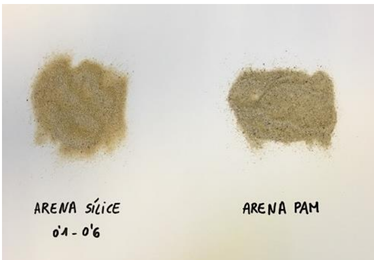

Figure 11: *Comparing siliceous sand and PAM sand (byproduct).*

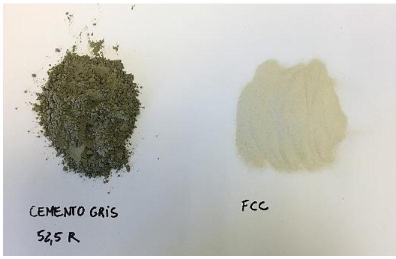

Figure 12: *Fluid Catalytic Cracking from BP oil industries and OPC 52,5 R.*

Figure 13: *Biomass ashes from Saica and OPC 42,5 R.*

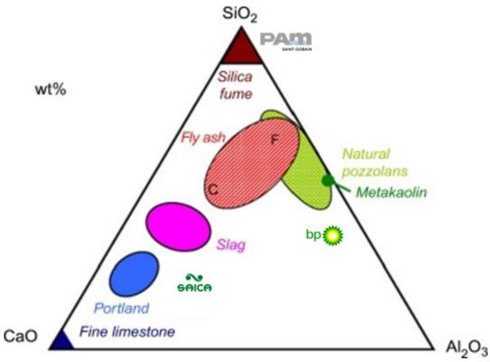

Figure 14: *Ternary diagram system placing the studied by-products. (PAM. BP oil and Saica).*

3.5.1 Sand alternatives

Foundry sands coming from the manufacturing processes of foundry elements in which molds of this material are used to shape the morphology of the different pieces, have a mostly siliceous composition.

These sands are surplus in the foundries of F. Ederlan since, for its process, it is necessary the continuous introduction of new sand to the circuit for the modeling of the cores that form the piece, and when it is removed from the circuit, it becomes the residue.

There are two types of foundry sands attending to the binder used in the foundry process. There are green foundry sands and chemical foundry sands. Green foundry sands are the most common, having siliceous sand (85%), bentonite (5-10%) and carbonaceous additive (2-10%) which it gives a dark color to the sand.

Chemical foundry sands have a lighter color, a higher siliceous sand content (93-99%), a chemical binder (1-3%) and a catalyst that endures the foundry mold. There are a wide variety of chemical binders, epoxy resins, furfyl alcohol and sodium silicates.

PAM is an iron piping system company belonging to the Saint Gobain group. PAM uses silica sand in the process of fabricating piping tubes molds. It is a chemical foundry sand and has a light color.

3.5.2 Fluid Catalytic Cracking (FCC)

Fluid catalytic cracking is generated as a waste product in the petroleum refinery, specifically in the fluidized bed catalytic cracking process. The FCC studied in this project comes from BP oil industries form Castellon (Spain). The FCC is going to replace the cement binder partially. This by-product consists of a SiO_2-Al_2O_3 zeolitic structure which reacts with hydrated lime for yielding; mainly calcium silicoaluminate hydrates. (12)

3.5.3 Biomass ashes

Biomass ashes are studied in order to replace this demand on the market. Biomass ashes come from the combustion of organic materials such as wood and plants.

The biomass ash selected for this project are biomass ashes coming from *Saica* which is a paper production plant which uses the combustion of wood as an energy source.

Biomass ashes is an heterogenous powder mainly composed of clusters of aggregated particles mixed with grains of quartz sand. The amorphous phases are enriched with CaO and therefore, this material exhibits a hydraulic behavior. (23)

4. OBJECTIVES

The objective of this project is to replace the raw materials of the mortar with the provided by-products coming from PAM industries, Saica and BP oil. The main objectives are:

-Characterize the physical and chemical parameters of the raw materials to be replaced in the mortar and see if they are likely to the ones in the reference product.

-Perform the adhesion tests on the new formulated mortars and see if they pass the UNE-EN 12004 regulation.

5. EXPERIMENTAL SECTION

5.1. MATERIALS AND METHODS

When willing to examine a new tile adhesive formula that is the result of a replacement of a raw material for a recycled by-product, the following method is followed:

Mortar base needs to be prepared attending to the proportions required in the mortar formula. Around 2 kg of mortar base is needed to perform a test adhesion.

In the table below there's an example of how we would proceed for preparing, a replacement of the ten and twenty percent of cement by a recycled by-product.

To assure homogeneity and to lower the error when weighting, a big base is prepared. This "big base" will contain the common proportion of all the three mortar bases. Afterwards, the "big base" will be separated into three smaller bases and the remaining cement or recycled by-product will be added.

In the example of the table, we would prepare a big base containing the common cement for all the three bases, the common sand, limestone and the common additives.

The big base is mixed in a V-shaped mixer for about 20 minutes and then divided into three.

	Reference	10% Cement replacement	20% Cement replacement	6 Kg Big Base	
OPC 42,5 R	25%	22,50%	20%	20%	1,2 Kg
Replacing by-product		2,50%	5%	0%	0 kg
Siliceous sand 0,1-0,6	55%	55%	55%	55%	3,3 Kg
Limestone	19%	19%	19%	19%	1,14 Kg
Additives	1%	1%	1%	1%	0,06 Kg
Total	**100%**	**100%**	**100%**	**95%**	**5,7 kg**

Table 8: *Calculations made when preparing a mortar base for testing.*

Afterwards, is carefully taken out from the mixer. Three clean and resistant plastic bags are named and tagged and the big base is divided in these three bags: reference, 10% cement replacement, 20% cement replacement. Then, the convenient additions are made to reach the 2 kg in each bag. Each bag is finally placed again into the V-shaped mixer to assure everything is well-mixed.

Figure 15: *Mixer for mixing testing mortar bases.*

In order to determine the convenient amount of water that the mortar will need, around 200 grams are weighted in a small plastic bowl. With a flash water bottle water is added carefully until we the mortar has the texture desired and it is the closest to the reference product.

Figure 16: *Mortar before being mixed.*

Figure 17: *Flashing water bottle and mortar to determine the convenient amount of water.*

Figure 18: *Fresh mortar mixed with the convenient amount of water.*

5.2. ADHERENCE PERFORMANCE TESTS

Adhesions tests have been performed to test the adherence of every new formulated tile adhesive formula. Adherence of a tile adhesive is defined as the force per unit of surface area that is needed to detach a tile adhesive mortar from the substrate, when the mortar is dried. It is mandatory that the new formulated products performance on adherence tests are those described at UNE 12004-02 in order to satisfy the requirements for the CE marking of the mortar.

The result is expressed within [N/mm2] attending to:

$$R = \frac{C}{S}$$

R= resistance to traction

C= applied strength [N]

S= tile Surface [mm2] (50 mm* 50 mm)

The first thing to do is to prepare all the material needed.

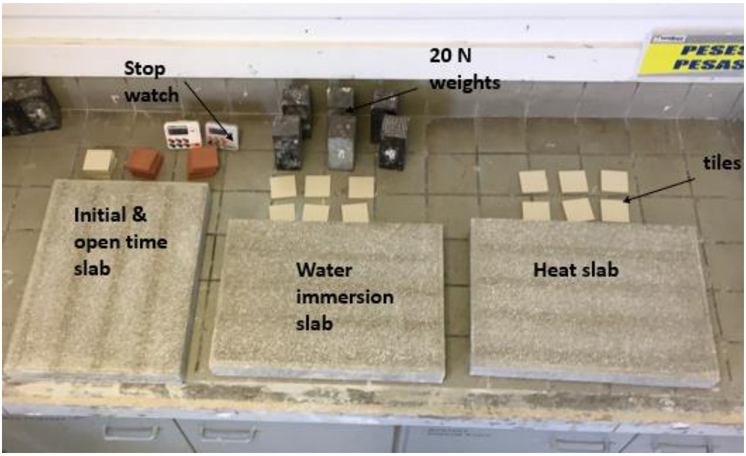

Figure 19: *Preparation of the material before starting the test.*

It is important everything is well-prepared before mixing the mortar with water. Remind that once water is added, hydration of the cement starts and therefore, the results on adherence would not be the same. Time is important when testing mortars.

To proceed around 1,8 kg are weighted and the convenient amount of water previously determined is added. Once the water is added, it is mixed with the time and velocities specified in the: *ME.LB.012. Amasado de un mortero.*

After the mortar is mixed with the convenient amount of water and reglementary time and speed, the timer is set and the application of the product starts on the slab helping yourself with the toothed trowel. After, apply a second thicker layer keeping a 60º angle with the trowel respect to the support. After five minutes of the application has finished on all the slabs, stick the six tiles on top of the application and put the 20 N weights on top of the tiles for 30 seconds. After the thirty seconds, the weights are removed.

After twenty minutes from the start of the application, six tile adhesives are stick on the first slab (initial adherence and open time adherence), and proceeding exactly the same way by putting on top the 20 N weights for 30 seconds on each tile. After the thirty seconds, the weights are removed.

After thirty minutes from the start of the application, six tile adhesives are stick on the first slab (initial adherence and open time adherence), and proceeding exactly the same way by putting on top the 20 N weights for 30 seconds on each tile. After the thirty seconds, the weights are removed.

The three slabs are immediately storage at the cure chamber. Seven days later after the test was performed, the slab that is going to be tested under water conditions it is immersed in water. The slab is going to be immersed for the remaining 21 days.

Figure 20: *Cured chamber where slabs dry.*

Figure 21 *Water immersion tub.*

Also, early adherence is tested seven days after the test. The slab containing the initial and open time tiles, it is taken out from the cure chamber and 6 sufferers (see the image below) are stick with epoxy glue. Six sufferers because: two corresponding on the initial adherence, two corresponding at open time at 20 minutes, and two corresponding to the open time at 30 minutes.

The next day after sticking the epoxy sufferers, the tile adhesives are pulled with the pulling machine and hence, the initial adherence it is determined.

Initial adherence it gives an idea of how the mortar is developing its properties, even though the one to look for is the adherence at 28 days, initial adherence gives an approximate idea without having to wait until the 28 days. So, it's kind of a check point.

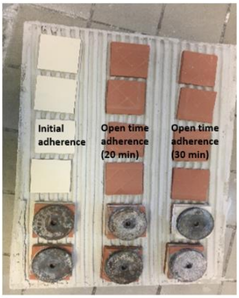

Figure 22: *Slab with six sticked sufferers ready to be pulled up for early adherence (7 days).*

Figure 23: *Pulling machine ready to pull up sufferers.).*

Figure 24: *Top view of the slab and the pulling machine.*

After tiles are pulled with the pulling machine and early adherence results have been recorded, the slab is stored again in the curing chamber three more weeks until the twenty-eight days.

Fourteen days after the application test, the slab that is going to be tested under heat conditions it is placed in the oven at 70 ºC. It will remain inside the oven for the remaining 14 days.

Twenty-eight days after the test, the water immersed slab and the slab inside the oven are taken out. Also, the initial and open time adherence slab is taken out from the cured chamber. Sufferers are stick on every tile adhesive from every slab and the next day every tile is going to be pulled up with the pulling machine. Results on initial adherence, open time (twenty and thirty minutes), water immersion and heat conditions adherence are obtained.

Figure 25: *Oven at 70ºC where slabs will remain during 14 days.*

5.3 SETTING TIME OF A MORTAR

Setting time of a mortar is the time that happens from the mortar is fresh until the mortar is hardened. Initial setting time (IST) or initial set is the moment when the fresh mortar starts hardening. The final set or final setting time (FST) is the time required for the paste to acquire a certain degree of hardness.

The method consists of placing the fresh mortar inside a mold that is 4 cm deep. When the mortar is fresh the needle is able to penetrate all the way down through the 4 cm deep of the mold. Nevertheless, when the mortar starts hardening, the needle is not able to penetrate all the way down to the 4 cm high that is the mold. When the mortar is completely hardened, the needle won't even be able to get in the mortar paste, and therefore, this point will determine the final setting time. Normally, the needle will measure between the defined intervals of time that the operator sets on the VICAT program. A graphic is generated indicating the time (x-axis) and the penetration of the VICAT needle (y-axis). To proceed, the mold is filled until the top with the fresh mortar that is left for the adherence test. From the 2 kg base made previously for the adherence, it will probably be enough mortar left for the setting time test. About 350 gr of the fresh mortar are placed in to the round shaped mold. Once the mold is filled and trimmed to the surface, the mold is placed in one of the eight spots that the VICAT apparatus has.

Figure 26: *Round-shaped mold for the setting time test.*

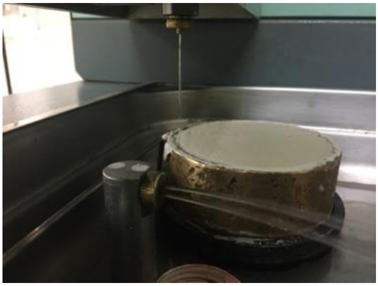

Figure 27: *Round-shaped mold filled with testing mortar.*

Before the start of the test, several parameters are introduced into the *ACMEL labo primsometre* program:

• Initial setting time: because we are working with tile adhesive mortar type C1, the approximate initial setting time should be around 10 hours. This means, that up until 10 hours the fresh mortar won't start hardening.

• Slow period measures: it is set the time between measures during the slow period. The slow period is the period before the mortar starts hardening. The interval time of the slow period is usually set around 10-15 minutes between measures.

• Rapid period: it is set the time between stings during the rapid period. The rapid period is the period right after the mortar starts hardening until the moment the mortar has hardened completely. The interval time of the rapid period is usually set around 5 minutes between measures.

Therefore, start of the setting time is determined when the VICAT needle penetrates the mold, and because, the mortar is still fresh the needle can penetrate all the way down through the 4cm mold. When the mortar starts hardening, the needle can't penetrate all the way down through the mold and that's reflected on the graph. When the mortar is completely hardened, the needle can't penetrate through the mold anymore and that's the determining factor of the final setting time.

In the graph below, there's an example for the setting time curve of mortar type C1TE. At the beginning, because the mortar is fresh, the needle can penetrate all the way down through the mold. When the mortar starts hardening, the needle can penetrate all the way down to the mold regardless from one millimeter and hence, that's the initial of the setting time. Upon this point, the space between measurements is shorten and measures are made more often to measure more accurately the final setting time. The final setting time will be determined when the needle can't penetrate any way down to the mold and it stops there, at 4mm which is the length of the mold.

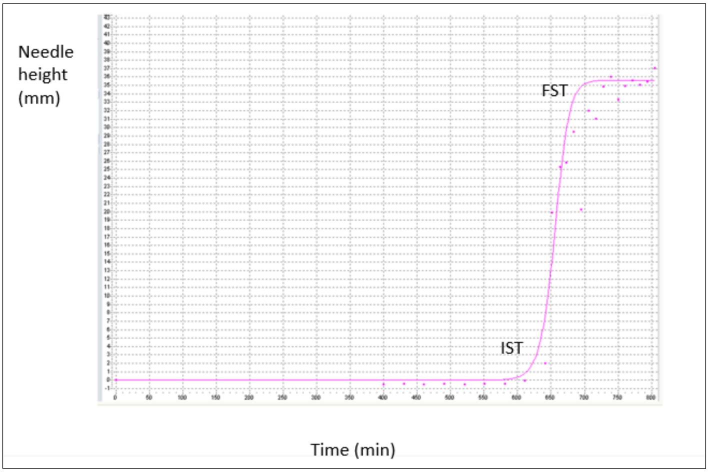

Figure 28: *Example graphic for the setting time of a mortar.*

5.4 SHRINKAGE AND LOSS OF WEIGHT

Shrinkage is the shortening that mortar undergoes during the hardening and drying process. It is mainly due to the evaporation loss of excess mixing water. To proceed, the same procedure of preparing the mortar bases is done as explained previously. Once the mortar is mixed with the convenient amount of water, is placed in a standardized 4x4x16 cm shaped mold. Afterwards, the mold is shaken and leveled.

Figure 29: *Standardized 4x4x16 cm molds for testing specimens.*

Figure 30: *Vibrating instruments for levelling specimens.*

The specimens are dried in air for 24 h and then taken out from the mold the next day. The molds have the particularity of containing screws with nuts on both sides of the cylinder and so, when the mortar dries (after the 24 h), screws are unscrewed and the nut remains in the mortar.

After the 24h the specimens are afterwards weighted and measured.

Figure 31: *Testing specimen with nut.*

Figure 32: *Shrinkage measure.*

These measures are repeated every 7 days until it reaches the 28 days when is considered that the mortar has developed all their mechanical properties. With comparing the initial weight to the final weight at 28 days and the length of the specimen before and after the 28 days, shrinkage and loss of weight can be calculated.

To proceed, the apparatus is calibrated with an iron calibration bar which is 16 cm, as well as the prepared testing specimen. When measuring, what is actually being measured is the difference less that differs from the original 16 cm. Every week up to four weeks measures are made and the following calculation is made:

Shrinkage = ((A-B) mm/(0,16(m))

Where A = measure at week n; B= measure at 24 h

The result is expressed as the millimeters that the mortar has retracted per meter of mortar.

The weight loss is also registered:

% Weight loss = ((C-D))/(D)) *100

Where C= initial weight of the specimen; D= final weight of the specimen

5.5. COMPRESSION AND FLEXURAL STRENGTH

Flexure is the capacity of a material to resist to perpendicular forces applied through its longitudinal axis. Compression is the effort that a material needs to do under a crushing force. The compression and flexural strength were evaluated according to UNE-EN 1015-11.

To proceed, specimens are prepared in the same way as for the shrinkage and loss of weight tests. Seven days after the specimens have been prepared, early flexure and compression are tested. Twenty-eight days after the specimens have been prepared the test is repeated. The first measure is a checking point to have an early result and make up an idea of the mechanical properties of the tested mortar.

Figure 33: *Flexural and compression strength machine.*

5.5.1 Flexural strength

To measure the flexural strength, the machine must be equipped with two steel support rollers, with a length between 45 mm and 50 mm and a diameter of 10 mm ± 0,5 mm, spaced 100,0 mm ± 0,5 mm apart and a third load roller of the same length and diameter installed centrally between the support rollers.

To test the flexure strength, the specimen is placed underneath the flexural canal. One of the faces of the specimen (which has been in contact with the mold walls during molding) is placed on the support rollers. The load is applied without abrupt accelerations, at a uniform speed between 10 N/s and 50 N/s, at a speed between 30 seconds and 90 seconds.

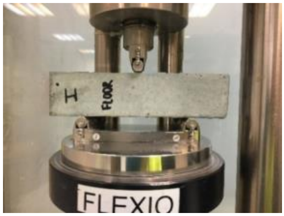

Figure 34: *Flexural strength measurement.*

5.5.2 Compression strength

The compression test is done right after the flexion test. At the flexure test the specimen is broken into two parts. Each resulting part is tested under compression.

To proceed, the specimen is carefully aligned so that the load is applied over the entire width of the faces in contact with the plates. The load is applied without abrupt accelerations, at a uniform speed between 50 N/s and 500 N/s, at a speed between 30 seconds and 90 seconds. The maximum load applied, in N, during the test is recorded as follows.

The resistance is calculated by dividing the maximum load supported by the specimen by its cross section.

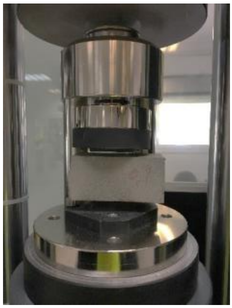

Figure 35: *Flexural strength measurement.*

6. CHARACTERIZATION AND REACTIVITY EVALUATION

In the table below, the characterization parameters for each by-product is determined:

Characterization parameters	PAM sand	FCC	Biomass ashes
Physical properties	Particle size distribution (Sieve analysis)	Particle size distribution (Laser granulometry)	Particle size distribution (Laser granulometry)
Chemical properties	Chemical composition (XRF)	Chemical composition (XRF)	Chemical composition (XRF)
Mineral properties	Mineral composition (XRD)	Mineral composition (XRD)	

Table 9: *Characterization parameters of the different studied by-products.*

6.1. PHYSICAL PROPERTIES

As for the physical properties, particle size distribution of a powder or a granular material defines the relative number of particles present according to their size. The smaller the particle, the larger the specific surface and hence, the higher the reactivity.

6.1.1 Sieve analysis

Sieve analysis is used to determine the particle size distribution of a granular material by allowing the material to pass through a series of sieves of progressively smaller mesh size. To proceed, 100 gr of the powder are weighted and placed on top of the sieve column.

Figure 36: *Sieve column.*

6.1.2 Laser granulometry

Laser granulometry is a diffraction measure that measures particle size distributions by measuring the angular variation in intensity of light scattered as a laser beam passes through a dispersed particulate sample. Large particles scatter light at small angles relative to the laser beam and small particles scatter light at large angles. The angular scattering intensity data is then analyzed to calculate the size of the particles responsible for creating the scattering pattern. To proceed, about 200 grams are introduced in the vacuum hole and laser rays start analyzing the sample while its being vacuumed. It takes around 10 minutes until all the sample is vacuumed. Immediately after, a graphic of the particle size distribution of the sample is immediately generated.

Figure 37: *Laser granulometry machine.*

Figure 38: *Vacuum.*

6.2. CHEMICAL PROPERTIES

6.2.1 X-RAY FLUORESCENCE (XRF)

In order to determine the chemical composition of the PAM sand, the FCC and the biomass ashes a sample is summited to XRF (X-Ray Fluorescence). X-Ray Fluorescence is an analytical technique which thorough exciting the closest electron of the nuclei, this electron is promoted to the next orbital. This transition releases energy and this energy is directly proportional to the amount of the element within our sample. XRF is an element quantification technique. In this project, samples have been sent to Saint Gobain Research (Paris) and XRF patterns are already associated with the elements with its oxide form.

6.6.2 X-RAY DIFFRACTION (XRD)

The objective of X-Ray Diffraction method is used to analyze the mineral phases from the crystal structure. X- ray powder diffraction technique is based on the concept that when a powder is subjected with X-rays, a diffraction pattern is obtained. Each diffraction pattern is characteristic to a particular crystal lattice energy and in a mixture, this crystal lattice gives rise to their individual pattern, independent of one another. The magnitude of peak intensities is a direct indicator of their crystalline nature. The amorphous compound, shows no diffraction pattern.

7. RESULTS ON PAM SAND

Adhesive formulas were prepared in order to carry out adhesion tests in various conditions. In the table below, formulas replacing siliceous sand 0,1-0,6mm by PAM sand substituting 50% siliceous sand and 100% siliceous sand.

	Reference	50 % PAM sand	100% PAM sand
OPC 52,5 R	25%	22,50%	20%
PAM sand	0%	22,50%	55%
Siliceous sand 0,1-0,6	55%	55%	0%
Limestone	19%	19%	19%
Additives	1%	1%	1%

Table 10: *Tile adhesive formulas for testing PAM sand.*

Adherence test

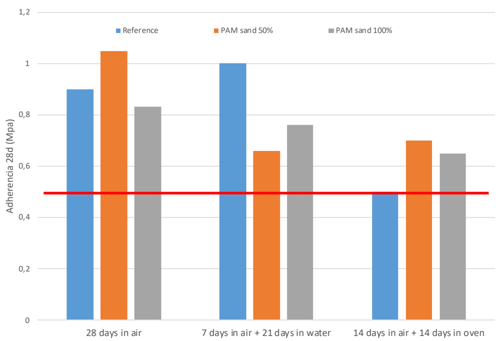

Figure 39: *Results on adhesive tests for PAM substitution. The red line is the adherence required for the UNE-EN 12004.*

In the graphic above, the results on the adhesions tests are shown. The red line indicates the limit regarding adherence that is required by the UNE-EN 14002 regulation.

Seeing the results, with a cure in air, 28 days after the start of hydration, the adhesion of the mortars containing 50% and 100% replacement with PAM's sand shown results among above the required adherence.

With a cure in air during 7 days and then in water during 21 days, the adhesion results for mortars containing replacements of 50% and 100% of the sand are both above the required adherence and the same is happening with a cure in air during 14 days and then in oven at

70ºC during 14 days, the results for the mortars with the replacements of 50% and 100% with PAM's sand are above the required value.

Mechanical properties and setting time

	Flexion (7 days) (Mpa)	Flexion 28 days (Mpa)	Compression 7days (Mpa)	Compression 28 days (Mpa)	Shrinkage (mm/m)	% weight loss
Reference	4,93	4,6	28,1	25,3	0,84	5,11
100% sand substitution	4,63	5,53	32,8	32,2	0,77	4,63

Table 11: *Results on compression and flexion tests.*

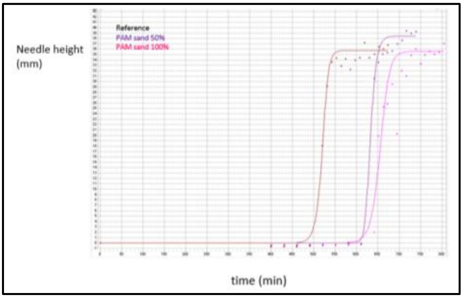

Figure 40: *Setting time for reference sand and replacements of 50% and 100%.*

Regarding the mechanical properties when replacing the reference sand by PAM's sand, there's no big change regarding the flexion or compression strength.

As for the setting time, when replacing the siliceous sand of the mortar by the PAM sand, the initial setting time of the mortar increases by an hour and a half but the time between the initial set and the final set is the same.

Physical and chemical properties

SiO_2	CaO	Al_2O_3	MgO	TiO_2	Fe_2O_3	K_2O	Na_2O	P_2O_5	MnO	ZrO_2
99,4	0,03	0,23	0,02	0,06	0,135	0,02	0,01	0,001	0,005	0,01

Table 12: *Chemical composition of PAM sand (XRF).*

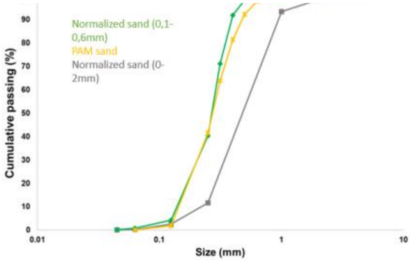

Figure 41 *Granulometry curve of PAM sand comparing to natural sands.*

The particle size distribution for the PAM sand its very close to the normalized sand (0,1-0,6 mm). Particle size distribution of a replacing sand is a very important factor since the sand in a mortar acts as the "skeleton" of the mortar. The granulometry needs to be compensated which means that is important that there's the same amount of smaller and bigger particles so that there's no interstitial holes. Hence, the structure of the mortar is compact and there's no place for the atmosphere air to get in the holes and therefore, the mortar won't show significant shrinkage.

As for the chemical properties, the 99,4 % of the sand is SiO_2 and as seen in the XRD, this silica is present in its crystalline form. Silica has three main crystalline varieties: quartz, tridymite and cristobalite. When testing the sample under X-Ray Diffraction, the sample shows up very well-defined peaks. The crystalline phases were identified by X-Ray Diffraction method, the peaks correspond to this of the solid crystalline structure of quartz.

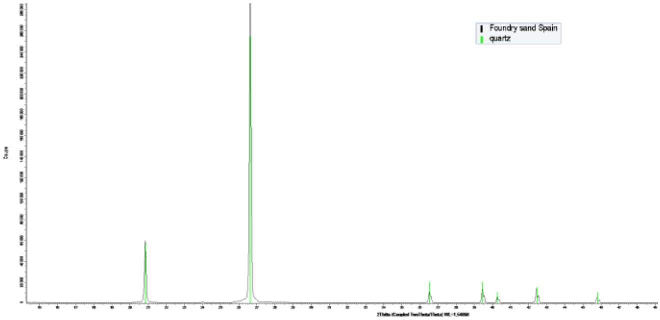

***Figure 42**: XRD peaks for PAM sand. Peaks correspond to crystalline structure of quartz.*

8. RESULTS ON FCC

Tile adhesive formulas were prepared in order to carry out adhesion tests in various conditions. Attending to the reference base containing an amount of 25% of cement this 25% has been substituted by 10% of FCC and 20% of FCC.

	Reference	10% FCC	20% FCC
OPC 52,5 R	25%	22,50%	20%
FCC OIL	0%	2,50%	5%
Siliceous sand 0,1-0,6	55%	55%	55%
Limestone	19%	19%	19%
Additives	1%	1%	1%

***Table 13:** Tile adhesive formulas for study FCC.*

Adherence tests

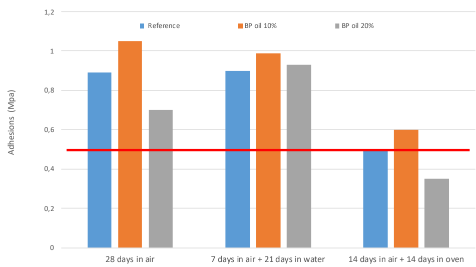

***Figure 43:** Results on adherence tests with FCC substitution. The red line indicates the required value attending to the regulation UNE-EN 12004.*

In the table above, results on adhesions are shown. The red line, with indicates an adhesion of 0,5 MPa which is required by the UNE-EN 12004 regulation.

In air, 28 days after the start of hydration, the adhesion of the mortars containing 10% and 20% FCC oil show good performances in adhesion tests.

With a cure in air during 7 days after the start of hydration and then in water during 21 days, the adhesion results for mortars containing 10% and 20% FCC oil were even higher than the reference mortar.

With a cure in air during 7 days and then in oven at 70ºC during 14 days, the results for the mortar containing 10% FCC oil were higher than the reference mortar. The adhesion for the mortar containing 20% of FCC was lower than the required value

Mechanical properties and setting time

	Flexion (7 days) (Mpa)	Flexion 28 days (Mpa)	Compression 7days (Mpa)	Compression 28 days (Mpa)	Shrinkage (mm/m)	% weight loss
Reference	6	4,35	27,5	29,3	0,94	5,38%
10% FCC substitution	5,33	5,33	14,8	27,3	0,95	5,81%
20% FCC substitution	4,61	4	14,5	21,06	0,99	13,8 %

Table 14: *Results on mechanical properties for FCC.*

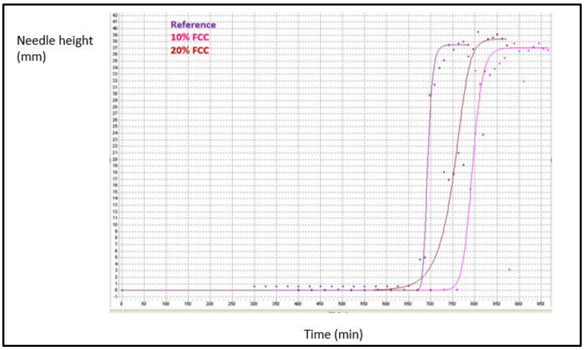

Figure 44: *Setting time for cement replacements with 10% and 20% FCC.*

Results on flexural and compression strength show that the replacement of the cement by FCC is acceptable because at 28 days flexion and compression are really close to the reference mortar.

Anyway, the replacement of 20% with FCC shows a weight loss of 13,8% and a higher shrinkage than the reference mortar.

FCC shows a high pozzolanic activity. The hydration reaction of the cement is slower because firstly, all the cement has to hydrate and when it's completed FCC starts reacting with the generated portlandite. Several studies have proved this throughout TGA. (20)

The setting time of the mortar hasn't been affected by FCC replacement.

Physical and chemical properties

Binder	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	Ti_2O_3	P_2O_5	Mn_2O	SrO
Cement	19.5	5.45	3.23	62.5	1.91	2.9	1.1	0	0.27	0.1	0.05	0.07
FCC	42.3	51.7	0.49	<0.1	0.01	0.18	0.04	0.29	1.04	0.19	0	0

Table 15: *Chemical composition of FCC comparing to the chemical composition of cement.*

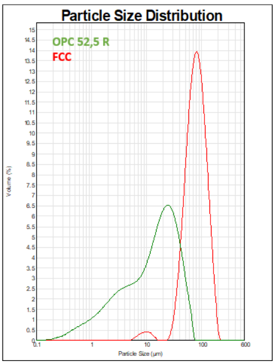

Figure 45: *Particle size distribution comparing FCC and OPC 52,5 R.*

As for the chemical composition it can be reaffirmed that FCC will show a pozzolanic behavior because of its high percentage of SiO_2 comparing to the OPC.

Also, the lower reactivity of the FCC comparing to the OPC can be justified because the particle size of the FCC is around 100 µm whereas the particle size distribution for the OPC is smaller (50 µm) and hence, the OPC will be more reactive. The smaller the particle, the higher the specific surface.

Anyway, the replacement of 20% with FCC shows a weight loss of 13,8% and a higher shrinkage than the reference mortar.

9.RESULTS ON BIOMASS ASHES

Tile adhesive formulas were prepared in order to carry out adhesion tests in various conditions. Attending to the reference base containing an amount of 25% of cement this 25% has been substituted by 50% of biomass ashes and it has been tested the different proportion of the water retention additive.

	Reference	**50% Biomass Ashes + 0,21 water retainer**	**50% Biomass Ashes + 0,18% water retainer**
OPC 42,5 R	25	12,50	12,5
Biomass Ashes		12,50	12,5
Siliceous sand 0,1-0,6	55	55	55
Limestone	19	19	19
Additives	1	1	1
Water retainer	0,21	0,21	0,18

Table 16: *tested tile adhesive formulas for testing BA.*

Adhesion tests

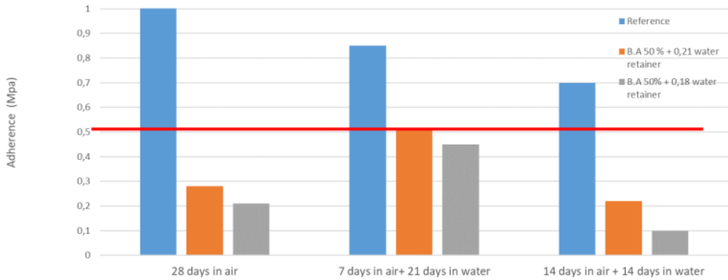

Figure 46: *Results on adherence tests with BA substitution. The red line indicates the required value attending to the regulation UNE-EN 12004.*

As the results in the table above show, when replacing the 50% of the cement mortar by biomass ashes, results on adhesion tests are much lower than the required value and this value is even lower when decreasing the water retention agent at 0,18% in formula.

The main purpose of a water retention agent is to retain water in the tile adhesive for a long period of time so that there is enough water available for the cement to hydrate. [23] As seen in the paper of SGR [22] that by reducing the content of the water retention agent would boost the reactivity of the biomass ashes did not actually work out with Saica's biomass ashes.

As seen in the laboratory, the BA from Saica are highly reactive and in order to boost the reactivity of the OPC: BA binder a retarder was used. Retarders slow the rate of the cement hydration. Highly active retarders are the sodium salts of fruit acids. [23]

The following mortar bases were prepared and tested using citric acid as a retarder.

	Reference	50% Biomass Ashes	50% Biomass Ashes
OPC 42,5 R	25%	11,25%	11,25%
Biomass Ashes		30,25%	30,25 %
Siliceous sand 0,1-0,6	55%	55%	55%
Limestone	19%	0%	0%
Additives	0,8%	0,6%	0,6%
Water retainer	0,21	0,24	0,21
Citric acid	0%	0,2	0,2

Table 17: *tested tile adhesive formulas for testing BA.*

Adhesion tests

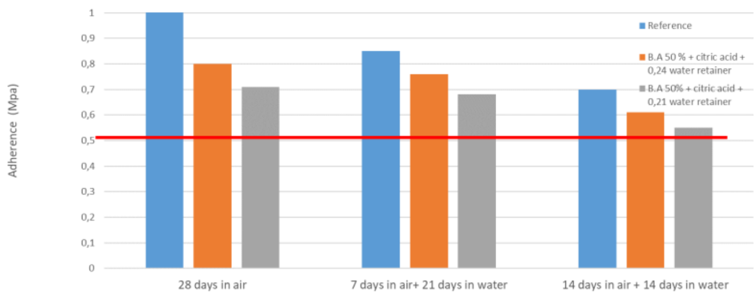

Figure 47: *Results on adherence tests with BA substitution. The red line indicates the required value attending to the regulation UNE-EN 12004.*

As the results show in the table above, when adding the citric acid to the mortar formula with BA, performances on adhesion tests are above the required value. Higher values on adhesion are shown when the water retention agent has been increased.

Mechanical properties

	Flexion (7 days) (Mpa)	Flexion 28 days (Mpa)	Compression 7days (Mpa)	Compression 28 days (Mpa)	Shrinkage (mm/m)	% weight loss
Reference	4,29	4,78	25,8	24,3	0,83	6,13
20% BA substitution	4,95	4,53	17,5	16	1,28	7,08
50% BA substitution	3,25	2,7	11,1	13,1	1,44	7,54

Table 18: *Mechanical properties on BA substitutions.*

As for the mechanical properties, when replacing the cement by the biomass ashes flexion and compression are lower than the reference mortar. The weight loss increases and the shrinkage as well.

Physical and chemical parameters

Binder	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	Ti_2O_3	P_2O_5	Mn_2O	SrO
OPC	19.5	5.45	3.23	62.5	1.91	2.9	1.1	0	0.27	0.1	0.05	0.07
Biomass ashes	15	10.3	3.41	44	1.75	1.26	3.05	1.02	0	1.54	0.46	0

Table 19: *Chemical composition of BA comparing to OPC (XRF).*

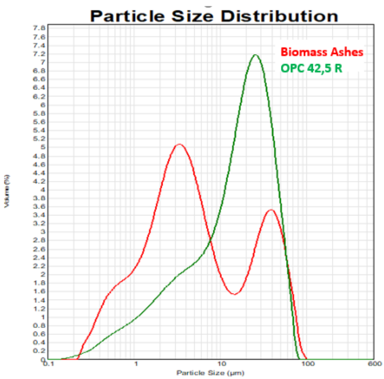

Figure 48: *Granulometry curve comparing Biomass ashes with OPC 42,5 R.*

As for the chemical properties, the BA have a high content of CaO and that explains their hydraulic behavior. The particle size distribution shows that the BA have to peaks where the particles are distributed. Most of the particles of the BA are below the 10 μm so that particles are thinner than cement, and hence the larger the specific surface and more reactive.

10. CONCLUSIONS

After all the replacements on the cement mortar have been studied by adhesions tests and characterization analysis, the following conclusions can be drawn:

As for the PAM sand replacements of 50% and 100% are both acceptable since the results on adhesions tests are the required ones by the UNE-EN 12004 regulation. Furthermore, as granulometry results show, the particle size distribution of the sand is very close to the reference sand. The characterization of the sample with the chemical characterization by XRF showed a high percentage of silica and XRD show that silica is mainly present in its crystalline quartz form.

For the replacement of the cementitious binder on the mortar by the FCC from BP oil industries it can be concluded that a replacement of the 10% of cement by FCC is acceptable as results in adherence are giving the required values. When coming up to a replacement of the 20% of the cement, the adherence tests are not giving the required values and the mortar base starts presenting issues regarding shrinkage and weight loss. That's because FCC showed a pozzolanic behavior which was supported by chemical analysis indicating a high percentage of silica and a very low percentage of CaO. This pozzolanic behavior made the rate of hydration reaction slow down.

Finally, when replacing the 50% of the cement of the mortar by biomass ashes the results on adherence did not meet the specified values for adherence. Nevertheless, when adding citric acid to the mortar formula, results on adhesions met the specified values.

Biomass ashes turned out being very reactive and showed a high hydration rate. This high reactivity can be explained because of the containing percentage of CaO of the BA. Adding a retardant (citric acid) helped slowing down the hydration reaction. Also, by increasing the amount of the water retainer boosted the reactivity of the mortar. After all these modifications, results on adhesion tests with the mortar containing biomass ashes met the specified values.

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12. ACRONYMS

OPC: Ordinary Portland Cement

FCC: Fluid Catalyst Cracking

FA: Fly Ashes

BA: Biomass Ashes

A: Al_2O_3

C: CaO

F: Fe_2O_3

H: H_2O

M: MgO

S: SiO2

Ŝ: SO3

C_3S: Tricalcium silicate (Alite)

C_2S: Dicalcium silicate (Belite)

C_3A: Tricalcium aluminate (Aluminate)

C_4AF: Tetracalcium aluminoferrite (Ferrite)

XRF: X-ray Fluorescence

XRD: X-ray Diffraction

SGR: Saint Gobain Research

TGA: Thermogravimetric analysis