# Biochar as eco-friendly filler to enhance the sustainable performance of cement



# **POLITECNICO DI TORINO**



## Department of architecture and design Master in Architecture for Sustainable Design

# Biochar as eco-friendly filler to enhance the sustainable performance of cement

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To my family

To students, researchers and those who face the new challenges of improving construction materials and making them more durable, sustainable and respectful with the environment

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# Sommario

La crescita della popolazione mondiale ha generato un aumento della domanda di costruzioni per soddisfare le esigenze abitative, di lavoro e di svago, comportando un maggiore consumo di materiali da costruzione. Al giorno d'oggi, però, è necessario che il processo di produzione di materiali tradizionali sia il più efficiente e sostenibile dal punto di vista ambientale ed economico.

Nel mondo delle costruzioni, il calcestruzzo è il materiale più prodotto e utilizzato al mondo. Uno dei suoi componenti principale è il cemento, la cui produzione negli ultimi anni ha raggiunto oltre 4 miliardi di tonnellate. Questo processo di produzione richiede grandi quantità di materia prima non rinnovabile e consuma un'elevata quantità di energia; inoltre, è altamente inquinante, essendo responsabile di oltre il 5% della CO<sub>2</sub> emessa nell'atmosfera ogni anno, generando gravi conseguenze ambientali.

Un altro grande problema irrisolto ad oggi è quello relativo alla gestione dei rifiuti organici (biomassa) che, a causa del loro basso tasso di riciclaggio, sono ormai un problema di primaria importanza per gli stati mondiali: il loro smaltimento causa seri problemi di contaminazione del suolo e dell'acqua, durante il processo di degradazione vengono generate emissioni di metano e altri gas serra, i costi di smaltimento e gestione sono elevati.

Recenti studi hanno dimostrato il potenziale energetico della biomassa trattata con processi termochimici, ad esempio quelli con ossigeno limitato come pirolisi o gassificazione, dai quali si ottengono bioolio, syngas e Biochar; i primi vengono usati come biocarburanti mentre il terzo, che è il sottoprodotto solido del processo, è stato utilizzato come ammendanti nei terreni. Tuttavia, diversi ricercatori nel campo dell'edilizia hanno verificato la potenziale applicazione del Biochar come inerte verde nelle paste cementizie, malte e calcestruzzi, migliorando le loro proprietà meccaniche come la resistenza alla compressione, alla flessione e la duttilità.

Quindi il presente lavoro si basa sull'utilizzo del Biochar "Borgotaro grigio", proveniente dal cippato di legno, come inerte per migliorare la resistenza meccanica nei materiali tradizionali a base di cemento. In aggiunta, lo scopo di questo lavoro è anche conoscere l'influenza del costo del Biochar aggiunto alla pasta cementizia.

Nell'attività di laboratorio è stato aggiunto il Biochar, ottenuto mediante gassificazione, in diverse percentuali rispetto al peso del cemento. Dai risultati finali è emerso che le micro-particelle di Biochar sono molto utili per migliorare le proprietà meccaniche dei composti cementizi. In particolare, l'aggiunta del 2% di Biochar ha mostrato un miglioramento della resistenza alla flessione, della tenacità e della duttilità poiché le particelle hanno la proprietà di deviare la frattura, generando una traiettoria molto più tortuosa e meno lineare del tipico cemento fragile, che si traduce in un aumento dell'energia di frattura. Per quanto riguarda la resistenza alla compressione, rispetto alla pasta cementizia pura, c'è stato un miglioramento di oltre il 5% a 7 giorni, tuttavia a 28 giorni non si è stato riscontrato un miglioramento di questa proprietà.

Per quanto riguarda il settore economico, è stato dimostrato che utilizzando il 2% di Biochar nell'impasto cementizio non si ha un aumento significativo del costo finale, ciò potrebbe aprire scenari interessanti riguardo la sua applicazione. Inoltre, sostituendo parte del cemento con la stessa quantità di Biochar (2%), si riesce a ottenere una miscela di circa 1% più economica rispetto all'impasto tradizionale a causa della minore quantità di cemento comportando anche un abbattimento delle emissioni di CO<sub>2</sub>.

In conclusione, con questo studio è stato possibile apprezzare come il Biochar abbia un grande potenziale come inerte nel cemento, con una riduzione dei costi in quanto si sostituisce il cemento con un materiale di scarto. In particolare, quest'ultima caratteristica implica un duplice vantaggio: da un lato, infatti, si ha un riuso di materiale altrimenti inutilizzabile; dall'altro lato, invece, abbiamo una riduzione dell'emissione dei gas serra nell'atmosfera sia per il minor uso di cemento, sia per la produzione stessa del Biochar che evita la generazione di quasi 900 kg di CO<sub>2</sub> equivalenti per ogni tonnellata di biomassa trattata.

# Summary

The growth of the world population has generated an increase in the demand for buildings to meet the needs of housing, work and leisure, leading to a greater consumption of building materials. However, nowadays, it is necessary that the process of producing traditional materials is the most efficient and sustainable from an environmental and economic point of view.

In the construction world, concrete is the most produced and used material in the world. One of its main components is cement, whose production has reached over 4 billion tons in recent years. This production process requires large quantities of non-renewable raw material and consumes a high amount of energy; moreover, it is highly polluting, being responsible for over 5% of  $CO_2$  emitted into the atmosphere each year, generating serious environmental consequences.

Another major problem unresolved to date is that related to the management of organic waste (biomass) which, due to their low recycling rate, are now a major problem for the world states: their disposal causes serious problems of contamination of the soil and water, during the degradation process, emissions of methane and other greenhouse gases are generated, the costs of disposal and management are high.

Recent studies have demonstrated the energy potential of biomass treated with thermochemical processes, for example those with limited oxygen such as pyrolysis or gasification, from which we obtain bio-oil, syngas and Biochar; the former are used as biofuels, while the third, which is the solid by-product of the process, has been used as soil improvers. However, several construction researchers have verified the potential application of Biochar as inert green in cement pastes, mortars and concretes, improving their mechanical properties such as compressive strength, flexural strength and ductility.

Therefore, the present work is based on the use of the "Borgotaro grigio" Biochar, coming from wood chips, as inert to improve the mechanical resistance in traditional cement-based materials. In addition, the purpose of this work is also to know the influence of the cost of Biochar dded to cement paste. In the laboratory activity was added the Biochar, obtained by gasification, in different percentages with respect to the weight of the cement. The final results showed that the micro-particles of Biochar are very useful for improving the mechanical properties of cement compounds. In particular, the addition of 2% of Biochar has shown an improvement in flexural strength, toughness and ductility since the particles have the property to divert the fracture, generating a much more tortuous and less linear trajectory of typical brittle cement, which results in an increase in fracture energy. As for the compressive strength, compared to pure cement paste, there was an improvement of more than 5% at 7 days, however at 28 days there was no improvement of this property.

Regarding the economic field, it has been shown that using 2% of Biochar in the cement mixture does not significantly increase the final cost, this could open interesting scenarios regarding its application. Moreover, by replacing part of the cement with the same amount of Biochar (2%), it is possible to obtain a mixture of almost 1% cheaper than the traditional mixture due to the lower amount of cement also leading to a reduction of  $CO_2$  emissions.

In conclusion, with this study it was possible to appreciate how the Biochar has a great potential as a filler in cement, with a reduction in costs as it replaces the cement with a waste material. In particular, this last feature implies a double advantage: on the one hand, in fact, there is a reuse of otherwise unusable material; on the other hand, we have a reduction in the emission of greenhouse gases into the atmosphere due to the reduced use of cement and the production of Biochar which avoids the generation of almost 900 kg of  $CO_2$  equivalent for each ton of biomass treated.

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# Introduction

#### **1.1 Problem statement**

The current worldwide environmental situation is as alarming as inevitable which repercussions are more than evident and are present at all scales. The real problem lies not only in the observed changes, but in how fast these are happening. The blame falls entirely on humans. Therefore, we are responsible for mitigating and eliminating as far as possible, the damage caused as a result from our behavior and actions.

Nowadays, rapid population growth, urbanization, and living standards have resulted in a massive generation of waste around the world. Only food losses accounted for more than 30% of the waste generated in the United States in 2010 [1]. In the Italian context , 17.7 million tons of food were thrown away, which translates into wasting the resources used in its production (such as land, water, energy, and inputs), generating unnecessary  $CO_2$  emissions and causing serious management problems and costs for the states [2].

On the other hand, climate changes force the construction industry to-rapidly implement innovations around the world. This automatically brings challenges to engineers, architects and other disciplines involved in construction to bet on measures and solutions that do not compromise the future of forthcoming generations, working towards sustainable development, while achieving building materials with better performance and environmental responsible at the same time.

Inevitably, only concrete can meet the demand for infrastructure and human habitats worldwide, due to the large global demand that makes it the construction material the most used. Although it can be considered an environmentally friendly material given its low embodied energy, the cement situation is completely different. Approximately, more than 4000 million tons of cement are produced annually worldwide, equivalent to more than 5% of the world's CO<sub>2</sub> emissions. Unfortunately, in the long term, there seems to be a tendency to increase [3].

Therefore, the main theme of this project is to improve the strength and tenacity in traditional cement-based materials by incorporating waste elements, introducing the use of Biochar -the by-product of the thermochemical process (pyrolysis or gasification) used for biomass treatment - in addition to the multiple benefits of its incorporation into the cement mixture and that will be detailed throughout this document, as a mitigation measure of climate change.

## **1.2 Objectives**

#### Main goal:

• Improve the performance of cement without harming the environment.

#### **Specific objectives:**

- Analysis of the Biochar properties used in this research thesis.
- Use the Biochar as an eco-friendly filler in cementitious composites.
- Determine the ideal percentage of Biochar that must be added in the mixture.
- Evaluate the mechanical/economic benefits associated with the use of Biochar in cementitious pastes.



## **1.3 Justification and importance**

The current concern for the conservation of the environment, as well as for trying to mitigate the effects of climate change, encourage the use of sustainable techniques, that are economically viable, environment friendly and socially beneficial. Within this field and this new vision, the development of the project can be framed, it aims to promote and follow the studies already carried out in recent years by researchers from the Politecnico di Torino and other institutions, where the use of various pyrolyzed/gasified organic wastes are incorporated to improve cement performance and reduce its environmental impact.

In recent years, there has been a significant interest on Biochar for various environmental applications, pollutants removal, carbon sequestration, and soil amelioration. In this occasion, the Biochar used was originated from virgin wood chips, treated by gasification. It is expected to improve the mechanical (i.e. bending and compression strength, fracture energy and ductility) and economic benefits of cement, generating a material not only more durable but also more sustainable. Given that the reduction in the use and production of cement and the use of the waste generated daily translates into the mitigation of climate change and the fulfillment of the commitments established in the Kyoto Protocol and the Paris Agreement. This allows assessment a multitude of losses which account for one of the main existing problems of the environment.





## Cement

#### 2.1 Introduction

Cement is a finely ground inorganic powder that, when mixed with water, forms a paste that sets and hardens. For many years it has played a key role in our lives, practically there is no work that can be undertaken without using it (Homes, schools, hospitals, bridges, ports, among others...). It is par excellence an economical and versatile adhesive and serves as an active element in mortar and concrete mixtures. Portland cement is the main hydraulic cement used today. At an environmental level, it is one of the construction materials that consumes the most energy for its manufacture, and is also responsible for a large amount of the greenhouse gases that are emitted each year in the atmosphere, which is why we seek to reduce the emissions of greenhouse gases of the cement industry, the reduction of the use of natural resources used in its manufacture and the use of waste or recycled materials. These objectives are translated into sustainable development, without destruction of natural resources.

### 2.2 Historical review

The history of cement is millenary as the history of humanity itself. The need of mankind to build his own habitat has been a well-defined goal since we stopped sleeping in caves, this situation enforced human being to seek alternatives and even the invention of materials to meet this need. In this search for protection, the man at night set fire and obtained calcined ash, which, when mixed with water, hardened.

Later years later the Egyptians incorporated gypsum and lime

into the mixture, the Greeks began using the volcanic rocks of the Santorini island for their first cement conglomerates. In Puteoli known today as Pozzuoli (Gulf of Naples, Italy), the Romans extracted volcanic material mixed with lime, hence this cement was called "pozzolan cement". The outcome cement was used to build the Roman Colosseum, the Pantheon of Agrippa or the Pont du Gard [4].

Unfortunately, when the Roman Empire was lost, the knowledge of the concrete management they had developed disappeared. This meant that, during the middle ages, the architects concentrated on built the main constructions such as castles or cathedrals with huge stones instead of concrete.

As far as 1756, the English John Smeaton found out that the best mortar was obtained when pozzolan was mixed with limestone that contained a high amount of clay material. Smeaton was the first to know the chemical properties of hydraulic lime, which later, in 1824, James Parker and Joseph Aspdin patented a new artificial hydraulic cement, manufactured by the joint combustion of limestone and coal, The Portland cement, called by its color similar to the stones of the English island Portland, nevertheless, the prototype of modern cement was produced on an industrial scale by Isaac Johnson who in 1845 managed to get high enough temperatures to make the mixture of clay and limestone obtaining what we know today as Clinker.

The intense development of the construction of railroads, bridges, ports, dams, etc., in the second half of the 19th century, gave enormous importance to cement and its factories began to spread everywhere. It is from 1900 when Portland cement was imposed on the engineering works and began a rapid decline in the consumption of natural cements. Great advances were made at the beginning of the 20th century, Thomas Edison, the American researcher managed to improve the production of cement that his compatriot David O. Saylor had introduced in the country in 1874, Edison managed to build a 150-foot-long Rotary mill (45.72m ), 70 feet (21.34m) longer than the mills that had been used to date [5]. On the other hand, between 1903 and 1907, Jürgen Heinrich Magens patents the rotary furnace for calcination whose patent Edison did not want to register. The tubular mill and the methods of transporting fresh concrete patented by Heinrich are still used today.

Currently, Portland cement has reached a great perfection and is the industrialized building material of greater consumption globally.

So far in the 21st century, China and India have been the largest cement producers in the world with 2347 and 297 million tons respectively in 2017, followed by the United States, Turkey, and Vietnam. It is estimated that total world production for 2018 will reach almost 4250 million tons [6], almost all the production is destined to link gravel and sand, to create the stone material that we know as concrete today.

#### 2.3 Cement production

Among the most common materials used to make cement are limestone, shells and chalks or marls combined with shales, clay, slate, blast furnace slag, silica sand and iron ore. These ingredients, when heated at high temperatures, form a substance like a rock that is ground into the fine dust commonly known as cement.

There are several methods of making cement: wet, semi-wet and dry method, in the case of Portland, the dry method is the most common. Everything begins when the main raw materials are extracted in the quarries, mainly limestone, clay and other materials, successively, the rock is crushed, going through different stages: The first crushing reduces the rock to a maximum size of approximately 6 inches, subsequently, the rock then passes to the secondary crushers to be reduced to the middle of the previous step, that is, 3 inches or less. From this moment the crushed material is stored in uniform layers to be subsequently selected in a controlled manner. The pre-homogenization allows to prepare the adequate dosage of the different components reducing its variability, the new ingredients are usually iron ore or fly ash. Finally, this mixture is milled, and the baking process is proceeded [7].

Generally, the rotary kilns reach almost 4 meters in diameter, large enough to accommodate a car and longer in many cases than the height of a 40-story building [8]. Large ovens are mounted with the shaft slightly inclined to the horizontal. The finely ground raw material or grout is fed at the upper end. At the lower end, there is a burst of roaring flames, produced by precisely controlled burning of powdered coal, oil, or gas under forced current.

Finding a way to make easy to cook the raw material, it is preheated before entering in the oven, afterwards it is introduced through

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the upper part of the tower and descends through it. Meanwhile, the gases coming from the furnace ascends in countercurrent, thus preheating the crude oil, which reaches 1000 °C before entering the kiln. Once it is inside, all the ingredients are heated in enormous rotary kilns of cylindrical steel coated with special refractory brick at almost 1500 °C, producing then the complex chemical reactions that give rise to the clinker, this has the size of marbles and has a gray color.

At the exit of the kiln, the clinker is introduced into the cooler, where cold air is injected from the outside to reduce its temperature from 1200 °C to 100 °C approximately [3]. The hot air from the refrigerators is returned to the kilns, a process that saves fuel, thus improving the energy efficiency of the process.

After the clinker cools, the cement plants grind it and mix it with small amounts of gypsum and limestone. The material is so fine that 1kg of cement contains 330 billion grains [8]. At this point, it is considered that the cement is ready to be transported, stored and consumed. Figure 1 illustrate the complete cement process.



Figure 1. Cement production process [9].

#### 2.4 Portland cement

Portland cement is a hydraulic cement mainly composed of calcium silicates. It sets and hardens by reacting chemically with water. During this process, the cement blends with water and create a hardened paste like a rock.

#### 2.4.1 Chemical properties

Portland cement consists essentially of lime compounds (calcium oxide, CaO) mixed with silica (silicon dioxide, SiO<sub>2</sub>) and alumina (aluminum oxide, Al<sub>2</sub>O<sub>3</sub>), these compounds are obtained from a calcareous raw material and the other oxides are derived from a clay material. Additional raw materials can be used such as silica sand, iron oxide (Fe<sub>2</sub>O<sub>3</sub>), and hydrated aluminum containing bauxite (Al (OH) <sub>3</sub>), in smaller quantities to obtain the desired composition [10].

Limestone and chalk are the most common limestone raw materials, nevertheless there are others also used, such as coral or shell deposits. In the case of clays, shales, slates and estuarine muds are the most common. Another raw material is blast furnace slag, which consists mainly of lime, silica, and alumina and is mixed with a calcareous material. Kaolin, a white clay that contains little iron oxide, is used as the clay component of white Portland cement. Industrial waste, such as fly ash and calcium carbonate from chemical manufacturing, are other possible raw materials, but their use is smaller compared to natural materials.

The content of magnesium oxide (MgO) of the raw materials must be low because the allowable limit in Portland cement is generally 4%, this percentage manages to control the expansion of the cement during the hydration process. Other impurities in the raw materials that must be strictly limited are compounds of fluorine, phosphates, metal oxides and sulfides and excessive alkalis.

Gypsum is another essential raw material of cement, of which 5% is added to the cement clinker burned during grinding to control the setting time. Portland cement can also be prepared in a process combined with sulfuric acid using calcium sulfate or anhydrite instead of calcium carbonate.

Table 1 shows a general idea of the composition of the cement,

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which indicates the limits of the mixture of the different oxides of Portland cement [11].

The compounds mentioned in Table 1 once inside the rotary kiln react with each other at different temperatures, forming what are considered the four main components of the cement; they are listed in Table 2, along with their abbreviation symbols. This abbreviated notation, used by cement chemists, describes each oxide with a letter:

CaO = C; Si<sub>2</sub> = S; Al<sub>2</sub>O3 = A, and Fe<sub>2</sub>O<sub>3</sub> = F [11].

| Compound                                     | Content (%) | Effect  |
|--|-------------|---|
| Lime (CaO)                                   | 60-67       | Controls strenght and<br>soundness                                      |
| Silica (SiO₂)                                | 17-25       | Gives strenght, excess quantity causes slow setting                     |
| Alumina (Al <sub>2</sub> O <sub>3</sub> )    | 3-8         | Gives a rapid setting quality   |
| Iron Oxide (Fe <sub>2</sub> O <sub>3</sub> ) | 0.5-6       | Provides color, hardness and<br>streng. Helps fussion of<br>ingredients |
| Magnesium oxide (MgO)                        | 0.5-4       | Color and hardness. Excess cause cracking                               |
| Sodium Oxide (Na <sub>2</sub> O)             | 0.3-1.2     | Controls residues. Excess<br>cause cracking                             |
| Sulphur Trioxide (SO <sub>3</sub> )          | 2-3.5       | Makes cement sound  |

| Table 1. | Approximate | composition | limits for | Portland | cement. |
|----------|-------------|-------------|------------|----------|---------|
|----------|-------------|-------------|------------|----------|---------|

| Table 2. | Portland   | cement   | compounds. |
|----------|------------|----------|------------|
|          | i oi ciana | connerie | compounds. |

| Compound                        | Composition   | Abbreviated<br>notation |
|---------------------------------|---|-------------------------|
| Tricalcium silicate             | 3CaO.SiO₂   | C₃S                     |
| Dicalcium silicate              | 2CaO.SiO <sub>2</sub>   | C <sub>2</sub> S        |
| Tricalcium aluminate            | 3CaO.Al <sub>2</sub> O <sub>3</sub>                               | C <sub>3</sub> A        |
| Tetra-calcium<br>aluminoferrite | CaO.Al <sub>2</sub> O <sub>3</sub> Fe <sub>2</sub> O <sub>3</sub> | C <sub>4</sub> AF       |

#### • Hydration of the cement and formation of the cementitious paste

The reaction by which Portland cement is transformed into a binding agent is produced in a paste of cement and water. In other words, in the presence of water the silicates and aluminates mentioned in Table 2 form hydration products, which, with the passage of time, produce a firm and hard mass known as cement paste.

Specifically, the calcium silicates react with water molecules to form calcium silicate hydrate ( $3CaO \cdot 2SiO_2 \cdot 3H_2O$ ) and calcium hydroxide (Ca  $[OH]_2$ ). These compounds are given the shorthand notations C–S–H (represented by the average formula  $C_3S_2H_3$ ) and CH, and the hydration reaction can be crudely represented by the following reactions:

2C3S + 6H = C3S2H3 + 3CH 2C2S + 4H = C3S2H3 + CH

During the initial stage of hydration, the original compounds dissolves and the dissolution of their chemical bonds generates a significant amount of heat. Subsequently, hydration stops for reasons that are not completely understood. This inactive period is extremely important in the placement of concrete. Without the period of inactivity, there would be no cement trucks and the spill should be done immediately after mixing [10].

After the period of inactivity, the cement begins to harden as CH and C-S-H are produced. These compounds are responsible for bonding the cement particles. As hydration progresses, water and cement are consumed continuously, transforming into crystalline hydrated compounds and gel. Initially, this gel is unstable due to the high-water content (Little by little, all the water turns into gel). As the development of the crystalline compounds progresses, the humidity of the gel decreases, becoming a stable gel that will later be responsible for the mechanical properties of the hardened pastes. Fortunately, the C-S-H and CH products occupy almost the same volume as the original cement and water; the volume is approximately preserved, and the contraction is manageable.

#### • Hydration heat

The chemical reactions that are generated during the hydration of the cement give rise to an exothermic reaction, and large amounts of heat can be released [11]. This property is affected by the water/cement ratio, fineness and curing temperature. Since the conductivity of concrete is relatively low, it acts as an insulator and, inside a large mass of concrete, hydration can produce a sharp increase in temperature. At the same time the outer mass of the concrete loses some heat so that a strong temperature gradient is produced, and during the subsequent cooling of the interior, serious cracking can take place. However, this behavior is modified by the fluence of the concrete or by isolation of the surfaces of the concrete mass.

At the other extreme, the heat produced by the hydration of the cement can prevent the freezing of water in the capillaries of the concrete, in cold climates and it is convenient, therefore, that there is a strong dispersion of heat. For this reason, it is advisable to know the heat producing properties of different cements to choose the most suitable for a given purpose.

## 2.4.2 Physical and mechanical properties

## • Specific weight (density)

The density of cement is determined by the ratio between the mass of a given quantity and the absolute volume of that mass [12]. In regular Portland cement, this value is very close to  $3.15 \text{ g/cm}^3$ , in the additions this value is close to  $2.90 \text{ g/cm}^3$ , depending on the amount of additions used.

Its main utility is related to the design and control of concrete mixtures since these are calculated by weight for a unit volume of concrete (1m<sup>3</sup> generally). Therefore, it is necessary to know the volume occupied by a given mass of cement within one cubic meter.

## • Fineness

Fineness is one of the most important properties of cement, this determines to a great extent the speed of hydration, the development of heat of hydration, the retraction and the acquisition of cement resistance [12]. A cement with fine grain hydrates much more easily. This parameter is determined by an indirect method with the Blaine apparatus, which consists in measuring the time necessary to pass through an

amount of air in a sample of known density. It is called surface area and is expressed in cm<sup>2</sup>/gr. Approximately 95% of the cement particles are smaller than 45 microns, with an average of 15 microns.

## • Fluidity

The consistency of the cement indicates the degree of fluidity where the cement paste can be handled. Cements have different water requirements, depending on whether or not they are added; generally, the added cements require more water since there is a greater specific surface. There is a fluidity for which a certain amount of water must be added, and this is what is called normal consistency. This parameter is determined with the Vicat needle, it means a cement paste of normal consistency when the needle penetrates 10mm +/- 1mm [13].

#### Setting and hardening

The setting describes the change of the plastic state to the solid state of a cement paste [12]. It can be divided into two stages, the initial setting is the time that elapses from the moment the cement paste receives the water and is losing fluidity until it has all its viscosity and its temperature rises and the final setting, that means, when the cement paste ceases to be deformable by relatively small loads, reaching its maximum temperature where the paste becomes hard. At this moment the process of hardening and acquiring mechanical resistance begins with the passage of time.

Several factors such as fineness and types of cement, gypsum mix, water content, temperature and humidity influence the setting and hardening of Portland cement. Knowing these parameters is important because it indicates how much time is counted to mix, transport, place, vibrate, refine and cure the concrete on site.

#### Mechanical strength

Cement has three types of strengths: compressive strength, tensile strength and flexural strength [13]. Generally, only the compression resistance is considered, being a stony material, the resistance to compression is the greatest of all. For this purpose, they usually make mortar or concrete cubes which are compressed by a machine to the point of failure, in the case of evaluation of the bending strength, prisms of 4x4x16 cm are subjected to three-point test. Cementitious paste speci-

mens are rarely used to evaluate these cement properties. The result of the tests can be affected for several reasons, such as the water-cement ratio, the curing and the humidity of the specimens. The evaluations of the different resistances are made at 3, 7 and 28 days because the resistance increases over time. On the twenty-eighth day, the compressive strength of a cementitious compound has already reached more than 90% of its strength.

## • Loss of Ignition

Heating a cement sample at 900 - 1000°C (that is, until a constant weight is obtained) causes weight loss. This loss of weight upon heating is calculated as a loss of ignition [13]. Improper and prolonged storage or adulteration during transport or transfer may lead to pre-hydration and carbonation, both of which might be indicated by increased loss of ignition.

#### • Bulk density

When the cementitious mix is elaborated, the water replaces areas where there would normally be air. Because of that, the bulk density of cement is not very important. Cement has a varying range of density depending on the cement composition percentage. The density of cement may be anywhere from 993 to 1249 Kg/m<sup>3</sup> [13].

## 2.4.3 Cement types

It is possible to modify the properties of Portland cements by adjusting the amounts of the constituent chemical compounds present on it. Physically and chemically, the different cement types differ primarily in their content of  $C_3A$  and in their fineness, in terms of performance, they differ primarily in the rate of early hydration and in their ability to resist sulfate attack. The ASTM has designated five types of Portland cement [14]:

## • Type I - Normal, ordinary Portland cement (OPC)

It is a normal cement and is obtained after mixing the clinker with the plaster. It is generally used in general construction (most buildings, bridges, pavements, precast units, etc.).

#### • Type II - Moderate Sulfate Resistance

We are facing a modified cement. Its action is moderate to the resistance of the sulfates and it is convenient to use it when we demand moderate heat of hydration. This cement is acquiring resistance more slowly than type I, finally equals it and, therefore, obtains the same resistance. It is frequently used in sewers, pipes and industrial areas, such as structures exposed to soil or water containing sulfate ions.

## • Type III - High Early Strength

This cement consists of a high initial resistance and is tremendously recommended when an accelerated resistance its needed. Concrete made with cement type III will increase the resistance incredibly when compared to type I and III. It must be borne in mind that it increases the initial resistance to very high levels. It is generally used for rapid construction or cold weather concreting.

#### • Type IV - Low Heat Hydration

It has a low heat of hydration and such an event is achieved if we limit the compounds that can most influence, that is, C<sub>2</sub>A and C<sub>3</sub>S. This cement gains resistance slowly. It is commonly used in large works, dams, and tunnels.

#### • Type V - High Sulfate Resistance

One of the characteristics of type V Portland cement is its resistance to the action of sulfates, therefore it is used in hydraulic structures and marine platforms. Sulfate resistance is achieved by minimizing the  $C_3A$  content since the said compound is very susceptible to sulfate.

## 2.5 Cement and sustainability

Nowadays, the construction sector is responsible for consuming 50% of natural resources, 40% of energy and represents more than 34% of the total waste generated [15]. In addition, the processing, transformation and transportation of non-renewable raw materials corresponding to at least 30% of the carbon impact on the planet.

Concrete is the most widely used building material in the world, by 2030, it is expected that its consumption will increase by more than 3 tons per person per year [16]. In its ordinary form it contains approximately 12% cement and 80% mass aggregate, this means that, worldwide, for the manufacture of concrete, sand, gravel and crushed rock are consumed at a rate of 10 to 11 billion tons each year approximately [17]. Extraction, processing and transport operations involving these large quantities of aggregates consume considerable amounts of energy and adversely affect the ecology of forested areas and river beds.

Cement is used in the production of concrete, acting as a binder for other key ingredients (sand and gravel), its global production is a highly polluting process. Making a ton of Portland cement requires approximately 6 GJ of energy (produced with fossil fuels generally), releasing almost 1 ton of  $CO_2$  per ton of clinker [6]. More than 4 billion tons of cement were produced in 2017 and represented more than 5% of the global  $CO_2$  emitted in the atmosphere [18], and it is estimated that these emissions will be around 28,000 million tons in the year 2050, according to the International Agency of Energy (AIE) [19]. It is important to note that the extraction of large quantities of natural raw materials, such as limestone and clay, causes extensive deforestation and the loss of the topsoil.

At this moment, for costs, versatility, and even availability of raw materials, we cannot dispense with this material. The Good news is that the latest years have emerged different studies around the world to improve concrete with alternative cements, in an environmentally friendly way. The researchers have found other materials to reduce the amount Portland cement or the use of waste materials (construction, industrial or biological disuse) such as one of its components or its production process, obtaining more sustainable concretes and cements.

In different circumstances, there are the added cements (also called composite, mixed or Portland-based cements) [20], these are formed by Portland or clinker cement, mixed or ground together with one or more materials known as additions, such as ground granulated blast furnace slag (GGBS), Silica Fume (SF), metakaolin (MK), fly ash (FA) and other products. The performance of these materials is closely related to the fundamental nature of the addition. Moreover, the economic and environmental factor has accelerated the use of these additions.

The selection of each supplementary material and its proportion in the concrete mix is made based on the required performance. GGBS is a non-metallic co-product produced in the metallurgical industry, consisting essentially of silicates, aluminosilicates, and calcium and alumina silicates. This material has latent hydraulic properties when so-

lidified and cooled rapid with water. Its composition may vary depending on the metallurgical process. Some slags are cementitious while others are not, however, the latter can be activated by the presence of Portland cement and alkaline substances.

Silica fume is a byproduct of the metallurgy of silicon alloys. It has a very low density (200-300 Kg/m<sup>3</sup>) and a large BET surface area (13000-30000 m<sup>2</sup>/Kg) and has more than 95% silica content, which is why it is considered a very active pozzolan, it is generally used in high-strength concrete. MK is a pozzolana of highly reactive aluminosilicates that is produced by calcining pure kaolinite at 750 °C. The specifications of these materials are classified by chemical and physical properties for standard requirements. FS is spherical and often hollow pozzolans produced in coal-fired power plants, mainly glassy. Its other phases are crystalline quartz, mulita, hematite and magnetite.

The use of these materials where no additional clinkering process is involved arises in the cement and concrete industry, focused on cost reduction and a significant reduction in CO<sub>2</sub> emissions since they are byproducts of the industrial manufacturing processes that provide benefits to obtain more resistant concrete and concrete [21].

Under other conditions, in Colombia, Restrepo J. C. et al. [22] have developed a new technique where calcium silicates (the main cement compound) are obtained with a process of between 5 and 20 seconds, and a temperature of 300  $^{\circ}$ C in contrast to the production hours of the clinker and the most of 1450  $^{\circ}$ C involved. With this alternative formula, CO<sub>2</sub> emissions would be reduced to only 0.05 CO<sub>2</sub> ton/ton of cement. Nowadays, Colombian researchers have only developed a pilot test with small amounts. Nonetheless, they affirm that the obtained characteristics are similar or better than the existing products in the market.

In Mexico, Ojeda O. [23] created a harmless cement for the environment from the metallurgical industries waste, replacing the base compounds of the concrete, specifically replacing the sand with industrial waste from mineral wool. Ojeda, by making use of industrial solid waste, created a reliable alternative contributing to a solution of the today's environmental issues and by being composed of recycled materials, this process decreases the final cost of the mixture, which also has a greater resistance and durability. The evaluation of the physical and mechanical properties of this new cement indicated that by replacing up to 60% of the sand with the mineral wool waste, the porosity was

reduced, increasing its density, improving the resistance to compression and bending in of 15% compared to traditional concrete.

Ekincioglu O. et al. [24] reported that cement production in Turkey has been using the disposal of sewage sludge generated for wastewater treatment plants. Cement companies can take advantage of these misuse sludges with heat energy potential as one of the alternative sources of fuel. Dry sludge is also used as an alternative fuel in its rotary kilns. One of the main benefits is that the use of sewage sludge does not generate additional emissions, on the contrary, processing it in a cement kiln offers the greatest reduction of  $CO_2$  equivalents.

Additionally, Turks have replaced natural raw materials with alternative raw materials, waste and industrial by-products. The Grid is followed by pyrite ash, excavation soil and blast furnace slag derived from the production of iron and steel, respectively. Marble wastes containing minerals such as aluminum and iron, as well as other wastes such as foundry sand, iron powder, scales, plaster chipping, bypass powder, volatile and iron dross, are also used in the production process cement. In addition, phosphogypsum, which is a contaminant residue obtained from the production of phosphoric acid in the phosphate fertilizer industry, is used as an alternative to gypsum. They also use the appropriate rocks extracted as well extracted from the excavations of building foundations like raw materials for the production of cement (in 2011 55000 tons of excavation rocks were used, corresponding to 2.5% of the raw materials produced in the Turkish quarries).

Ayer N. et al. [25] reported the potential environmental benefits of substituting fossil fuels for biooil and Biochar from mobile fast pyrolysis of forest harvest residues in an average cement plant in Quebec, Canada. Bioenergy Pathways for cement production showed reductions in non-biogenic GHG emissions as high as 50% relative to the Reference Pathway for energy provision in the plant. The use of biooil and Biochar increased the share of renewable energy in the cement plant energy mix from just under 15% in the Reference Pathway up to 47% and 73% in the Bioenergy Pathways, depending on the scale of fuel substitution. Bioenergy Pathways also led to decreases in potential acidification, ozone depletion, respiratory effects, and eutrophication impacts, with slightly higher contributions to smog-forming emissions.

Now the challenge is to make a change in cement production plants and be able to extrapolate those results on a large scale.





## **Biochar**

## **3.1 Production process**

The production of Biochar is potential waste management solution that captures and stores carbon from biomass. The International Biochar initiative (IBI) defines it as "a solid material obtained from the thermochemical conversion of biomass in an oxygen-limited environment" [26] and can be analogous to charcoal found in nature. It is important to emphasize that its production is quite different to charcoal as it is not primarily used as a fuel, but for atmospheric carbon capture and storage, or bio-sequestration.

The sources where Biochar can be produced are quite diverse, such as wood waste, municipal solid waste, crop residues, rice husks, quinoa and lupin residual, cassava rhizome, tobacco seed, paper mill sludge, olive mill sludge, algal biomass, and many others [27-36]. Pyrolysis, gasification, torrefaction and hydrothermal carbonization are the thermochemical processes used to transform the biomass and produce bio-oil, syngas and Biochar [37-38].

Pyrolysis is the most common technology to produce Biochar, depending on the heating rate and residence time it can be categorized into slow and fast pyrolysis [39]. Slow pyrolysis is also called conventional carbonization, it produces Biochar by heating biomass at a low heating rate about 400 °C, for a relatively long residence time in absence of oxygen. This method has been used to generate charcoal for centuries, slow pyrolysis commonly provides high yields of Biochar (35%) from biomass, together with tar (25%), non-condensable gasses (25%) and losses (15%). According to Song and Guo [40], slow pyrolysis

is a simple, robust, and inexpensive process that is applicable to smallscale and farm-based Biochar production. On the other hand, fast pyrolysis produces Biochar at a high heating rate between 500-1000 °C in absence of oxygen too, and short residence time, this type of pyrolysis generally provides high yields of biofuel (75%) from biomass, together with non-condensable gasses (13%) and Biochar (12%) [41]. The greatest difference between the two pyrolysis methods are the yields of Biochar and bio-oil: slow pyrolysis favors high yield of Biochar while fast pyrolysis favors high yield of bio-oil [42].

Gasification is a process whereby biomass is transformed into primarily a gaseous mixture, (syngas containing CO, H<sub>2</sub>, CO<sub>2</sub>, CH<sub>4</sub>, and smaller quantities of higher hydrocarbons) by supplying a controlled amount of oxidizing agent with temperatures bigger than 700 °C, the oxidizing agent used in gasification can be oxygen, air, steam or mixtures of these gases. In gasification the typical Biochar yield averages about 10wt% of biomass while the liquid product (5%), consisting of aromatic hydrocarbon compounds, moreover, syngas is produced too and is used as fuel in gas power engines and gas turbines. Depending on the gasification medium (air, oxygen, or steam, etc.), the calorific value of the syngas varies, and can below (4–6 MJ/Nm<sup>3</sup>), medium (12–18 MJ/Nm<sup>3</sup>), or high (40 MJ/Nm<sup>3</sup>) [43] [44].

Another way to manage the biomass is Torrefaction, a pretreatment technology where biomass is thermally degraded in an oxygen-free environment under atmospheric pressure and at reaction temperatures of 200–300 °C [45]. The moisture and thermal degradation of cellulose, hemicellulose, and lignin in lignocellulosic biomass can be remove through this process as well as that of carbohydrates, proteins, and lipids [46] [47]. Due to the improvement in biomass properties from torrefaction, the upgraded biomass achieves a better fuel quality, moreover, torrefaction can lower the transportation and storage cost of various biomass wastes [48]. Although it is more economic and technological feasibility, torrefaction has been suggested as a promising route to produce alternatives to coal for industrial applications [49].

Finally, Hydrothermal carbonization (HTC) of biomass takes place in Water at elevated temperatures (160–800 °C). Since the water Temperature is above 100 °C, the action pressure also must be elevated to maintain the water in a liquid form. Based on reaction temperature, HTC can be divided into high-temperature HTC (between 300 and 800 °C) and low-temperature HTC (below 300 °C). Considering their action of high- temperature HTC (above 300 °C) are beyond the stability condition of most organic compounds, the dominant reaction during high-temperature HTC is hydrothermal gasification and the dominant products are gases, such as methane and hydrogen. Below 300 °C, gasification is limited and carbonization of biomass to char dominates their action. Char yield of low-temperature HTC varies from 30% to 60% depending on the feedstock properties, reaction temperature, and pressure [50].

## **3.2 Properties and applications**

The properties of Biochar vary due to the raw material and the thermochemical process used in its production [51-53], however, in general terms, it is a fine, porous and light material, which gives it a great absorption capacity and a large surface area, as well as having a basic pH and being a material rich in carbon (in the case of the Biochar of woody sources, the carbon content can be more than 95%), the carbon compounds in the Biochar are stable for long periods of time (hundreds or thousands of years), consequently, it is believed that they are effective for long-term C sequestration [54-56].

Furthermore, The International Biochar Initiative (IBI) proposed three general types determined by the organic carbon content [57]. Biochar with a Corg mass fraction of  $\geq$  60% would be of Class 1, Biochar with Corg  $\geq$  30% but less than 60% would be of Class 2, and Biochar with Corg  $\leq$  10% would be of Class 3. Materials with Corg < 10% or with an H/C ratio of >0.7 mol^(-1) would not be classified as Biochar.

Because to these properties, the concern on the several uses have been growing as a result of its wide range of applications in carbon sequestration, soil quality improvement, environmental remediation and to a lesser extent in the field of construction.

The utilization of Biochar in soils occurs mostly in the case of low fertility, it affects the physical properties that can subsequently have a direct effect on the growth of the plant, thanks to the ability to reduce the effects of drought increasing the soil moisture content (water retention), thus decreasing nutrient leaching and erosion due to the retention of nutrients in soils, increasing soil pH capacity and soil resistance to acidification and also, with the Biochar it has been possible to reduce GHG emissions, particularly nitrous oxide (N<sub>2</sub>O) and methane (CH<sub>4</sub>), it is also a mechanism for carbon capture [27] [58-63].

Biochar is also emerging as an economic substitute for activated carbon to eliminate various organic pollutants such as agrochemicals, antibiotics, polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), volatile organic compounds (VOCs) and a series of inorganic contaminants like, heavy metals, ammonia, nitrate, phosphate, sulfur, etc., of aqueous, gaseous and/or solid phases. The recent work focuses Biochar on adapting their properties to improve their effectiveness in eliminating organic and inorganic contaminants. This make it a potential material for the treatment of environmental problems like contaminated soils and wastewater [64-71].

Other research has shown that Biochar can be successfully used as a bulking agent for improving the overall food waste compost process [72-73], as well as a high potential to replace expensive and nonrenewable conventional catalysts, showing improvement in various reactions such as the production of biodiesel, removal of tar in bio-oil and syngas, NOx, syngas production, and biomass hydrolysis [74]. It has been even as a mattresses and pillows filler, since the Biochar can absorb perspiration and odors, being a thermal insulator that reflects the heat, which allows a comfortable sleep in summer, it also protects against electromagnetic radiation and eliminates negative ions of the skin, this is supposed to prevent insomnia and neck tension [75].

In the construction field, Biochar is a recent component, a new and interesting research is being developed on the optimization of it use, with the to minimize  $CO_2$  and other GHG emissions into the atmosphere. By having a micrometric scale can be inserted into the cement mixture as a filler since it does not react with the cement matrix. An important feature of the use of Biochar in cementitious mixtures is its porosity, which helps water retention within the particle's microstructures, as water does not chemically bind to Biochar, it is released gradually during the hydration phase of the cement, this phenomenon helps the hydration process in the first days of curing by providing good polymerization conditions for the concrete and allowing the development of the microstructure, reducing the porosity that could be caused by the very rapid evaporation of water, consequently improving the durability of concrete.

Several researchers have sought to identify the optimal dose of Biochar required to provide better performance to the concrete, to maximize its mechanical achievement avoiding the use of other substances and reduce the environmental impact of cement production, contributing to the creation of construction materials nobles with the environment, showing promising results. The inert carbonized particles improve the resistance to bending, compression and fracture behavior [17] [76-83].

Thanks to the organic origin, it is important to stand out that regardless of where it is applied, Biochar is the loss of a waste, its use contributes to the best use of natural resources, responding to the current needs of combating global pollution.

The Figure 2 illustrates the main characteristics of Biochar that are relevant for it applications, according to Oliveira et al. [84].



Figure 2. Biochar characteristics and suitability for specific applications [84].





# **Experimental activity**

## 4.1 Paradigm of research

The initial stage of the formulation of a research project is essential to know and position yourself in a certain paradigm that guides the investigative process, since, as Guba and Lincoln affirm, you cannot enter the field of research without having a clear perception and knowledge of what paradigm directs the approach that the researcher has towards the phenomenon of study. [85].

Paradigms are not unique or universal and depend on the way each person constructs it, according to Flores, a paradigm encompasses a belief system about reality, the world view, the place that the individual occupies in it and the different relationships that this position would allow what is considered to be existing [86].

Nowadays there are two well-defined tendencies, each one of them presents a way of carrying out a process of investigation: the interpretative paradigm that has a qualitative approach and the positivist paradigm that has a quantitative approach, where Ricoy indicates that the positivist paradigm is qualified as quantitative, empirical-analytical, rationalist, managerial, and scientific-technological [87]. Therefore, the positivist paradigm will sustain research that aims to test a hypothesis by statistical means or determine the parameters of a given variable through numerical expression.

In this sense, the proposed research -that consists of evaluating the characteristics and performance of the Biochar-cementitious mixtures, subjected to bending and compression efforts- is considered quantitative, also based on the guidelines of Hurtado and Toro [88], in other words, using techniques for collecting measurable data, paying special attention to the subjective state of the actors, oriented towards results and experimental processes, with emphasis on replicability data as a characteristic process of the natural sciences.

## 4.2 Naturality of research

The present investigation aims to evaluate the characteristics and the performance of the Biochar made from wood wastes, provided by VIS energy S.p.A. used as an eco-friendly filler in the cement material, using different percentages of it respect the weight of cement in the cement mixture, subjected to bending and compressive test.

In this way, the present investigation is considered an experimental research in agreement with Arias that define it as the process that involves subjecting an object or group of individuals to certain conditions or stimuli, to observe the effects that occur [89].

Following the concept described above, this research has taken the data directly from the reality of the study cases, also, the investigation has collected all the information and recorded the most relevant facts of the object of study, to obtain the completest study possible.

## 4.3 Population and sample

According to Morles [90], the population or universe refers to the set for which the conclusions obtained will be valid: to the elements or units (people, institutions or things) involved in the investigation. The population of this research is composed of three types of cement paste; the plain specimens, those containing Biochar as an ecological filler in different percentages with respect to the weight of the cement (0.8%, 1%, 1.5%, 2%, and 2.5%), and finally, the specimens where a 2% of cement was replaced by Biochar. All the specimens were made at Turin, Italy, between June and September 2018.

All of them constitute the study population for the proposed research, for which the results will be generalized, the population is of finite type, since it is constituted by a certain number of elements, which in relation to this study is limited to 88 units. On the other hand, Sampieri [91], defined a sample of population as a subset of the elements that belong to that set defined in its characteristics that we call population, furthermore, the sample type of this research is an intentional non-probabilistic, defined by Morles [90] as the selection procedure in which does not know the probability that the elements of the population have to integrate the shows, the elements will be selected based on criteria or judgments of the researcher.

Due to the characteristics of the study population previously indicated, this is a small and finite population, practically all the samples were taken into consideration as study and research samples. Of the total performed, 9 specimens were excluded for a total of 79 tested specimens.

## 4.4 Materials and methods

The experimental specimens were prepared with the following materials:

## 4.4.1 Cement, water an d superplasticizer

## • Cement

Portland cement type I, CEM1 52.5 R, grey color (i.tech ULTRACEM 52,5 R provided by Italcementi S.p.A.) was used in this study, it contains 95% of clinker, while the remainder is made up of any secondary constituents conforming to UNI EN 197-1. Physical-mechanical and chemical requirements are shown in Table 3 and Table 4.

## • Water

Deionized water for mixing procedure and tap water for cast and curing.

## • Superplasticizer

Mapei Dynamon SP1, an admixture based on modified acrylic polymer (with no formaldehyde) that can efficiently disperse the cement grains. specially designed for the precast concrete industry. Its characteristics are shown in Table 5.

| Posistanco | r                          | Compressive<br>resistance (MPa) |                         |   | Evnansion |  |
|------------|----------------------------|---------------------------------|-------------------------|---|-----------|--|
| class      | Ini <sup>.</sup><br>resist | tial<br>tance                   | Standardized resistance | time                                    | (mm)      |  |
|            | 2 days                     | 7 days                          | 28 days                 | (,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,, |           |  |
| 52.5R      | ≥ 30                       | -                               | ≥ 52.5                  | ≥ 45                                    | ≤ 10      |  |

Table 3. Physical-mechanical requirements (UNI EN 197-1).

 Table 4. Chemical requirements (UNI EN 197-1).

| Propierties       | Requiriments |
|-------------------|--------------|
| Loss to fire      | ≤ 5.0%       |
| Insoluble residue | ≤ 5.0%       |
| Sulfates (as SO3) | ≤ 4.0%       |
| Chlorides         | ≤ 0.10%      |

Table 5. Characteristics of Mapei Dynamon SP1.

| Characteristics   | Value     |
|-------------------|-----------|
| Color             | Amber     |
| Density (g/cm³)   | 1.09±0.02 |
| Solid content (%) | 30.5±1.5  |
| Chlorides (%)     | < 0.10    |
| рН                | 6.5±1.0   |

## 4.4.2 Biochar

#### 4.4.2.1 Production of Biochar used in this research

Gray Borgotaro Blochar (provided by VIS energy S.p.A.) was produced from virgin wood chips through gasification, specifically, the equi-current fixed bed system "downdraft" which means that the direction of the fuel (wood) and the gas flow in the same direction at a temperature of about 700 C.

## 4.4.2.2 Properties of Biochar used in this research

Different tests were carried out to characterize the Biochar used: pH, GC-MS, TGA/DTGA, FTIR-ATR, granulometric analysis, BET and water retention capacity.

## • Potential of Hydrogen (pH) analysis

In chemistry, pH, is a logarithmic scale used to specify the acidity or basicity of an aqueous solution [92]. The pH of Biochar was measured by preparing a homogeneous aqueous suspension according to a Biochar weight ratio: water 1:10 and after 90 minutes of stirring the pH was measured using a pH meter Crison pH meter Basic 20.

#### • Gas chromatography with mass spectrometric (GC-MS)

According to Sparkman et al. [93], gas chromatography-mass spectrometry (GC-MS) is an analytical method that combines the features of gas-chromatography and mass spectrometry to identify different substances within a test sample.

First, 5g of Biochar was extracted by the acetone mixture: hexane=1:1 and the clean-up was done; the solvent was then evaporated by means of a rotary evaporator and the whole was weighed. The extracted fraction was then recovered with the acetone mixture: hexane = 1: 1 (1-2 ml), adsorbed on a small silica column (5 ml), recovered by washing with 5 ml of the usual mixture and finally dried. Subsequently a known amount of the extract (2.8, 23.6, 19.6 and 81.4 mg respectively) was recovered with toluene (28, 236, 196 and 814 µl respectively), a quantity of diphenyl was added, used as internal standard, calculated as a function of the extract, namely mg extracted: mg diphenyl = 10: 1, and injected into the gas-mass instrument (GC-MS) to evaluate the possible presence of polycyclic aromatic hydrocarbons (1µl split mode 1/20 ratio).

The instrument used is a GC-MS (Agilent) equipped with a capillary column of 30m (0.25mm i.d., 0.25mcm f.t) connected with a 5m silica pre-column (i.d0.53mm). Data were recorded in a scan mode of 70eV (41-440 m/z). A standard mixture containing 16 common IPAs was injected under the same conditions to allow identification and quantification of polycyclic aromatic hydrocarbons possibly present in the Biochar samples.

## • Thermogravimetric analysis (TGA)

Coats and Redfern [94] define the thermogravimetric analysis (TGA) as a method of thermal analysis in which the mass of a sample is measured over time as the temperature changes. This measurement provides information about physical phenomena, such as phase transitions, absorption and desorption; as well as chemical phenomena including chemisorption's, thermal decomposition, and solid-gas reactions (e.g., oxidation or reduction).

## • Infrared spectroscopy (FTIR-ATR)

Infrared Spectroscopy is the analysis of infrared light interacting with a molecule. This can be analyzed in three ways by measuring absorption, emission and reflection. The main use of this technique is in organic and inorganic chemistry. It is used by chemists to determine functional groups in molecules. IR Spectroscopy measures the vibrations of atoms and based on this it is possible to determine the functional groups, generally, stronger bonds and light atoms will vibrate at a high stretching frequency (wavenumber) [95].

Few mg of sample were placed on a diamond crystal and infrared analysis was recorded in total attenuated reflectivity (ATR) mode.

## • Mass Spectrometry with Inductively Coupled Plasma (ICP-MS)

ICP-MS (Mass Spectrometry with Inductively Coupled Plasma) is an elemental and isotopic inorganic analysis technique capable of determining and quantifying most of the elements of the periodic table in a linear dynamic range of 8 orders of magnitude (ng/L - mg/L) in addition to being able to carry out the determination of the elements in a multielement analysis that provides the composition of the sample analyzed. It can also carry out the quantification of the isotopic composition and studies of the stability of trace isotopes.

## • Granulometric analysis

The granulometric analysis on the Biochar was made using the Fritsch laser analyzer 22 compact laser (Figure 3). For this purpose, the sample-holding cell was filled with the suspension liquid, manually operating the stirring and carrying out a measurement of the reference blank. Then the sample was introduced into the sample cell in small increments by means of a pipette up to a sample dilution percentage of 7%.

## •BET analysis

The specific area and porosity of the Biochar was determined by the adsorption isotherm and desorption of nitrogen ( $N_2$ ) at 77.35 K (liquid nitrogen temperature) by the BET method (Brunauer, Emmett and Teller) [96]. The nitrogen adsorption–desorption isotherms were determined at liquid nitrogen's boiling point using a Tristar II Krypton 3020.



Figure 3. Granulometric analysis, sample container.

## • Water retention capacity

According to Gupta et al. [80] water retention by Biochar makes it a potential material in cementitious matrix thanks to their morphology and surface pores, furthermore, Gray et al. [97] reported that micro-pores and pyrogenic nano-pores provide site for adsorption of aqueous solutions.

Water retention capacity was determined according the method used by Gupta et al. [78], 30g of Biochar were dried in a continuous airflow oven at 70.3°C for 24 hours before performing the test to eliminate moisture that might be present in the powder, then, three containers were prepared with 10g of Biochar and 100g of distilled water previously weighted. The three specimens were subsequently sealed and allowed to stand for 48 hours (Figure 4), subsequently each solution was subjected to a vacuum filtering process (with cellulose filter) until there was no free water flow (Figure 5). The weight of the soaked Biochar was then subtracted from the weight of dry Biochar (Figure 6), consequently the mass of water absorbed in the Biochar was calculated.



Figure 4. Water-Biochar solution specimens.



Figure 5. Filtering process.



Figure 6. Filtered Biochar.

## • FE-SEM

The morphology of samples was observed through a SEM EDS microscope by Zeiss, at 20 kV and increased by more than 6K.

TThe pH, GC-MS, TGA/DTGA, FTIR-ATR and ICP-MS analysis were carried out at the Neotron analysis center at Modena, Italy while granulometric analysis, BET analysis, water retention capacity and FE-SEM was carried out in the Department of Applied Science and Technology -DISAT (by its Italian acronyms) - of the Politecnico di Torino.

## 4.5 Mixing procedure and preparation of specimens

Eleven types of cement paste specimens were made (refer to Table 6 ) in the MASTR-LAB of the Department of Structural, Building and Geotechnical Engineering (DISEG by its Italian acronym) of the Politecnico di Torino, in eight of them the Biochar was added in six different percentages based on the weight of the cement: 0.8%, 1%, 1.5%, 2%, and 2.5%. The design of mixtures is based on the experience of previous studies carried out in the Politecnico di Torino [17] [83]. All mixtures were prepared with a w/c ratio of 0.35 except in two cases. A set of specimens with 2% of Biochar in the mixture, and another where 2% of cement was replaced with Biochar, in these cases a w/c ratio of 0.40 was used, considering the effective reduction of water in the mixture, generated by the Biochar (Biochar being prepared at high temperature, has high pore fractions that can absorb moisture quickly, retaining part of the mixing water), this was corroborated with the result obtained in the water absorption test of the Biochar (see point 4.7).

| CEM Mix          | Description   | Cement<br>(g) | w/c  | Water<br>(g) | SP1 | SP1 (g) |
|------------------|---|---------------|------|--------------|-----|---------|
| OPC              | Plain cement paste  | 460           | 0.35 | 161          | 1%  | -       |
| BC 0.8%          | Paste with 0.8% of Biochar mixed in water   | 460           | 0.35 | 161          | 1%  | 4.6     |
| BC 1%            | Paste with 1% of Biochar mixed in water   | 460           | 0.35 | 161          | 1%  | 4.6     |
| BC 1.5%          | Paste with 1.5% of Biochar mixed in water   | 460           | 0.35 | 161          | 1%  | 4.6     |
| BC 2%            | Paste with 2% of Biochar mixed in water   | 460           | 0.35 | 161          | 1%  | 4.6     |
| BC 2%_S          | Paste with 2% of Biochar mixed in cement  | 460           | 0.35 | 161          | 1%  | 4.6     |
| BC 2%_0.40       | Paste with 2% of Biochar mixed in water, w/c ratio: 0.40                                    | 230           | 0.4  | 92           | 1%  | 2.3     |
| BC 2.5%          | Paste with 2.5% of Biochar mixed in water   | 460           | 0.35 | 161          | 1%  | 4.6     |
| BC 2%_Sost*      | Sostitution of 2% of cement (respect to BC 2%) with Biochar mixed in water.                 | 225.4         | 0.35 | 80.5         | 1%  | 2.3     |
| BC 2%_S_Sost*    | Sostitution of 2% of cement (respect to BC 2%_S) with Biochar mixed in cement               | 225.4         | 0.35 | 80.5         | 1%  | 2.3     |
| BC 2% 0.40_Sost* | Sostitution of 2% of cement (respect to BC 2%_0.40) with biochar mixed in water, w/c ratio: | 225.4         | 0.4  | 92           | 1%  | 2.3     |

| Table 6. | Mix | proportions o | f different | components | in | different |
|----------|-----|---------------|-------------|------------|----|-----------|
|          |     | types of a    | cement pas  | ste mix.   |    |           |

\*The amount of water and superplasticizer is calculated based on the sum of the cement plus the biochar

The materials mentioned above used in the preparation cement paste mixtures were weighed using a Kern KB 240-3N laboratory technology balance (Figure 7), following the indicated quantities of Table 5, after weighing the materials, the liquids (water and super plasticizer) were mixed manually for approximately 30 seconds, to this solution was successively added the Biochar (Figure 8), it is important to notice that in the case of the mixture BC 2%\_S and BC 2%\_S\_Sost, which are referred to in Table 6, the Biochar was mixed manually with dry cement for approximately 5 minutes until a homogeneous mixture was obtained (Figure 9).



Figure 7. Materials and laboratory instruments.



Figure 8. Water-superplasticizer-Biochar solution. (Left) Figure 9. Cement-Biochar dry mix. (Right)

Continuing the process, the aqueous solution was transferred to a plastic mixing vessel, in 0.8% and 1% mixing cases the water-superplasticizer-Biochar solution was subjected to an ultrasound bath. The mixtures were carried out at a temperature of about 25 °C with at 440 rpm for 3 minutes, in the first 1.5 minutes the cement was added gradually to the liquid part, in the following half it was continued mixing, after 3 minutes, the speed of the agitator was increased to 630 rpm and the mixing continued for another 3 minutes, investing a total of 6 minutes of mixing (Figure 10).

In the cases BC 2%\_0.40, BC 2% \_Sost., BC 2%\_S\_Sost and BC 2%\_0.40\_Sost, the mixing process was lower, due to the smaller amount of ingredients, in this opportunity, the process lasted 4 minutes. In the first minute, the cement was added following the first speed mentioned above, in the second minute it was continued mixing until reaching the second half, at this moment the speed was increased, and the mixing process was continued until reaching 4 minutes.

The mixture, once finished, was poured slowly to avoid air confinement in stainless steel molds previously impregnated with release agent, the mold has a capacity for 4 specimens of 2cm x 2cm x 8cm (Figure 11). Subsequently, the molds were covered with a sheet of polythene and preserved in the room of the maturation tank of the DISEG, this room has a humidity of approximately 90% (Figure 12), after 24 hours, the specimens were demolded (Figure 13), named and submerged (curing process) in water for 7 and 28 days before being removed for the flexion and compression tests (Figure 14).



**Figure 10.** Mix in process. (Left) **Figure 11.** Pouring of cement paste mixture. (Right)



**Figure 12.** Cast time. (Left) **Figure 13.** Demolding of specimens. (Right)



Figure 14. Cement pastes in curing tank.

Once the maturation in water was finished (7 or 28 days), a U-shaped cut of 6mm depth was made in the middle of the orthogonal face of the pouring surface of the specimens using the TR100S Remet cutter (Figure 15) with a 2mm thick diamond edge blade (Figure 16). This procedure was made in the Burner Rig lab of the DISAT.



**Figure 15.** TR100S Remet cutter. (Left) **Figure 16.** 2mm x 6mm U shape cut in process. (Right)

Totally, 88 specimens were made, (Table 7). The quantities of each group are reported according to the amounts of Biochar present in the mixture. It is important to specify that in some cases the specimens to be analyzed at 28 days were not made due to technical and time inconveniences.

| CEM mix         | № specimens<br>for 7 days | Nº specimens<br>for 28 days |
|-----------------|---------------------------|-----------------------------|
| OPC             | 4                         | 4                           |
| BC 0.8%         | 4                         | 4                           |
| BC 1%           | 8                         | 8                           |
| BC 1.5%         | 4                         | 4                           |
| BC 2%           | 12                        | 4                           |
| BC 2%_S         | 8                         | -                           |
| BC 2% 0.40      | 4                         | -                           |
| BC 2.5%         | 4                         | 4                           |
| BC 2%_Sost      | 4                         | -                           |
| BC 2%_S_Sost    | 4                         | -                           |
| BC 2%_0.40_Sost | 4                         | -                           |
| Tot.            | 60                        | 28                          |

#### Table 7. Set of prepared specimens.

## 4.6 Mechanical test activity

## 4.6.1 Three points bending test in Crack Mouth Opening Displacement

The three points bending test (TPB) was carried out for each notched sample which was considered optimal for the test, using a single column displacement-controlled testing machine Zwick Line-Z010, with load cell of 1 kN.

The test was performed by controlling the CMOD (Crack Mouth Opening Displacement) with a strain gauge and the test speed of 0,005 mm/min was adopted. The span adopted was 70 mm.

To evaluate the flexural strength of the specimens, Modulus of Rupture (MOR) was used:

$$\sigma f$$
,max=Fmax  $\cdot \frac{3L}{2bh^2}$  [MPa]

in which: Fmax is the maximum applied force on the prism at the instant of failure;\

"L" is the effective span;

"b" is the prism width and

"h" is considered as the height of the specimen under the point of the application of the load.

Before the start of each test, the sample is weighed and prepared for the test. Figure 17, Figure 18, Figure 19, Figure 20, Figure 21, illustrates the preparation that consists in gluing two bases and two knives on the surface of the specimen, which act for the insertion of the strain gauge. After this operation, the sample is carefully placed in the test position, with the notch facing down (Figure 22 and Figure 23).



Figure 17. Glues used to fix knives.





**Figure 18.** Guide lines for U-Shape cut. (Left) **Figure 19.** U-Shape and new guide lines for knives basis. (Right)



**Figure 20.** Knives basis. (Left) **Figure 21.** Specimen ready to test. (Right)


**Figure 22.** TPB test activity. **Figure 23.** Broken specimen during TPB test. (In the circle)

# 4.6.2 Compressive test

Compressive strength is the maximum compressive stress that under a gradually applied load a given solid material can sustain without fracture [98]. Once the bending test has been completed, the portions of broken prisms were tested in compression with displacement-controlled testing machine Zwick Line-Z010 (Figure 25), with load cell capacity of 50 kN. The displacement rate was set at 0,5 mm/min.

To evaluate cement-based composites, the compression test is the most common performance measure used by engineers in designing buildings and other structures. Compressive strength was calculated by dividing the maximum load by the original cross-sectional area of a specimen:

$$\sigma_{c,max} = \frac{F_{max}}{A}$$
 [MPa]

Figure 24, Figure 26, Figure 27 and Figure 28 shown an example of the compression test process carried out in one of the specimens.



Figure 24. Portions of broken prisms after TPB test



Figure 25. Compression test activity.



Figure 26. First portion of broken prism. (1)
Figure 27. Portion of broken prism - first crack. (2)
Figure 28. Portions of broken prism collapsed. (3)
Figure 29. Collapsed specimen. (4)

# 4.6.3 Determination of fracture energy Gf by the JCI-S-001 standard

The definitive reason why a piece or structure fails is not by overcoming an elastic limit in a point or region, but by the appearance and propagation of cracks that, ultimately, lead to the fragmentation of this and the complete loss of its resistant capacity. The study of nucleation and crack propagation is complex, but necessary to definitively identify the mechanical capacity of a body.

The fracture mechanics is a science that studies the mechanisms and processes of crack propagation in solids, as well as the distribution of stresses and deformations that occur in a cracked or discon-

tinuity material, submitted to some external tension. This science arose with the fundamental works of Griffith [99] on criteria of propagation of cracks in solids, based on concepts of transformation of elastic energy into surface energy and for this reason is known as an energetic formulation of fracture mechanics.

In simple terms, the energetic formulation of fracture mechanics consists of comparing the available energy for the propagation of a crack in a structure with the energy necessary to produce its cracking. The energy available for crack advance per unit area is called the energy release rate (G) and the energy needed for cracking is called the critical rate of energy release or fracture energy (Gf).

The behavior at tensile fracture, particularly the toughness, is an essential property of concrete. A stable uniaxial tensile test is the most direct way to determine GF, however in quasi-brittle materials it is very difficult to perform stable and representative tensile tests [100]. Stable bending tests on notched samples are much easier to perform, particularly, The Japan Concrete Institute Standard document [101], describe the simplest possible test to determine GF is the three-point bending test on a notched beam (the fracture energy, Gf, measures the amount of energy absorbed until the sample breaks into two parts), also the JCI-S-001 standard reports all the recommendations to carry out the test:

Specimens shall be beams of rectangular cross section with a notch at the mid-length to a depth of 0.3 times the beam depth as shown in Figure 30.

#### Specifically:

a) The depth of the cross section (D) of the specimen shall be not less than 4 times the maximum aggregate size (da).

b) The width of the cross section (B) of the specimen shall be not less than 4 times the maximum aggregate size (da).

c) The loading span (S) shall be 3D. The total length of the specimen (L) shall be not less than 3.5D.

d) The notch depth (a0) and notch width (n0) shall be 0.3D and not more than 5mm, respectively.

The CMOD (Crack Mouth Opening Displacement) and the corre-

sponding load F are recorded until the notched beam falls under its own weight. It is necessary, in addition to the imposed load, also evaluate the weight of the beam and some of the testing equipment act on the beam. The fracture energy is evaluated from the follow expressions:

$$G_{F} = \frac{0.75 \text{ W0+W1}}{\text{Alig}} = G_{F0} + G_{Fcorr} \qquad [N/mm^2]$$

$$W_1 = 0.75(\frac{l}{L}m_1 + 2m_2)g \cdot CMOD_c \quad [N \cdot mm]$$

Where:

- GF= fracture energy (N/mm2)
- W0= area below CMOD curve up to rupture of specimen ((N·mm)
- W1= work done by deadweight of specimen and loading jig (N·mm)
- Alig= area of broken ligament (b·h) (mm2)
- m1 = mass of specimen (kg)
- l= loading span (mm)
- L= total length of specimen (mm)

- m<sub>2</sub>= mass of jig not attached to machine but placed on specimen until rupture (kg)

- g = gravitational acceleration (9.807 m/s<sup>2</sup>)

- CMODc = crack mouth opening displacement at the time of rupture (mm)



Figure 30. Notched beam geometry.

# 4.7 Results: Biochar analysis

# 4.7.1 Chemical characterization of Biochar

## • pH analysis

As expected, the tested Biochar sample is alkaline; probably due to the presence of: organic functional groups, carbonates or inorganic alkalis. Results are reported in Table 8.

| Table 8. Biochar pH. |       |        |           |  |  |  |  |  |  |
|----------------------|-------|--------|-----------|--|--|--|--|--|--|
| Sample               | рН    | pH 24h | pH 5 days |  |  |  |  |  |  |
| Grey Borgotaro       | 10.22 | 10.8   | 10.95     |  |  |  |  |  |  |

## • GC-MS analysis

Table 9 shows the total content of PAH (polycyclic aromatic hydrocarbon) present in the analyzed Biochar. The number of PAHs is below the detection limit (LOD) of the instrument.

## **Table 9.** Content of PAH (mg/kg) present in the Biochar sample.

| Sample name          | Unit  | Value | Method |
|----------------------|-------|-------|--------|
| Naphthalene          | mg/Kg | < LOD | GC-MS  |
| Acenaphthylene       | mg/Kg | < LOD | GC-MS  |
| Acenaphthene         | mg/Kg | < LOD | GC-MS  |
| Fluorene             | mg/Kg | < LOD | GC-MS  |
| Phenanthrene         | mg/Kg | 0.25  | GC-MS  |
| Anthracene           | mg/Kg | < LOD | GC-MS  |
| Fluoranthene         | mg/Kg | < LOD | GC-MS  |
| Pyrene               | mg/Kg | < LOD | GC-MS  |
| Benz(a)anthracene    | mg/Kg | < LOD | GC-MS  |
| Chrysene             | mg/Kg | < LOD | GC-MS  |
| Benzo(b)fluoranthene | mg/Kg | < LOD | GC-MS  |
| Benzo(a)pyrene       | mg/Kg | < LOD | GC-MS  |
| Benzo(b)pyrene       | mg/Kg | < LOD | GC-MS  |
| total of PAH         |       | 0.25  |        |

LOD: 0.050 mg/kg

### • TGA analysis

The sample from gray Borgotaro appears to be thermally stable (Figure 31). The thermostability of a Biochar depends, as explained in the previous chapter, on the temperature at which it was generated: in fact, with the increase of temperature, more stable carbon forms with a high heat resistance originate inside the material [102]. Considering the above, it can therefore be assumed that this sample is produced by thermoconversions occurring at high temperatures.

To verify if the residual % weight is due to more stable forms of the carbon formed during the gasification, thermo gravimetric analyzes were carried out in air because the organic compounds, in contrast to the inorganic ones, tend to give combustion reaction in the presence of oxygen.

In fact, the thermogram (Figure 32) of the sample analyzed showed a lower % in residual weight due to the combustion of the most stable forms of carbon. As regard the unburnt fraction, this could be attributed to the presence of inorganic compounds or metals.



Figure 31. TGA. Thermogravimetric analysis conducted under the flow of N<sub>2</sub>.



Figure 32. TGA. Thermogravimetric analysis conducted under the flow of O<sub>2</sub>.

## • FTIR-ATR analysis

The ATR spectrum reported in Figure 33 shows for the sample of Biochar some characteristic peaks such as the peak at 1410 cm-1 which could be attributable to the presence of ions carboxylase R-COO- and the peak around 876 cm-1 probably belonging to rings aromatics present in the samples.



Figure 33. FTIR-ATR spectrum.

#### • ICP-MS analysis

Table 10 shows the amount of heavy metals calculated, that is, the Biochar toxicity report used in this research.

| Table | 10. | Quantity | of | heavy | metals   | calculated   | by  | inductively | coupled | plasma |
|-------|-----|----------|----|-------|----------|--------------|-----|-------------|---------|--------|
|       |     |          |    | та    | ss spect | trometry (IC | P-N | 1S).        |         |        |

| Metals and not metals in ICP | Unit   | Value   | Method                  |
|------------------------------|--------|---------|-------------------------|
| Arsenic As                   | mg/Kg  | 0.364   | IPC-MS-PT-IPC mass      |
| Cadmium Cd                   | mg/Kg  | 1.94    | IPC-MS-PT-IPC mass      |
| Chrome Cr                    | mg/Kg  | 6.35    | IPC-MS 12m-IPCmass      |
| Iron Fe                      | mg/Kg  | 1310    | M05/IPC-OES-IPC optical |
| Magnesium Mg                 | g/100g | 1.52    | M05/IPC-OES-IPC optical |
| Mercury Hg                   | mg/Kg  | < limit | IPC-MS-PT-IPC mass      |
| Nickel Ni                    | mg/Kg  | 59      | IPC-MS-PT-IPC mass      |
| Lead Pb                      | mg/Kg  | 11.3    | IPC-MS-PT-IPC mass      |
| Potassium K                  | g/100g | 6.07    | M05/IPC-OES-IPC optical |
| Copper Cu                    | mg/Kg  | 57.2    | M05/IPC-OES-IPC optical |
| Sodium Sa                    | g/100g | 0.115   | M05/IPC-OES-IPC optical |
| Zinc Zn                      | mg/Kg  | 230     | M05/IPC-OES-IPC optical |

### • Granulometric analysis

The results obtained show that more than half of the particles are below 8 µm, which makes it possible to establish a standard procedure for the use of Biochar in cement mixtures and guarantee repeatability. Table 11 shows part of the granulometric analysis of the Biochar. Figure 34 illustrates the granulometric curve.

| Date       | Time  | MesNr | Class Low | Size   | Class High | Frequ. | Cum.Frequ. |
|------------|-------|-------|-----------|--------|------------|--------|------------|
| 06-05-2018 | 12:43 | 8     | 0.57      | 0.61   | 0.64       | 0.38   | 1.16       |
| 06-05-2018 | 12:43 | 8     | 1.55      | 1.65   | 1.74       | 1.68   | 10.97      |
| 06-05-2018 | 12:43 | 8     | 4.22      | 4.46   | 4.71       | 2.79   | 31.37      |
| 06-05-2018 | 12:43 | 8     | 5.88      | 6.23   | 6.57       | 3.21   | 40.60      |
| 06-05-2018 | 12:43 | 8     | 8.20      | 8.68   | 9.16       | 3.68   | 51.12      |
| 06-05-2018 | 12:43 | 8     | 11.44     | 12.11  | 12.78      | 4.43   | 63.61      |
| 06-05-2018 | 12:43 | 8     | 14.28     | 15.11  | 15.95      | 4.87   | 73.17      |
| 06-05-2018 | 12:43 | 8     | 17.82     | 18.87  | 19.91      | 4.83   | 82.93      |
| 06-05-2018 | 12:43 | 8     | 22.25     | 23.55  | 24.86      | 3.91   | 91.33      |
| 06-05-2018 | 12:43 | 8     | 255.05    | 270.01 | 284.96     | 0.00   | 100.00     |

#### Table 11. Granulometric analysis of Biochar.



Figure 34. Granulometric curve of Biochar.

## • BET analysis

This technique aimed to characterize one of the physical properties of Biochar. From the nitrogen adsorption curve (N<sub>2</sub>) of the Biochar, it was possible to observe that there is a high nitrogen adsorption which translates a high specific area of 28.06 m<sup>2</sup>/g. also the size of the pores is 32.73 Å, or, 3.27nm. These values facilitate any access to adsorption.

#### • Water retention capacity

The water retention capacity expressed as the mass of absorber water per gram of dry biochar was calculated as 2.17 g of  $H_2O/g$  of dry Biochar.

#### • FE-SEM

SEM images of the feedstock and Biochar are shown in Figure 35, Figure 36, Figure 37 and Figure 38 at different magnifications, respectively. It can be observed that the Biochar maintain part of the biomass fibrous structure, also is clearly seen to be porous in all of the SEM images. The porous structure of char could be derived from the porous structure existing in raw biomass or was formed during the gasification process.

The surface of the gray Borgotaro Biochar showed a high porosity. This corroborates the results obtained in the BET analysis, mentioned before. Additionally, the presence of organic particles, unidentified inorganic and filamentous compounds were found.



**Figure 35.** FESEM of grey Borgotaro Biochar, 566X zoom. (Above) **Figure 36.** FESEM of grey Borgotaro Biochar, 1670X zoom. (Down)

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*Figure 37.* FESEM of grey Borgotaro Biochar, 2080X zoom. (Above) *Figure 38.* FESEM of grey Borgotaro Biochar, 6960X zoom. (Down)

#### 4.8 Results: mechanical characterization of Nano-reinforced cements

First, it is important to notice that the results obtained in this study were compared with the previous study carried out by Cosentino [83] at the Politecnico di Torino. This research was taken as a reference in order to obtain a comparison of the behavior of the Biochar as a filler, due to the similarities of the Biochar used by Cosentino [83] (softwoods Biochar) to the one used in this study (virgin wood chips), as well as the softwood Biochar was produced through pyrolysis at a temperature of 700°C in contrast to the gasification process with which the gray Borgotaro was produced at the same temperature.

From Cosentino work [83] were taken the results obtained from the TPB tests and fracture energy made to the pure cementitious paste (Without Biochar). The pure mixture of this study was carried out only to being able to carry out the compression test.

#### 4.8.1 TPB tests results

Cement paste, like ordinary concrete, suffers a brittle collapse when the force causing the first fracture is reached, causing the fracture to spread rapidly (Figure 39). The results obtained from the flexion test were difficult to interpret due to the great dispersion of data of the specimens treated with gray borgotaro Biochar, that is why the standard deviation of all the specimens was calculated. The high values of dispersion are a sample of the biggest problem found in the preparation of the specimens, that is, the proper dispersion of the Biochar particles with the cementitious matrix, despite, all the results were not discouraging (Figure 40, Figure 41 and Figure 42).



Figure 39. Load-CMOD curve of one specimen of plain cement.



Figure 40. Load-CMOD curves, Plane Cement Vs. reinforced cements with grey Borgotaro Biochar (0.8%, 1% and 1.5%).



**Figure 41.** Load-CMOD curves, Plane Cement Vs. reinforced cements with grey Borgotaro Biochar (2%, 2%\_S, 2%\_0.40 and 2.5%).



**Figure 42.** Load-CMOD curves, Plane Cement Vs. reinforced cements with grey Borgotaro Biochar (2%\_Sost, 2%\_S\_Sost and 2%\_0.40\_Sost).

With the aim of easy reading of the results and immediate feedback, the average modulus of rupture (MOR) of the specimens was evaluated, Figure 43 and Figure 44 shows the flexural strength of the specimens reinforced with Biochar at 7 and 28 days respectively.

Figure 43 shows the flexible resistance obtained by the specimens elaborated by Cosentino [83] and those of this study. It can be recognized that the cement pastes reinforced with gray Borgotaro by 0.8% and 1% show a decrease in the flexible resistance compared to the plain cementitious paste and those reinforced with softwood Biochar [83], this tendency is repeated in those specimens with 1.5%, which means that the gray borgotaro has less effect in the development of the flexible resistance applied in low quantities, however, the addition of 2% and 2.5% resulted in the overcoming of the resistance respect to the reference with 15% and almost 9% respectively respect to the reference.

On the other hand, the results at 28 days (Figure 44) were slightly different, the flexible resistance in the specimens with 2% of grey Borgotaro Biochar was reduced by 26% and the improvement was found in the samples reinforced with 1% and 1.5% showing an increase of 7% and 10% respectively, respect to the reference. Regarding to the specimens treated with 0.8%, a decrease of almost 37% is observed compared to plain cement and 46% with respect to specimens with softwood Biochar used by Cosentino [83].

Figure 45 shows the average of flexible strength of the cementitious pastes where 2% of "Gray Borgotaro" was used as a reinforcer (being mixed by water or dry process) and 2% of cement was replaced by this at 7 days. The flexible resistance decreased when the cement was replaced by the particles coming from gasification, contrary to what happened when the Biochar is added with respect to the weight of the cement, additionally, it was found that when the Biochar is mixed directly into the cement powder (BC 2%\_S) it does not represent an improvement over the reference, as it happens when the Biochar is mixed in the water-superplasticizer solution. Regarding the substitution method, that is, when the Biochar microparticles were used as a replacement for the cement, a remarkable reduction in the mechanical performance of the cement paste was found up to 40% less than the reference (BC 2%\_0.40\_ Sost). Finding the correct measurement can change this result.



Figure 43. MOR [MPa] - Average value for each specimen sets at 7 days.



Figure 44. MOR [MPa] - Average value for each specimen sets at 28 days.



### 4.8.2 Fracture behavior of reinforced cement

Starting from the three-point bending tests it was possible to study the fracture energy (GF) of the experimental samples with the introduction of Biochar in the cement paste. Particularly, in this research, the JCI-S-001 standard was used to calculate the GF, by integrating the area below the load-CMOD curve.

The 7- and 28-day fracture energy of Biochar-containing cement pastes are shown in Figure 46 and Figure 47 respectively. Figure 48 shows the comparison between the different specimens with 2% (those where the Biochar was added with respect to the weight of the cement and where the cement was supported), with respect to the pure specimens.

On the one hand, observing Figure 46 it is evident that the increase in the fracture energy of the specimens at 7 days was variable, it can be seen that those with 1%, 1.5%, 2% and 2.5% increased considerably respect to the cement-less paste without Biochar, by more than 100% in the case of 1% and more than 150% for the last three types; however, in the samples with 0.8%, the fracture energy was reduced slightly more than 15% compared to the reference. Regarding the behavior in relation to the results obtained by Cosentino [83], the specimens with 0.8% contracted more than 21%, however, those with 1% showed an improvement of almost 23%, it is also evident that the last three groups of specimens (1.5%, 2%, 2.5%) exceeded the resistance of the previous study. On the other hand, the results at 28 days (Figure 47) underwent variations, in this case, highlight the reinforced specimens with 0.8%, who had an improvement of almost three times in relation to the simple cementitious paste and 231% more with respect to those reinforced with soft wood Biochar. Likewise, in those samples with 1% Biochar, the fracture energy showed an increase, both in relation to the Cosentino study [83] compared to pure pasta with 30.43 and almost 1157%, respectively.

Finally, Figure 48 shows a constant, whether the specimens are reinforced with 2% Biochar as those where 2% cement was replaced by the grey borgotaro particles. In both situations, the increase in fracture energy with respect to the reference was achieved. Even if the specimens with less cement do not exceed the improvement achieved by those where the cement was not replaced, the Biochar has a great effect in improving the tenacity of the cementitious paste, reaching more than 207% (BC 2% \_Sost) in comparison to the reference.

The increase in the fracture energy provided by the Biochar to the cementitious pastes is due to the generation of a more articulated and tortuous fracture trajectory and, therefore, much less linear than the typical brittle fracture of the cement. This explains the variation in postpeak behavior of the material and the increase in the ability to absorb energy before breaking. This fact can be corroborated on the FE-SEM analysis carried out by Restuccia [17].







Figure 49. Fracture energy at 7 days, of different types of 2% specimens.

### 4.8.3 Compressive test results

Like the flexion test, the compression force was evaluated through the average of the specimens. The test was carried out with the two halves that make up each broken specimen due to the flexion test.

The 7- and 28-day compressive strength of Biochar-containing cement pastes are shown in Figure 49 and Figure 50 respectively. The results presented in Figure 49, shows that samples with 2% Biochar mixed in water or with cement powder, reached a resistance of 53.1 MPa and 52.57 MPa respectively, after 7 days of curing, which results in an improvement of more than 4% Regarding the simple cement paste, however, the improvement is considerably higher in the case of the cement paste reinforced with 2.5% of gray borgotaro, reaching almost 60 MPa, which represents more than 18% compared to the reference. Contrary to what was expected, the cementitious pastes made with a higher water cement ratio, showed a decrease of almost 11% with respect to the reference.

On the other hand, the results of the 28-day test are reported in Figure 50, it can be seen that compression resistance in those specimens reinforced with Biochar was no improved, it can be extracted that those specimens with 1% and 2% of Biochar represent 99% and almost 94% of the plain cement paste respectively. The reduction of the compressive strength can be attributed to a poor dispersion of the Biochar particles in the mixture, this fact can generate localized weak zones affecting the strength of the cementitious paste.













# **Economic and environmental benefits**

# 5.1 Cost and emissions of biomass treatment

The most common definition of food-waste is perhaps that of purchased and not consumed food that ends up in the trash. Although certainly referring to a substantial part of food waste, this is certainly not the only valid meani g, because, along with the entire agri-food chain, there are several reasons why it often happens that foodstuffs are still edible discarded.

Even if the definition of food-waste is very varied, recently, the Commission for Agriculture and Rural Development considered it as "the set of products discarded by the agri-food chain, which (for economic, aesthetic or the proximity of the consumption expiry, even if still edible and therefore potentially destined for human consumption), in the absence of a possible alternative use, are destined to be eliminated and disposed of, producing negative effects from the environmental point of view, economic costs and lost profits for companies" [103]. In Italy, on the other hand, Segrè A. et al. [2] defines food-waste as "food products discarded by the agri-food chain, which have lost commercial value, but can still be destined for human consumption".

The analysis carried out by FAO in 2011 [104] estimates food waste in the world at 1.3 billion tons per year, equal to about a third of total food production for human consumption. Thi et al. [105] reported that the per capita food waste in d veloped and developing countries are 107 kg/year and 56 kg/year, respectively. According to the USDA [1], 31% of food for human consumption is wasted each year in the United States, especially at home and in restaurants. In Europe the amount of food

wasted annually amounts to 89 million tons, equal to 180 kg per capita, but this figure does not consider the losses during production and harvesting [106]. Only in Italy in 2009 the agricultural goods left in the fields amounted to 17.7 million tons, equal to 3.25% of the total production as reported by Segrè et al. [107].

There is a lot to do to understand the losses causes, in the phases of the first transformation of agricultural products and semi-finished products, the causes that determine waste are mainly technical malfunctions and inefficiencies in production processes. Both wholesale and retail, waste in distribution and sales depend on multiple causes, including inappropriate orders and incorrect demand forecasts. As far as domestic waste is concerned, they arise from the consumer's difficulty in correctly interpreting food labeling; or because too large portions are prepared; o due to errors made during the planning of purchases; or finally when foods are not stored properly.

Other than food wastes, another significant type of waste is generated from the wood processing industry. Only Italy generated almost 3 million tons (a significant portion of 60% is recycled, nevertheless, the rest 40% is disposed in landfills or combusted) according to Rilegno [108] and these were generated from wooden packaging released for consumption across the country.

Because of the problem with garbage, not only organic, separate waste collection has been established, this is a strategic theme for environmental sustainability. It allows waste resources to fall within the production and consumption cycles, with a view to maximum efficiency in the use of resources, in full consistency with the principles and objectives of the new European strategy on the circular economy.

Therefore, food losses and waste generate negative environmental and economic impacts. To estimate the environmental impact of a food it is necessary to consider its entire life cycle, covering all stages of the food supply chain. The indicators that can be considered are three:

# Carbon Footprint

The carbon footprint is the total amount of greenhouse gases produced to directly and indirectly support human activities, usually expressed in equivalent tons of carbon dioxide (CO<sup>2</sup>) [109].

# • Ecological Footprint

The ecological Footprint is a resource that measures how much biologically productive land and sea is used by a given population or activity and compares this to how much land and sea is available. Productive land and sea areas support human demands for food, fiber, timber, energy, and space for infrastructure. These areas also absorb the waste products from the human economy. The Ecological Footprint measures the sum of these areas, wherever they physically occur on the planet [110].

# • Water Footprint

The water footprint is the amount of water used to produce each of the goods and services we use. It can be measured for a single process, such as growing rice, for a product, such as a pair of jeans, for the fuel we put in our car, or for an entire multi-national company. The water footprint can also tell us how much water is being consumed by a country – or globally – in a specific river basin or from an aquifer [111].

In Italy, the data collected showed that fruit and vegetables (biomass) thrown away in stores, have led to the consumption of more than 73 million m<sup>3</sup> of water (Water Footprint) in a year (with this amount of water it would be possible to fill 29200 Olympic pools [112]), the use of environmental resources equal to almost 400 millions m<sup>2</sup> equivalent (Ecological Footprint) and the emission of more than 8 million kg of CO<sup>2</sup> equivalent (Carbon Footprint) [2]. The problem lies in the fact that year after year these values increase, as stated in the study done for UTILITALIA [113], the organic collection has passed from an incidence of 8% in 2007 on the total urban waste to 19% in 2014; the growth in volumes was over 3 million tons, corresponding to an increase of 11%.

From the economic impact point of view, a value of 10 billion euros a year is estimated for the losses that occur in agriculture, 1.2 billion for industrial waste and 1.5 billion for those concentrated in the distribution phase, for a total of about 12.7 billion euros [2]. As far as waste disposal costs are concerned, the collection of organic waste costs 132  $\in$ /Ton, transportation to treaty establishments is 22  $\in$ /Ton and, finally, the treatment of biomass has a cost of 88  $\in$ /Ton [113], obtaining a total of 242 $\in$ /Ton.

The organic fraction plays a particularly important role within the system of differentiated collections of urban waste. It has recorded the highest growth rates in the last years, maintaining realistic prospects for an increase in volumes collected and processed soon. The organic fraction therefore proves to be strategic not only for the purposes of pursuing the objectives of separate collection and recycling from Directive 98/2008/EC [114], but also, thanks to anaerobic digestion, to promote the use of energy from renewable sources and the limitation of greenhouse gas emissions [115]. However, the progressive growth of the intercepted quantities inevitably also corresponds to an increasing incidence, within the cost of the service and the costs related to the management of this fraction.

Whereas organic matter must be collected and taken to treatment plants, in most cases this biomass is converted into compost or incinerated. It is true that composting provides benefits for the land and agriculture, however, the composting process requires great efforts, it require relatively large areas, and odor control is a common problem, in addition the times involved in its realization are not precisely short, to this is added the effort required for its spreading on the land once the final product is achieved, furthermore, the nutrients that were present in the initial phase of production are not always the same in the final product or at least it have been reduced in a significant way if the compost is not conserved correctly, another disadvantage of the compost is the methane emissions and the need for large quantities of material to be able to fertilize a land, according to Coleman [116], to fertilize a land of 81 m<sup>2</sup>, more than 2m<sup>3</sup> of compost is needed, that is almost two full truck loads with a standard short-bed pickup. On the other hand, Traditional incineration of waste in oxygen-rich environment produces huge amounts of greenhouse gases and toxic dioxins and furans, which have adverse health and environmental effects. It is also a costly method.

To address the low recycling rates food/agricultural and wood waste, an alternative way is recycling and converting biomass into Biochar. Fortunately, biomass is a rich source of energy, which can be treated by pyrolysis or gasification, obtaining an energy recovery, which translates into a way to recover the large losses of money mentioned above, plus a significant contribution to reduce emissions of greenhouse gases emissions [117] [118] [119], actually, Biochar has the potential to reduce net greenhouse gas emissions by approximately 870 kg of  $CO_2$  equivalent per ton of dry matter, of which almost 64% is obtained from the capture and storage of Biochar raw material [120]. These values can

change depending on the type of raw material and the preparation conditions used.

# 5.2 Biochar production costs

Producing Biochar from organic residues is a potential method to integrate carbon sequestration and residue management costs, although there are no standards for their commercialization, there are several studies of production costs where the raw material used (biomass) comes from different sources:

Huang et al. [121] perform a technical and economic analysis of generating Biochar together with electricity and/or heat from poultry litter waste in the UK, the results suggest that when paying  $\in$ 22/ton for handling and storing the feedstock without any options of selling either heat or electricity, the break-even selling price (BESP) of biochar is around  $\in$ 244/ton. If the electricity and heat produced are sold and a gate fee is introduced the BESP can be further reduced to  $\in$ 73/ton.

Shackley et al. [28] developed in the UK a comprehensive review on cost of Biochar depending on three different scales of production: large, medium and small according to the feed-stock consumption 185.000, 16.000 and 2000 oven dry tons of biochar/year respectively, using biomass from straw, arboriculture, miscanthus, waste wood, green waste & sewage sludge and animal waste. The cost of Biochar including production, transportation and field application has been estimated between 195 and 513 €/ton.

Gupta et al. [81] executes in Singapore a production cost analysis converting wood waste to Biochar at two different pyrolysis temperature ranges (300 and 500  $^{\circ}$ C) considering the investment facility, rental for space, cost on man power, electricity tariff and general monthly fee. The cost calculation showed that production of Biochar incurs a net positive cost of about  $\notin$  266 and  $\notin$ 426 per ton of BC 300 and BC 500 respectively.

Wrobel-Tobiszewska et al. [122], carry out an economic analysis that considered on-site Biochar production system using post-harvest forestry residues from eucalypt plantations in Tasmania, with Biochar being utilized within the system, or sold as a product, Wrobel-Tobiszewska et al. found a potential annual income of over €157.425 (2014 value).

Mohammadi et al. [123] investigated economic returns and energy use of alternative rice production systems in North Vietnam with various residue management options, they found that biochar addition enhanced the NPV of rice by 12% and reduced the non-renewable energy intensity by 27%, crediting GHG emissions abatement in low and high carbon price scenarios.

The values obtained by several researchers, such as Shackley et al. [28] and Gupta et al. [81] are relatively close, inclusive, the prices of the small-scale Biochar produced by them is similar to the price found by Huang et al. [121] (referring to the price without consideration of the sale of electricity, heat and gate-fee). Nevertheless, there is dearth of comprehensive economic analysis of production of Biochar in Italy.

It is important to notice that according to the different authors, the price of Biochar can be reduced or even be free as production increases in popularity, that is, a large-scale production.

## 5.3 Biochar-cement mixture: economic/environmental analysis

Based on previous analyzes and paying special attention to the type of Biochar used in the present investigation, the price of medium-scale Biochar from wood-waste calculated by Shackley et al. [28] was considered for this study to know the influence of the use of Biochar when it is used as a filler in cement paste mixtures even if the Biochar used in this study is a waste of Borgotaro Hospital at Parma, Italy. The results have been compared with the simple cement paste in Table 12.

To begin, it was assumed that 1796.90 Kg of cement would be used (it is the amount required to produce  $1m^3$  of cementitious paste), the rest values were calculated following the same principle of the recipes used to produce the specimens of this investigation. The price of the cement used in this research (i.tech ULTRACEM 52.5 R) is 203  $\in$ /ton, 1 L (1Kg) of water cost  $\in$  1.44, the superplasticizer (Mapei Dynamon SP1) has a cost of 4  $\in$ /Kg and Biochar price is 19  $\in$ /ton. The cement and superplasticizer prices were derived from the data offered by the manufacturer, the water price was calculated based on an average of the sale price of different websites, and the Biochar price was taken from Shackley et al. [28]. To keep the results consistent some prices were converted from  $\notin$  or \$ to  $\notin$ .

The results presented in Table 12 suggest that when Biochar its added to cement paste, the price is practically the same compared to plain mixture, notwithstanding, in the mixture where 2% of cement was replaced with Biochar and using a w/c of 0.35, it showed a decrease by almost  $\notin$ 7 compared to the price of plain cement paste due to minor cement content.

From the environmental point of view, producing 1 m<sup>3</sup> of cement paste - where 2% of cement was replaced with Biochar - means to stop emitting more than 67 kg of CO<sub>2</sub> in the atmosphere. Furthermore, assuming that the Biochar succeeds in replacing the 2% of annual cement production, more than 85 million CO<sub>2</sub> derived from the cement production process and almost 74 million tons of CO<sub>2</sub> equivalent due to the treatment of the biomass will stop emitting, for a total emissions reduction reduction of 159 million tons of CO<sub>2</sub> approximately.

Using Biochar in cement mixtures it is still premature, to understand its full potential and economic viability more investigations needs to be undertaken. As outlined above, it is conceivable that if Biochar is produced in a large-scale unit, this could potentially contain overall net zero cost for the production, while there is possibility of profit being generated through the sales of electricity and power generation.

| Mortar mix           | Description   | Cement<br>[Kg] | Cement<br>cost € | Water<br>[Kg] | Water<br>cost € | SP1 (1%)<br>[kg] | SP1 cost<br>€ | Biochar<br>[Kg] | Biochar<br>cost € | Total<br>€/m³ | € comp.<br>OPC |
|----------------------|---|----------------|------------------|---------------|-----------------|------------------|---------------|-----------------|-------------------|---------------|----------------|
| OPC                  | Plain cement paste  | 1796.9         | 364.8            | 628.92        | 905.6           | 17.97            | 71.9          | -               | -                 | 1342.3        |                |
| BC 0.8%              | Cement paste with 0.8%<br>biochar in water                  | 1796.9         | 364.8            | 628.92        | 905.6           | 17.97            | 71.9          | 14.38           | 0.29              | 1342.6        | 0.29           |
| BC 1%                | Cement paste with 1%<br>biochar in water                    | 1796.9         | 364.8            | 628.92        | 905.6           | 17.97            | 71.9          | 17.97           | 0.36              | 1342.7        | 0.36           |
| BC 1.5%              | Cement paste with 1.5%<br>biochar in water                  | 1796.9         | 364.8            | 628.92        | 905.6           | 17.97            | 71.9          | 26.95           | 0.54              | 1342.8        | 0.54           |
| BC 2%*               | Cement paste with 2%<br>biochar in water or<br>cement       | 1796.9         | 364.8            | 628.92        | 905.6           | 17.97            | 71.9          | 35.94           | 0.72              | 1343.0        | 0.72           |
| BC 2% (0.40)         | Cement paste with 2%,<br>w/c: 0.40                          | 1796.9         | 364.8            | 718.76        | 1035.0          | 17.97            | 71.9          | 35.94           | 0.72              | 1472.4        | 130.10         |
| BC 2.5%              | Cement paste with 2.5%<br>biochar in water                  | 1796.9         | 364.8            | 628.92        | 905.6           | 17.97            | 71.9          | 44.92           | 0.90              | 1343.2        | 0.90           |
| BC 2% Sost*          | 2% of cement was<br>sustuited by biochar**                  | 1761.0         | 357.5            | 628.92        | 905.6           | 17.97            | 71.9          | 35.94           | 0.72              | 1335.7        | -6.58          |
| BC 2% Sost<br>(0.40) | 2% of cement was<br>sustuited by biochar** and<br>w/c: 0.40 | 1761.0         | 357.5            | 718.76        | 1035.0          | 17.97            | 71.9          | 35.94           | 0.72              | 1465.1        | 122.80         |

#### Table 12. Cost analysis of different cement pastes.

\* Same procedure applies by mixing the Biochar directly on the cement

\*\* Regarding the sum of the weight of cement plus that of the Biochar





# Conclusions

Nowadays, the construction fields demand materials with better performance and lower environmental impact. Cement is the most used construction material in the world and at the same time responsible for a large portion of the annual  $\rm CO_2$  emissions. On the other hand, the continuous population growth has generated an over production of waste, causing negative effects to the environment and management problems for the states.

This thesis work explored the possibility of using Biochar in cementitious composites with different percentages of addition with respect the weight of the cement, in order toto improve the mechanical properties and reduce the carbon footprint of cement.

By analyzing the previous studies carried out at Politecnico di Torino and external entities [17] [77-83], the experimental research activity has been carried out trying to focuses the attention on the optimal mix design for each mixture and the proper dispersion on carbon particles within the cement matrix.

In this study, the Biochar used was obtained through a standardized process of gasification of woody waste, simplifying the production process of high-performance cement paste. This Biochar is a waste of the VIS Energy SPA.

Based on the experimental results, the following conclusions are drawn:

• In terms of resistance to flexural strength, fracture energy and compressive strength (even when it was not exceeded after 28
days), adding 2% of "Gray Borgotaro" Biochar were found to be the most suitable replacement as filler in cementitious mixtures.

• The results of the mechanical tests showed that the addition of "Gray Borgotaro" Biochar increased the resistance to bending and generated a ductile behavior with respect to the typical brittle behavior of the pure cementitious paste, especially when 2% of "Gray Borgotaro" was used.

• The resistance to bending increase in more than 15% and fracture energy more than 150% higher than 7 days, so it is concluded that the Biochar acts as a micro-reinforcement in the cement paste which helps to deflect the trajectory of the fracture, generating multiple fractures, which is the same, a ductile failure. On the other hand, adding 0.8% and 1% of gray borgotaro does not produce the same effect achieved by the Biochar of pyrolytic origin of the Cosentino study [83]. This could be related to the different production process (of the Biochar) and the biomass source, however, the use with the use of the gray borgotaro, the fracture energy was highly improved. In relation to the specimens where the cement was replaced by Biochar, it was possible to obtain a great increase in the fracture energy even when the resistance to bending was lower compared to the reference.

• Compression strength increased especially at 7 days, the specimens showed an increase of more than 4% and 15%, respectively, when 2% and 2.5% of Biochar was used, however, at 28 days the resistance of the pure paste was lower compared to the control specimen, showing a deterioration of up to 25%, so it is concluded that its use involves less strong specimens.

• The economic analysis, even if based on a hypothesis, shows that using Biochar in cement pastes leads to a minimum price increase compared to reference one. Producing a m<sup>3</sup> of cement paste with 2% Biochar has an additional value of less than  $\in$  1, and when the Biochar is used as a substitute for cement, the price decreases by almost 1%, it is important to notice that, until the Biochar is not commercially exploited, it continuous to be, in many cases, a waste, so called, free material.

• The ignorance of the amount of waste that is generated every day, its environmental impact and the cost of its treatment,

certainly does not help to deal with the problem of waste. The use of pyrolysis or gasification are processes that considerably reduce the environmental impact compared to ordinary incineration. Its implementation improves waste management, reduces toxic emissions, contaminations associated with the elimination of food waste (on water and soil) and allows an important energy recovery.

In general, the objective of the thesis was reached, and the results of the tests were positive and satisfactory. It is expected that the results of this study will generate motivation for future research that explores the use of different sources of Biochar (such as food waste or agricultural waste), the replacement of cement in concrete mixtures (which translates into lower  $CO_2$  emissions due not only to the lower amount of cement, but also, the use of Biochar can be considered a method of carbon sequestration, independently of the source) and contribute to the fact that waste derived materials are accepted as an optimal solution for the reduction of greenhouse gases in the production of construction materials.





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## Scheda Tecnica i.tech ULTRACEM 52,5 R CEM I 52,5 R

#### Descrizione

i.tech ULTRACEM 52,5 R è un cemento Portland tipo I ad altissima resistenza normalizzata ed a resistenza iniziale elevata

#### Composizione

Contiene, conformemente alla composizione prescritta dalla norma UNI EN 197-1 (riferita cioè alla massa del cemento ad esclusione del solfato di calcio e degli additivi), 95% ÷ 100% di clinker, mentre la restante parte è costituita da eventuali costituenti secondari.

#### Requisiti di norma (UNI EN 197-1)

#### **REQUISITI CHIMICI\***

| Perdita a fuoco                 | ≤ 5,0%  |
|---------------------------------|---------|
| Residuo insolubile              | ≤ 5,0%  |
| Solfati (come SO <sub>3</sub> ) | ≤ 4,0%  |
| Cloruri                         | ≤ 0,10% |

#### **REQUISITI FISICI\***

| Tempo di inizio presa | ≥ 45 min |
|-----------------------|----------|
| Espansione            | ≤ 10 mm  |

#### **REQUISITI MECCANICI\***

Resistenze alla compressione

| 2 giorni  | ≥ 30,0 MPa |
|-----------|------------|
| 7 giorni  | N.R.       |
| 28 giorni | ≥ 52,5 MPa |

\* dati caratteristici



#### Utilizzo

Le elevate resistenze meccaniche in particolare alle brevi stagionature ne rendono ideale l'impiego per:

- la prefabbricazione (con o senza ciclo termico), specialmente per strutture di rilevante impegno statico e/o architettonico;
- strutture gettate in opera precompresse;
- strutture non precompresse in elevazione o morfologicamente snelle;
- manufatti e/o getti che necessitano di scasserature e/o movimentazioni veloci;
- opere di gunitaggio ("spritz beton").

#### Vantaggi

Sono notevoli i vantaggi conseguibili nei trattamenti di stagionatura con vapore: alle elevate resistenze finali fanno infatti riscontro le elevate resistenze anche a 16 e 24 ore. Il rapido sviluppo del calore di idratazione del prodotto offre la possibilità di realizzare getti di calcestruzzo in climi rigidi e di ridurre i costi energetici nei trattamenti termici.

#### Precauzioni

Si consiglia l'uso del prodotto per opere di mole contenuta. Per lavori che prevedono l'utilizzo di grandi masse di calcestruzzo si consiglia di orientare la scelta, in funzione dei calcoli progettuali od esecutivi, al cemento i.work TECNOCEM A-LL 42,5 R o ai nostri prodotti a ridotto sviluppo di calore d'idratazione (Ferrico AA.R.S, Ferrico Pozzolanico AA.R.S., pozzolanici e alla loppa).

#### Confezionamento e stoccaggio

Il periodo di conservazione è riportato sul D.D.T ed eventualmente, ove disponibile, sul sacco.

Prodotto a uso professionale. L'uso del prodotto dovrà essere basato su ricerche e valutazioni proprie dell'applicatore.

#### Italcementi

i.lab (Kilometro Rosso) Via Stezzano, 87 24126 Bergamo - Italia Tel. +39 035 396 111 www.italcementi.it www.i-nova.net Assistenza Tecnica N° Verde 800 820 116 sat@italcementi.net

Scheda aggiornata a novembre 2013





# Listino Prezzi

Decorrenza 4 gennaio 2016

|             |   |           |  | PRE         | EZZI         |
|-------------|---|-----------|--|-------------|--------------|
| Dorformanco | Dradatta  |           | Tino o descriziono prodotto  | (Euro/ton I | IVA esclusa) |
| Performance | Prodotto  |           | Tipo e descrizione prodotto  | Sfuso       | Sacchi       |
|             |   |           |  |             | 25кg         |
| i.work      | i.work TECNOCEM® 32,5 R   | CEM II    | (A-LL; B-LL) Portland al calcare   | 155,50      | 178,00       |
|             | i.work TECNOCEM® 42,5 R   | CEM II    | (A-LL; B-LL) Portland al calcare   | 166,00      | 188,50       |
|             | i.pro CITYCEM® 32.5 B   | CEM II    | (A-LL) Portland al calcare   | 158.50      |              |
|             | i.pro DURACEM® 32.5 R   | CEM IV    | (A) Pozzolanico  | 155.50      | 178.00       |
|             | i.pro DURACEM® 32,5 R SR  | CEM IV    | (A) Pozzolanico SR   | 155,50      |              |
|             | i.pro DURACEM® 32,5 R ARS   | CEM IV    | (B) Pozzolanico Alta Resistenza ai Solfati   | 155,50      | 178,00       |
|             | i.pro DURACEM® 32,5 R ARS SR  | CEM IV    | (A; B) Pozzolanico Alta Resistenza ai Solfati SR   | 155,50      | 178,00       |
|             | i.pro DURACEM® 32,5 R ARS LH SR                                     | CEM IV    | (B) Pozzolanico Alta Resistenza ai Solfati LH SR   | 155,50      | 178,00       |
|             | i.pro TERMOCEM® GREEN 32,5 N LH                                     | CEM III   | (A) Altoforno LH   | 154,00      |              |
|             | i.pro TERMOCEM® GREEN 32,5 R LH                                     | CEM III   | (A) Altoforno LH   | 155,50      |              |
|             | i.pro DURACEM® 42,5 N   | CEM IV    | (A) Pozzolanico  | 164,50      |              |
| : nro       | i.pro DURACEM® 42,5 N ARS   | CEM IV    | (A ) Pozzolanico Alta Resistenza ai Solfati  | 164,50      |              |
| i.pro       | i.pro DURACEM® 42,5 R ARS SR  | CEM IV    | (A) Pozzolanico Alta Resistenza ai Solfati SR  | 166,00      | 188,50       |
|             | i.pro TERMOCEM® GREEN 42,5 N  | CEM III   | (A) Altoforno  | 164,50      |              |
|             | i.pro CALIX® NHL 3,5 <sup>(3)</sup>                                 | NHL       | Calce idraulica naturale   | 269,00      | 307,00       |
|             | i.pro CALIX® BLANCA NHL 3,5 <sup>(3)</sup>                          | NHL       | Calce idraulica naturale bianca  | 344,00      | 379,00       |
|             | i.pro CHAUX PURE® NHL 5 <sup>(3)(4)</sup>                           | NHL       | Calce idraulica naturale   | 274,00      | 312,00       |
|             | i.pro CHAUX RABOT® NHL 5 <sup>(4)</sup>                             | NHL       | Calce idraulica naturale   |             | 324,00       |
|             | i.pro PLASTOCEM®  | HBC       | Legante idraulico per malte da intonaco  | 121,00      | 140,50       |
|             | i.pro MURACEM®  | MC        | Cemento per murature   | 160,00      | 185,00       |
|             | i.pro PAVI FORTE®   | Legante   | Legante per massetti   | 166,00      | 191,00       |
|             | i.pro U-COAT® GREEN   | Premix    | Malta premiscelata multiuso  |             | 150,00       |
|             | i.tech ULTRACEM® 42,5 N   | CEM I     | Portland   | 166,00      |              |
|             | i.tech ULTRACEM® 42,5 R   | CEM I     | Portland   | 166,00      |              |
|             | i.tech PORTLAND FERRICO <sup>®</sup> 42,5 N AARS SR0 <sup>(5)</sup> | CEM I     | Portland ferrico Altissima Resistenza ai Solfati SR0   | 185,00      |              |
| 1 A A A A A | i.tech ULTRACEM® 52,5 R   | CEM I     | Portland   | 175,00      | 203,00       |
| i.tech      | i.tech FIBROCEM®  | Legante   | Legante speciale per manufatti fibrorinforzati   | 159,00      |              |
|             | i.tech GEOCEM®  | API G HSR | Cemento per pozzi Classe G ad Alta Resistenza ai Solfati   | 202,00      |              |
|             | i.tech GEOTERM®   | Legante   | Legante speciale per pozzi ad alte temperature   | 219,00      |              |
|             | i.tech ALI CEM® GREEN   | Legante   | Legante solfoalluminoso a composizione bilanciata  | 360,00      | 410,00       |
| 00000       | i.tech ALI PRE® GREEN   | Clinker   | Clinker solfoalluminoso macinato   | 410,00      | 460,00       |
| 24242422    | I.tech CARGO®   | Premix    | Intasante per asfalti open grade   |             | 600,00       |
|             | i.design AQUILABIANCA® 32,5 R                                       | CEM II    | (B-LL) Portland Bianco   | 213,00      | 234,00       |
| i.desian    | i.design AQUILABIANCA® 32,5 R in sacchi da 18 kg                    | CEM II    | (B-LL) Portland Bianco   |             | 310,00       |
|             | i.design ROCCABIANCA® 42,5 R  | CEM II    | (A-LL; B-LL) Portland Bianco   | 224,00      | 246,00       |
|             | I.design ITALBIANCO® 52,5 R   | CEM I     | Portland Bianco  | 260,00      | 283,00       |
|             | I.design EFFIX®   | Premix    | Maita ad alte prestazioni per elementi di arredo   |             | 1.200,00     |
|             | i.active TECNO® BIANCO 42,5 R                                       | CEM II    | (B-LL) 42,5 R Portland bianco fotocatalitico al calcare  | 850,00      | 850,00       |
| i activa    | Lactive ULTRA® BIANCO 52,5 R  | CEM I     | Portland bianco fotocatalitico   | 1.000,00    | 1.100,00     |
| I.active    | Lactive CARGO*  | Premix    | Intasante per astalti open grade totocatalitico  |             | 1.000,00     |
|             | Lactive BIODYNAMIC  | Premix    | Naita lotocatantica au alte prestazioni per panneni architettonici                               |             | 1.100,00     |
| บเล้กหลักเป | Lactive COAT® H35   | Premix    | Rasante fotocatalitico a spruzzo   |             | 850,00       |
| UX0XIIXX    | Lactive COAT- HSS   | Premix    | Rasante lotocatantico a mano   |             | 830,00       |
|             | I.Idro DRAIN® GRIGIO  | Premix    | Predosato per pavimentazioni drenanti (Grigio)   |             | 210,00       |
| i.idro      | LIGTO UKAIN® BIANCO   | Promix    | Preuosato per pavimentazioni drenanti (Bianco)<br>Predesato per pavimentazioni drenanti (Bianco) |             | 220,00       |
|             | LIUTO DRAIN" KIT IN SECOLO & 5 Kg                                   | CEMIN     | (P) Pozzolanico nor diabo  | 152.00      | 3.000,00     |
|             | i idro GIGA® BC 22 5 <sup>(8)</sup>                                 | CEMIN     | (B) Pozzolanico per digire   | 142.00      |              |
|             | LIUTO GIGA - DC 22,5 -  | Legentr   | legente regide   | 240.00      | 265.00       |
| i speed     | i speed ALLELASH® in sarshi da 5 kg                                 | Legante   | Legante rapido   | 240,00      | 265,00       |
| n.speed     | i speed ALLEASH® IN SECCHI DE 5 Kg                                  | Reamix    | Legante rapido   |             | 460,00       |
|             | i sneed All FASY® in sarchi da 5 kg                                 | Premix    | Premiscelato rapido  |             | 400.00       |
| - And May   | паресо на слат ни засени на а кр                                    | Territa   | remaciato rapido   |             | 400,00       |
| i.light     | i light® mix per pappelle trasparente                               | Bromix    | Premiscelato grigio o bianco appositamente studiato per la                                       | C           | hiorta       |
| 2222222     | Lingite mix per parmeno trasparente                                 | riennx    | realizzazione dei pannelli di cemento trasparente  | SU FIC      | meald        |

Incluios sacco. Escluso pallet e film protettivo
 Icitation sacco. Escluso pallet e film protettivo
 Icitation immi: sacco 25kg to scoo 15kg catolo da 100kg
 Shoo franco stabilimento di ascut (Franci), sacco franco stabilimento di Reznato, compreso pallet e film protettivo
 Icitation at balimento di ascut (A 25 kg, compreso pallet e film protettivo
 Opospolita survisitati anche in sacchi da 25 kg, compreso pallet e film protettivo
 Opospolita survisitati anche in sacchi da 15 torni. com/onco 20,00
 Protectiva disponibile survisitati, per un minimo d'ordinel di 102. Disponibile anche in big bag
 Disponibile anche in scenio L e XL (pezzatura dimetro mas 6 mm per la versione L e diametro max 11 mm per la versione XL)
 Disponibile survisitate a per totti minimi da definire

N.B. I prezzi riportati nel Listino si intendono riferiti franco stabilimento di produzione



#### DESCRIPTION OF PRODUCT

Dynamon SP1 is an admixture based on modified acrylic polymer specially designed for the precast concrete industry, part of the MAPEI Dynamon SP system.

#### WHERE TO USE

Adaran

Concrete with **Dynamon SP1** has a high level of workability (consistency class S4 or S5, according to EN 206-1), and is consequently easy to apply when fresh. At the same time it offers excellent mechanical performances when hardened.

Dynamon SP1 is especially suitable for precast concrete and wherever there is the need for a strong water reduction, along with a relatively high acceleration of mechanical strengths at an early age in different consistency classes and at curing temperatures above +15°C or with accelerated steam curing treatment. Its performance makes it particularly suitable for manufacturing self-compacting concrete since Dynamon SP1 can ensure high workability. At the same time it does not significantly slow down the development of mechanical strengths at early age. The main applications of Dynamon SP1 are the production of concrete for:

- manufacturing pre-stressed reinforced beams with a high level of workability and a minimum compressive strength, R<sub>ein</sub> to cut the prestressed tendons, equal to 36 N/mm<sup>2</sup>;
- manufacturing pre-stressed reinforced concrete roofing slabs, with a high level of workability, and a minimum R<sub>ein</sub> to cut the prestressed tendons, equal to 35 N/mm<sup>2</sup> and with an excellent appearance;
- manufacturing cladding panels with a high level of workability, a very refined surface and an excellent appearance;

 self-compacting concrete for precasting. Together with Viscoffuid SCC/10 or Viscostar 3K, viscosity modifying admixtures, Dynamon SP1 is suitable for manufacturing self-compacting concrete which can be poured without vibration. Its characteristics of fluidity and resistance to segregation are also suitable for a fast casting procedure.

#### **TECHNICAL CHARACTERISTICS**

**Dynamon SP1** consists of a water solution containing acrylic polymers (with no formaldehyde). The polymers can efficiently disperse the cement grains.

#### APPLICATION PROCEDURE

Dynamon SP1 develops maximum dispersing action when added after other mixture ingredients (cement, aggregates, mineral additions or filler and at least 80% of the mixing water) and before Viscofluid SCC/10 or Viscostar 3K.

#### **COMPATIBILITY WITH OTHER PRODUCTS**

**Dynamon SP1** admixture is compatible with other products for preparing special concretes, especially with:

- hardening accelerating chloride-free admixtures from the Mapefast range for reaching very high mechanical strengths at early ages;
- air entraining admixtures from the Mapeair AE range used in the production of concrete resistant to freeze/thaw cycles:
- freeze/thaw cycles;
   Viscofluid SCC/10 or Viscostar 3K, viscosity modifying admixtures for manufacturing selfcompacting concretes;
- Mapeplast SF, silica fume based powder admixture for manufacturing "top-quality" concrete (strength, impermeability, durability);

112 .

# Dynamon SP1

#### **TECHNICAL DATA (typical values)**

| PRODUCT IDENTITY  |  |
|---|--|
| Consistency:  | liquid   |
| Colour:   | amber  |
| Density according to ISO 758 (g/m <sup>3</sup> ):                         | 1.08 ± 0.02 at +20°C   |
| Main action:  | increased workability and/or reduction of mixing water<br>and rapid development of mechanical strengths at early<br>ages and at T > 15°C |
| Classification according to EN 934-2:                                     | high range water reducing, hardening accelerating, superplasticizer, tables 3.1, 3.2 and 7   |
| Classification according to ASTM C494:                                    | type F and type C  |
| Classification according to ASTM C1017:                                   | type I   |
| Chlorides soluble in water according to EN 480-10 (%):                    | < 0.1 (absent according to EN 934-2)   |
| Alkali content (Na <sub>2</sub> O equivalent) according to EN 480-12 (%): | < 3.0  |
| pH content according to ISO 4316:   | 6.5 ± 1.0  |

- Expancrete, expansive agent for manufacturing shrinkage compensated concrete;
- fly ash for manufacturing standard and self-compacting concrete;
- different types of limestone fillers for manufacturing self-compacting concrete and any other type of concrete that require these fillers;
- DMA and Mapeform Eco form-release agents, for releasing concrete from formworks:
- curing compounds from Mapecure range to protect form-released concrete structures from rapid mixing water evaporation (floorings).

Our technical services department is available to evaluate which admixture is the most suitable to manufacture freeze/thaw cycles resistant concretes, depending on the type of cement used.

#### CONSUMPTION

Dosage by volume: from 0.6 to 1.2 l per 100 kg of cement.

Different dosages from those suggested must be previously tested through concrete trials, in addition to consulting MAPEI Technical Services Department.

#### PACKAGING

**Dynamon SP1** is available in bulk, 200 I drums, 1000 I tanks

#### STORAGE

Jaaa

Dynamon SP1 may by stored 12 months in sealed containers and protect from frost. Exposure to direct sunlight can provoke variations of the colour tone without altering in any way the performances of the product.

# SAFETY INSTRUCTIONS FOR THE PREPARATION AND APPLICATION

Dynamon SP1 is not considered dangerous according to the European regulation regarding the classification of mixtures. It is recommended to wear gloves and goggles and to take the usual precautions taken for the handling of chemicals. For further and complete information about.

the safe use of our product please refer to the latest version of our Material Safety Data Sheet.

PRODUCT FOR PROFESSIONAL USE.

#### WARNING

Although the technical details and recommendations contained in this product data sheet correspond to the best of our knowledge and experience, all the above information must, in every case, be taken as merely indicative and subject to confirmation after long-term practical application; for this reason, anyone who intends to use the product must ensure beforehand that it is suitable for the envisaged application. In every case, the user alone is fully responsible for any consequences deriving from the use of the product.

Please refer to the current version of the Technical Data Sheet, available from our website www.mapei.com

All relevant references for the product are available upon request and from www.mapei.com





| 7/8/2018              | Acqua                             | demineralizzat  | a BioFair® (2  | 5L) dopo VDE 0   | 510 - 5 x 5 litri (acqu                                | ıa distillata) - Spe                                       | edizione gratuita: A     | mazon.it: Auto e Moto     |
|-----------------------|-----------------------------------|---|--|--|--|--|--------------------------|---------------------------|
|                       | Iscriviti a Prime                 | Auto e Moto 🔻   | acqua distil   | ata  |  |  |                          |                           |
| ll tuo ind<br>Turin 1 | irizzo di consegna:<br>0122       | Scegli per<br>categoria ◄   | II mio Amaz  | ton.it Offerte   | Buoni Regalo   | Ciao. Accedi<br>Account e liste                            | Iscri<br>e – Ordini Prir | viti a 0<br>ne → Carrello |
| Auto B                | estseller Moto                    | GPS ed elettroni  | ca per veicoli   | Attrezzi e attrezza  | tura Cerchioni e pne                                   | umatici Caravan  | e Touring Tuning         | Cura dell'auto Offerte    |
| Scopri<br>Scegli per  | per veicolo:<br>r marca e modello | Marca   | Modello  | Versione   | Tipo motore  | Vai  |                          | II mio garage (0)         |
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| -                     |                                   |   |  |  | Acqua der  | nineralizzata  | Condivid                 | li -                      |
|                       |                                   |   |  |  | BioFair® (2  | 25L) dopo  | FUR 34 50 +              | Spedizione GRATIJITA      |
| Si <b>o</b> Fair'     |                                   |   |  |  | VDE 0510   | – 5 x 5 litri  | Disponibilità            | immediata. Venduto        |
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|                       | 0                                 |   |  |  | Spedizion  | e gratuita   | Il tuo indi<br>Turin 101 | izzo di consegna:         |
|                       |                                   | •   |  |  | di BioFair   |  | Quantità                 |                           |
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|                       |                                   |   |  |  | cliente  |  |                          |                           |
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|                       |                                   | Destruterbi   | atterien, Haushalt,  | Contraineenalisient<br>each you esto   | Disponibilit   | à immediata  | Aggiungi a               | Illa Lista                |
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|                       |                                   | Bielder Caulter<br>Konrad Zeier Sn. 1<br>D-S4152 Norden<br>Nickeleer chamie | de ledoù chemie gwenvigerde  | SLiter   | 34,50  |  |                          |                           |
|                       |                                   |   |  |  | <ul> <li>enti onisert,</li> </ul>                      | alla norma VDE   |                          |                           |
|                       |                                   |   |  |  | 0510 per ve  | icoli, artigianato e                                       | 2                        |                           |
|                       |                                   |   |  |  | <ul> <li>di bilancio</li> <li>Altre indica:</li> </ul> | zioni: VE-2 acqua  |                          |                           |
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|                       |                                   |   |  |  | deionat, bat<br>Applicazion                            | teria, staffa acqua<br>e: in auto kühlern                  | 1                        |                           |
|                       |                                   |   |  |  | batterie e la  | vacristallo – ferro  |                          |                           |
|                       |                                   |   |  |  | da stiro non   | e pulitore a   |                          |                           |
|                       | Clicca sull'                      | immagine per la   | a visualizzazion   | e estesa   | macchie) – i   | n cosmetici e  |                          |                           |
|                       |                                   |   |  |  | laboratorio  | - in campeggio e   |                          |                           |
|                       |                                   |   |  |  | gu nobby –<br>impianti di i                            | iscaldamento e   | ,                        |                           |
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|                       |                                   |   |  |  | <ul> <li>Per Finestra<br/>con acqua d</li> </ul>       | (dopo la pulizia<br>emineralisiertem                       |                          |                           |
|                       |                                   |   |  |  | klarspülen:  | 1/2 tazza Pro M <sup>2</sup>                               |                          |                           |
|                       |                                   |   |  |  | senza lascia   | re aloni – Non   |                          |                           |
|                       |                                   |   |  |  | <ul> <li>- Per bespri</li> </ul>                       | ihen le piante da  |                          |                           |
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|                       |                                   |   |  |  | calcificazior  | e della terra)   |                          |                           |

 $https://www.amazon.it/demineralizzata-BioFair\%C2\%AE-distillata-Spedizione-gratuita/dp/B01D1S79W4/ref=sr_1_3?ie=UTF8&qid=1534446364&sr=8-\dots 1/5$ 



eot-dir/008/86 ed.8 01/07/2010



Modena (Italy), li 27/10/2017

Data Inizio Analisi 16/10/2017 Data di accettazione 16/10/2017

## RAPPORTO DI PROVA nº 17P13246-lt-0

OS-feedmonitoring OS-residuemonitoring OS-Labor für Frisches Obst Gemüse und Kartoffen. OS-Labor für Futtermitel.

Pagina 1 di 1

UNIV. PARMA - Dipartimento di Scienze Chimiche, della Vita e della Sostenibilità Ambientale Parco Area delle Scienze, 11/a 43124 PARMA PR

CAMPIONE 17P13246

Descrizione dichiarata: MATERIALE CARBONIOSO "BORGOTARO GRIGIO" - DATA ARRIVO CAMPIONE 16/10/2017, CAMPIONAMENTO ESEGUITO DA: COMMITTENTE, TRASPORTO EFFETTUATO DA: CORRIERE. Stato all'arrivo in Laboratorio: temperatura ambiente

| DESCRIZIONE ANALISI                        | RISULTATO | u       | REC. % | UNITA' DI MISURA | LQ    | LD | METODO                     | DATA FINE<br>ANALISI |
|--|-----------|---------|--------|------------------|-------|----|----------------------------|----------------------|
| RICERCA DI METALLI E NON METALLI<br>IN ICP |           | . 0.126 |        |                  |       |    | 100 MC 07 100              | 25/40/2017           |
| Arsenico come As [331]                     | 0,364     | ± 0,130 |        | mgang            |       |    | ICF-MO-FT - ICF IIIass     | 25/10/2017           |
| Cadmio come Cd [331]                       | 1.94      | ± 0,56  |        | mg/kg            |       |    | ICP-MS-PT - ICP mass       | 25/10/2017           |
| Cromo come Cr [331]                        | 6.35      | ± 1,54  |        | mg/kg            |       |    | ICP-MS 12m - ICP mass      | 25/10/2017           |
| Ferro come Fe                              | 1310      | ± 197   |        | mg/kg            |       |    | M05/ ICP-OES - ICP optical | 27/10/2017           |
| Magnesio come Mg                           | 1,52      | ± 0,23  |        | g/100 g          |       |    | M05/ ICP-OES - ICP optical | 27/10/2017           |
| Mercurio come Hg                           | < LQ      |         |        | mg/kg            | 0,005 |    | ICP-MS-PT - ICP mass       | 25/10/2017           |
| Nichel come Ni [331]                       | 59,0      | ± 10,2  |        | mg/kg            |       |    | ICP-MS-PT - ICP mass       | 25/10/2017           |
| Piombo come Pb [331]                       | 11,3      | ± 2,5   |        | mg/kg            |       |    | ICP-MS-PT - ICP mass       | 25/10/2017           |
| Potassio come K                            | 6,07      | ± 0,91  |        | g/100 g          |       |    | M05/ ICP-OES - ICP optical | 27/10/2017           |
| Rame come Cu                               | 57.2      | ± 8,6   |        | mg/kg            |       |    | M05/ ICP-OES - ICP optical | 27/10/2017           |
| Sodio come Na                              | 0,115     | ± 0,017 |        | g/100 g          |       |    | M05/ ICP-OES - ICP optical | 27/10/2017           |
| Zinco come Zn                              | 230       | ± 35    |        | mgikg            |       |    | M05/ ICP-OES - ICP optical | 27/10/2017           |

FINE RAPPORTO DI PROVA

- File con Firma Digitale autorizzata dall'Ordine dei Chimici: 17P13246-It-0-DigitalSignature.pdf

Note e Riferimento metodi:

Clo: Information meson.
Clo: Information meson.
Il dato di incertezza di misura non è sinonimo di qualche forma di positività ma solamente della performance del metodo.
In caso di campionamento effettuato da Neotron, il laboratorio applica la Procedura Operativa Interna codice: NEOT-DIR/006/53

U: L'incentezza riportata è l'incentezza estesa calcolata utilizzando un fattore di copertura pari a 2 che da un livello di fiducia approssimativamente del 95%. Per le incerche microbiologiche sono indicati i limite inferiore e superiore de l'infinerata di confideraz aon livello di probabilità del 95% K=2, o l'intervali o di confideraz stesso. Insultat delle prove microbiologiche sono emessi in accordo a quanto previsto dalla norma ISO 7218:2007Mm 1:2013.Quando i risultati sono espressi con «4 (UPCIm) o 40 (UPC/g) i interogramismi sono presenti ma in numero inferiora e 4 (UPCIm) o 40 (UPC/g) rispettivamente.

LO: Limite di Quantificazione: è la più bassa concentrazione di analita nel campione che può essere rivelata con acettabile precisione (ripetilitità) e accuratezza in condizioni ben pecificate. Si precista che ogni risultato espresso come 4'LO non indica, in ogni caso, l'assenza del parametro i correcta ne ta empione in esame. LD: Limite di Rilevabilità: è la più bassa concentrazione di analita nel campione che può essere rivelata ma non necessariamente quantificata in condizioni ben specificate.

specificate. Pareri di conformità: valori conformi e non conformi a leggi, decreti, normative nazionali e comunitarie, specifiche fornite dal cliente sono valutati caso per caso anche tenuto conto dell'incertezza di misura delle singole prove e delle norme relative all'arrotondamento dei valori, e indicati quando sono ritenuti non conformi. >>> Rec%: Recupero% "+" indica che il recupero è stato applicato da risultato. >>> I risultati numerici compresi tra parentesi (...) dopo l'espressione <LQ sono puramente indicati di tracce non esattamente quantificabili.

#### Note parametri:

[331]: Incertezza estesa calcolata secondo l'equazione di HORWITZ utilizzando un fattore di copertura pari a 2 che dà un livello di fiducia del 95%

RAPPORTO DI PROVA VALIDO A TUTTI GLI EFETTI DI LEGGE ai sensi dell'ari 16 R.D. 13-1028 nº1642 - anti 16 e 16 Logge 16-1-1071 nº170 DI L25-3186. I dalla espressi ne trapoto di prova si diretiscora di soci camipone provido in laboratorio. La denominazione o qualissi alto rifermento dei espressi ne angioto agrico a divenzione sonti adi cliente. La riproduzione paraite deve esere autorizzata con approvazione sotti ad un Laboratorio. ARCHIVIAZIONE DATI E CONSERVAZIONE CAMPIONI: Dali grezzi e tracciati oromatografici sono archiviali ger anni 3.0. Loncitorcampione de conservato per mesi 2.

RESPONSABILE DI LABORATORIO: IL CHIMICO DOTT. GIAN CARLO GATTI - N. 124 ORDINE DEI CHIMICI MODENA - MEMBER OF AOAC N. VM 90231001 - EURCHEM Approvato dal Responsabile analisi per la sezione METALLI

#### NEOTRON SPA

Stradello Aggazzotti, 104 41126 MODENA - ITALY Tel: +39 059461711 - Fax: +39 059461777 www.neotron.it - neotron@neotron.it GMP Pharmaceutical Laboratories Authorized by AIFA Italian Medicine Agency n° AM-552015. Laboratorio Calification DM 28-2-31 AT 4 Legas 4480 per la Ricerca Applicate e Innovazione Tecr Regione Emilia Romagna - AUTORIZZAZIONE Autocontrollo N° 008MOC008 BNN-Monitoring FOrult and Vegetables Approved Laboratory Holontoring DEVA AG Fortu and Vegetables Registered Laboratory

1

| CEMENT MIX RECIPES  |                         |     |      |      |  |  |
|---|-------------------------|-----|------|------|--|--|
| Recipe n° 1   |                         |     |      |      |  |  |
| Name:     Daniel/Yasmine       Date:     29-06-18       Time:     11:32 (finished mix) - 11:36 (casting time)       ID MIXTURE:     OPC_7 & 28 days |                         |     |      |      |  |  |
| Materials Quantity % wt*  |                         |     |      |      |  |  |
| Cement "CEM I 52,5 F  | R Ultracem Italcementi" | g   | 460  |      |  |  |
| Deionized Water   | g                       | 161 |      |      |  |  |
| Superplasticizer "Dyna  | g                       | 4.6 | 1.0% |      |  |  |
| Biochar   |                         | g   | 0    | 0.0% |  |  |

w/c ratio =

0.35

NOTE:

| MIXING PROCEDURE   |                                   |          |       |  |  |
|--|-----------------------------------|----------|-------|--|--|
| First 1.5 minutes:       Speed motor 4 - Gradually pour all the cement         Next 1.5 minutes:       Speed motor 4         fifth minute:       Speed motor 6         sixth minute:       Speed motor 6 |                                   |          |       |  |  |
| Formwork removal   | date:                             | 30-06-18 | time: |  |  |
| Tests 7 days   | expected date:<br>effective date: | 05-07-18 | time: |  |  |
| Tests 28 days  | expected date:<br>effective date: | 26-07-18 | time: |  |  |

| CEMENT MIX RECIPES  |                         |     |        |       |  |  |
|---|-------------------------|-----|--------|-------|--|--|
| Recipe n° 2   |                         |     |        |       |  |  |
| Name:     Luciana/Isabella       Date:     06-06-18       Time:     11:40 (casting time)       ID MIXTURE:     BC 0.8%_7 days |                         |     |        |       |  |  |
|   | Materials               | Qua | antity | % wt* |  |  |
| Cement "CEM I 52,5 F  | R Ultracem Italcementi" | g   | 230    |       |  |  |
| Deionized Water   |                         | g   | 80.5   |       |  |  |
| Superplasticizer "Dyna  | g                       | 2.3 | 1.0%   |       |  |  |
| Biochar   |                         | g   | 1.84   | 0.8%  |  |  |

w/c ratio =

0.35

NOTE:

• Ultrasonic bath for 5 minutes

mixture ok

• 2 minutes of waiting before emptying

| MIXING PROCEDURE   |                                   |          |       |  |  |  |
|--|-----------------------------------|----------|-------|--|--|--|
| First minute:       Speed motor 4 - Gradually pour all the cement         second minute:       Speed motor 4         thirth minute:       Speed motor 6         fourth minute:       Speed motor 6 |                                   |          |       |  |  |  |
| Formwork removal   | date:                             | 07-06-18 | time: |  |  |  |
| Tests 7 days   | expected date:<br>effective date: | 12-06-18 | time: |  |  |  |
| Tests 28 days  | expected date:<br>effective date: |          | time: |  |  |  |

| CEMENT MIX RECIPES  |                         |     |      |      |  |  |  |
|---|-------------------------|-----|------|------|--|--|--|
| Recipe n° 2   | ecipe n° 2              |     |      |      |  |  |  |
| Name:     Rocio/Yasmine       Date:     07-06-18       Time:     11:10 (casting time)       ID MIXTURE:     BC 0.8%_28 days |                         |     |      |      |  |  |  |
| Materials Quantity % wt*  |                         |     |      |      |  |  |  |
| Cement "CEM I 52,5 F  | R Ultracem Italcementi" | g   | 230  |      |  |  |  |
| Deionized Water   |                         | g   | 80.5 |      |  |  |  |
| Superplasticizer "Dyna  | g                       | 2.3 | 1.0% |      |  |  |  |
| Biochar   |                         | g   | 1.84 | 0.8% |  |  |  |

w/c ratio =

0.35

NOTE:

Ultrasonic bath for 5 minutes

• Very dense mixture with lumps

• During the ultrasonic bath the biochar settled on the bottom of the beaker

| MIXING PROCEDURE  |   |          |       |  |
|---|---|----------|-------|--|
| First minute:<br>second minute:<br>thirth minute:<br>fourth minute: | Speed motor 4 - Gradually pour <b>all</b> the cement<br>Speed motor 4<br>Speed motor 6<br>Speed motor 6 |          |       |  |
| Formwork removal  | date:   | 08-06-18 | time: |  |
| Tests 7 days  | expected date:<br>effective date:   |          | time: |  |
| Tests 28 days   | expected date:<br>effective date:   | 04-07-18 | time: |  |

| CEMENT MIX RECIPES  |             |   |       |      |  |  |
|---|-------------|---|-------|------|--|--|
| Recipe n° 3   | Recipe n° 3 |   |       |      |  |  |
| Name:PaulDate:06-06-18Time:12:05 (finished mixture) 12:08 (casting time)ID MIXTURE:BC 1%_7 days |             |   |       |      |  |  |
|   | Quantity %  |   | % wt* |      |  |  |
| Cement "CEM I 52,5 R Ultracem Italcementi"  |             | g | 230   |      |  |  |
| Deionized Water   |             | g | 80.5  |      |  |  |
| Superplasticizer "Dynamon Sp1 Mapei"  |             | g | 2.3   | 1.0% |  |  |
| Biochar   |             | g | 2.3   | 1.0% |  |  |

w/c ratio =

0.35

• Cement not added in the first minute

NOTE:

| MIXING PROCEDURE   |                                   |          |       |  |  |
|--|-----------------------------------|----------|-------|--|--|
| First minute:     Speed motor 4 - Gradually pour all the cement       second minute:     Speed motor 4       thirth minute:     Speed motor 6       fourth minute:     Speed motor 6 |                                   |          |       |  |  |
| Formwork removal   | date:                             | 07-06-18 | time: |  |  |
| Tests 7 days   | expected date:<br>effective date: |          | time: |  |  |
| Tests 28 days  | expected date:<br>effective date: | 03-07-18 | time: |  |  |

| CEMENT MIX RECIPES  |          |   |       |      |
|---|----------|---|-------|------|
| Recipe n° 3   |          |   |       |      |
| Name:     Daniel       Date:     07-06-18       Time:     11:35 (finished mixture) 11:40 (casting time)       ID MIXTURE:     BC 1%_28 days |          |   |       |      |
|   | Quantity |   | % wt* |      |
| Cement "CEM I 52,5 R Ultracem Italcementi"  |          | g | 230   |      |
| Deionized Water   |          | g | 80.5  |      |
| Superplasticizer "Dynamon Sp1 Mapei"  |          | g | 2.3   | 1.0% |
| Biochar   |          | g | 2.3   | 1.0% |

w/c ratio =

0.35

NOTE:

Mixture ok

| MIXING PROCEDURE  |  |          |       |  |  |
|---|--|----------|-------|--|--|
| First minute:<br>second minute:<br>thirth minute:<br>fourth minute: | Speed motor 4 - Gradually pour all the cement       cond minute:     Speed motor 4       nirth minute:     Speed motor 6       wurth minute:     Speed motor 6 |          |       |  |  |
| Formwork removal  | date:  | 08-06-18 | time: |  |  |
| Tests 7 days  | expected date:<br>effective date:  |          | time: |  |  |
|   |  |          |       |  |  |
| Tests 28 days   | expected date:<br>effective date:  | 04-07-18 | time: |  |  |

| CEMENT MIX RECIPES  |             |   |       |      |  |
|---|-------------|---|-------|------|--|
| Recipe n° 3   | Recipe n° 3 |   |       |      |  |
| Name:     Daniel       Date:     19-07-18       Time:     11:05 (finished mix) - 11:10 (casting time)       ID MIXTURE:     BC 1%_7 and 28 days |             |   |       |      |  |
|   | Quantity %  |   | % wt* |      |  |
| Cement "CEM I 52,5 R Ultracem Italcementi"  |             | g | 460   |      |  |
| Deionized Water   |             | g | 161   |      |  |
| Superplasticizer "Dynamon Sp1 Mapei"  |             | g | 4.6   | 1.0% |  |
| Biochar   |             | g | 4.6   | 1.0% |  |

w/c ratio =

0.35

NOTE:

Ultrasonic bath for 8 minutes

Part of the cement and the solution spilled outwards during the mixing phase

Mix with some small lumps

• Part of the cement mix flowed through the formwork

| MIXING PROCEDURE  |   |          |       |  |
|---|---|----------|-------|--|
| First 1.5 minutes:<br>Next 1.5 minutes:<br>fifth minute:<br>sixth minute: | Speed motor 4 - Gradually pour <b>all</b> the cement<br>Speed motor 4<br>Speed motor 6<br>Speed motor 6 |          |       |  |
| Formwork removal  | date:   | 20-07-18 | time: |  |
| Tests 7 days  | expected date:<br>effective date:   | 25-07-18 | time: |  |
| Tests 28 days   | expected date:<br>effective date:   | 15-08-18 | time: |  |

| CEMENT MIX RECIPES   |     |        |       |      |  |
|--|-----|--------|-------|------|--|
| Recipe n° 4  |     |        |       |      |  |
| Name:     Yasmine       Date:     19-06-18       Time:     11:05 (finished mix) - 11:10 (casting time)       ID MIXTURE:     BC 1.5%_7 and 28 days |     |        |       |      |  |
|  | Qua | antity | % wt* |      |  |
| Cement "CEM I 52,5 R Ultracem Italcementi"   |     | g      | 460   |      |  |
| Deionized Water  |     | g      | 161   |      |  |
| Superplasticizer "Dynamon Sp1 Mapei"   |     | g      | 4.6   | 1.0% |  |
| Biochar  |     | g      | 6.9   | 1.5% |  |

w/c ratio =

0.35

NOTE:

No ultrasonic bath

Biochar mixed in water manually for 2 minutes

Mixture with some small lumps

Second half of the cement added shortly after the initial 1.5 minutes

| MIXING PROCEDURE  |   |          |       |  |
|---|---|----------|-------|--|
| First 1.5 minutes:<br>Next 1.5 minutes:<br>fifth minute:<br>sixth minute: | 1.5 minutes:       Speed motor 4 - Gradually pour all the cement         : 1.5 minutes:       Speed motor 4         ith minute:       Speed motor 6         xth minute:       Speed motor 6 |          |       |  |
| Formwork removal  | date:   | 20-06-18 | time: |  |
|   | expected date:  | 25-06-18 |       |  |
| Tests 7 days  | effective date:   | 25 00 10 | time: |  |
|   |   |          |       |  |
| Tests 28 days   | expected date:<br>effective date:   | 16-07-18 | time: |  |
| CEMENT MIX RECIPES                     |  |   |     |       |
|--|--|---|-----|-------|
| Recipe n° 5                            |  |   |     |       |
| Name:<br>Date:<br>Time:<br>ID MIXTURE: | Name: Yasmine   Date: 27-06-18   Time: 11:22 (finished mix) - 11:30(casting time)   MIXTURE: BC 2%_7&28 days |   |     |       |
|  | Materials  |   |     | % wt* |
| Cement "CEM   52,5 F                   | R Ultracem Italcementi"  | g | 460 |       |
| Deionized Water                        |  | g | 161 |       |
| Superplasticizer "Dynamon Sp1 Mapei"   |  | g | 4.6 | 1.0%  |
| Biochar                                |  | g | 9.2 | 2.0%  |

w/c ratio =

0.35

NOTE: • No ultrasonic bath • Part of the cement and the solution spilled outwards during the mixing phase • Small lumps in the mixture • Very dense mixture

| MIXING PROCEDURE  |   |          |       |  |
|---|---|----------|-------|--|
| First 1.5 minutes:<br>Next 1.5 minutes:<br>fifth minute:<br>sixth minute: | Speed motor 4 - Gradually pour <b>all</b> the cement<br>Speed motor 4<br>Speed motor 6<br>Speed motor 6 |          |       |  |
| Formwork removal  | date:   | 28-06-18 | time: |  |
| Tests 7 days  | expected date:<br>effective date:   | 03-07-18 | time: |  |
|   |   |          |       |  |
| Tests 28 days   | expected date:<br>effective date:   | 24-07-18 | time: |  |

| CEMENT MIX RECIPES                     |  |        |       |      |
|--|--|--------|-------|------|
| Recipe n° 5                            |  |        |       |      |
| Name:<br>Date:<br>Time:<br>ID MIXTURE: | Yasmine<br>10-07-18<br>10:19 (finished mix) - 10:25 (casting time)<br>BC 2%_7 days |        |       |      |
|  | Qua  | antity | % wt* |      |
| Cement "CEM I 52,5 F                   | R Ultracem Italcementi"  | g      | 460   |      |
| Deionized Water                        |  | g      | 161   |      |
| Superplasticizer "Dynamon Sp1 Mapei"   |  | g      | 4.6   | 1.0% |
| Biochar                                |  | g      | 9.2   | 2.0% |

w/c ratio =

0.35

NOTE:

Micture ok

Biochar powder mixed with water-sp1 solution

No lumps

|   | MIXING PROCEDURE  |          |       |  |  |
|---|---|----------|-------|--|--|
| First 1.5 minutes:<br>Next 1.5 minutes:<br>fifth minute:<br>sixth minute: | Speed motor 4 - Gradually pour <b>all</b> the cement<br>Speed motor 4<br>Speed motor 6<br>Speed motor 6 |          |       |  |  |
| Formwork removal  | date:   | 11-07-18 | time: |  |  |
| Tests 7 days  | expected date:<br>effective date:   | 16-07-18 | time: |  |  |
|   |   |          |       |  |  |
| Tests 28 days   | expected date:<br>effective date:   |          | time: |  |  |

| CEMENT MIX RECIPES                     |   |            |     |       |  |
|--|---|------------|-----|-------|--|
| Recipe n° 5                            | ecipe n° 5  |            |     |       |  |
| Name:<br>Date:<br>Time:<br>ID MIXTURE: | Daniel/Yasmine<br>10-07-18<br>9:48 (finished mix) - 9:55 (casting time)<br>BC 2%_S_7D |            |     |       |  |
| Materials                              |   | Quantity % |     | % wt* |  |
| Cement "CEM I 52,5 F                   | R Ultracem Italcementi"   | g          | 460 |       |  |
| Deionized Water                        |   | g          | 161 |       |  |
| Superplasticizer "Dynamon Sp1 Mapei"   |   | g          | 4.6 | 1.0%  |  |
| Biochar                                |   | g          | 9.2 | 2.0%  |  |

w/c ratio =

0.35

NOTE:

No ultrasonic bath

Biochar powder mixed with cement

No lumps

| MIXING PROCEDURE  |   |          |       |  |
|---|---|----------|-------|--|
| First 1.5 minutes:<br>Next 1.5 minutes:<br>fifth minute:<br>sixth minute: | Speed motor 4 - Gradually pour <b>all</b> the cement<br>Speed motor 4<br>Speed motor 6<br>Speed motor 6 |          |       |  |
| Formwork removal  | date:   | 11-07-18 | time: |  |
| Tests 7 days  | expected date:<br>effective date:   | 16-07-18 | time: |  |
| Tests 28 days   | expected date:<br>effective date:   |          | time: |  |

| CEMENT MIX RECIPES                     |   |        |       |      |
|--|---|--------|-------|------|
| Recipe n° 6                            |   |        |       |      |
| Name:<br>Date:<br>Time:<br>ID MIXTURE: | Daniel<br>19-07-18<br>11:25 (finished mix) - 11:30 (casting time)<br>BC 2%_0.40 _7 days |        |       |      |
|  | Qua   | antity | % wt* |      |
| Cement "CEM I 52,5 F                   | R Ultracem Italcementi"   | g      | 230   |      |
| Deionized Water                        |   | g      | 92    |      |
| Superplasticizer "Dynamon Sp1 Mapei"   |   | g      | 2.3   | 1.0% |
| Biochar                                |   | g      | 4.6   | 2.0% |

w/c ratio =

0.4

NOTE:

| MIXING PROCEDURE  |   |          |       |  |
|---|---|----------|-------|--|
| First 1.5 minutes:<br>Next 1.5 minutes:<br>fifth minute:<br>sixth minute: | Speed motor 4 - Gradually pour <b>all</b> the cement<br>Speed motor 4<br>Speed motor 6<br>Speed motor 6 |          |       |  |
| Formwork removal  | date:   | 20-07-18 | time: |  |
| Tests 7 days  | expected date:<br>effective date:   | 25-07-18 | time: |  |
|   |   |          |       |  |
| Tests 28 days   | expected date:<br>effective date:   | 15-08-18 | time: |  |

| CEMENT MIX RECIPES                     |  |        |       |      |
|--|--|--------|-------|------|
| Recipe n° 7                            |  |        |       |      |
| Name:<br>Date:<br>Time:<br>ID MIXTURE: | Daniel<br>27-06-18<br>12:22 (finished mix) - 11:30 (casting time)<br>BC 2.5%_7&28 days |        |       |      |
|  | Qua  | antity | % wt* |      |
| Cement "CEM I 52,5 F                   | R Ultracem Italcementi"  | g      | 460   |      |
| Deionized Water                        |  | g      | 161   |      |
| Superplasticizer "Dynamon Sp1 Mapei"   |  | g      | 4.6   | 1.0% |
| Biochar                                |  | g      | 11.5  | 2.5% |

w/c ratio =

0.35

NOTE: • No ultrasonic bath • 15 minutes for mix manually biochar and water-superplasticizer solution • No lumps • Verry dense mixture • Mixing speed different from other recipes

| MIXING PROCEDURE  |                                   |   |            |  |
|---|-----------------------------------|---|------------|--|
| First 1.5 minutes:<br>Next 1.5 minutes:<br>fifth minute:<br>sixth minute: | Speed moto                        | or 5 - Gradually pour <b>all</b><br>Speed motor 5<br>Speed motor 6<br>Speed motor 6 | the cement |  |
| Formwork removal  | date:                             | 28-06-18  | time:      |  |
| Tests 7 days  | expected date:<br>effective date: | 03-07-18  | time:      |  |
| Tests 28 days   | expected date:<br>effective date: | 24-07-18  | time:      |  |

| CEMENT MIX RECIPES                     |  |        |       |      |  |
|--|--|--------|-------|------|--|
| Recipe n° 8                            | cipe n° 8  |        |       |      |  |
| Name:<br>Date:<br>Time:<br>ID MIXTURE: | Daniel<br>06-09-18<br>10:55 (finished mix) - 11:00 (casting time)<br>BC 2%_Sost_7D |        |       |      |  |
|  | Qua  | antity | % wt  |      |  |
| Cement "CEM   52,5 F                   | R Ultracem Italcementi"  | g      | 225.4 |      |  |
| Deionized Water                        |  | g      | 80.5* |      |  |
| Superplasticizer "Dynamon Sp1 Mapei"   |  | g      | 2.3   | 1%*  |  |
| Biochar                                |  | g      | 4.6   | 2%** |  |

\* Regarding the sum of the weight of cement plus that of the biochar

NOTE:

\*\* Starting from a quantity of 230g of cement

w/c ratio =

0.35

No ultrasonic bath

Mixture ok

|   | MIXING PROCEDURE  |          |       |  |  |
|---|---|----------|-------|--|--|
| First 1.5 minutes:<br>Next 1.5 minutes:<br>fifth minute:<br>sixth minute: | Speed motor 4 - Gradually pour <b>all</b> the cement<br>Speed motor 4<br>Speed motor 6<br>Speed motor 6 |          |       |  |  |
| Formwork removal  | date:   | 07-09-18 | time: |  |  |
| Tests 7 days  | expected date:<br>effective date:   | 12-09-18 | time: |  |  |
|   |   |          |       |  |  |
| Tests 28 days   | expected date:<br>effective date:   |          | time: |  |  |

| CEMENT MIX RECIPES                         |  |          |       |      |
|--|--|----------|-------|------|
| Recipe n° 8                                |  |          |       |      |
| Name:<br>Date:<br>Time:<br>ID MIXTURE:     | Daniel<br>06-09-18<br>11:15 (finished mix) - 11:18 (casting time)<br>BC 2%_S_Sost_7D |          |       |      |
| Materials                                  |  | Quantity |       | % wt |
| Cement "CEM I 52,5 R Ultracem Italcementi" |  | g        | 225.4 |      |
| Deionized Water                            |  | g        | 80.5* |      |
| Superplasticizer "Dynamon Sp1 Mapei"       |  | g        | 2.3   | 1%*  |
| Biochar                                    |  | g        | 4.6   | 2%** |

\* Regarding the sum of the weight of cement plus that of the biochar \*\* Starting from a quantity of 230g of cement

w/c ratio =

0.35

NOTE: No ultrasonic bath Mixture ok

|   | MIXING PR                         | OCEDURE   |       |  |
|---|-----------------------------------|---|-------|--|
| First 1.5 minutes:<br>Next 1.5 minutes:<br>fifth minute:<br>sixth minute: | Speed moto                        | Speed motor 4 - Gradually pour <b>all</b> the cement<br>Speed motor 4<br>Speed motor 6<br>Speed motor 6 |       |  |
| Formwork removal  | date:                             | 07-09-18  | time: |  |
| Tests 7 days  | expected date:<br>effective date: | 12-09-18  | time: |  |
| Tests 28 days   | expected date:<br>effective date: |   | time: |  |

| CEMENT MIX RECIPES                         |  |          |       |       |
|--|--|----------|-------|-------|
| Recipe n° 9                                |  |          |       |       |
| Name:<br>Date:<br>Time:<br>ID MIXTURE:     | Daniel<br>06-09-18<br>11:32 (finished mix) - 11:36 (casting time)<br>BC 2%_0.40_7D |          |       |       |
| Materials                                  |  | Quantity |       | % wt* |
| Cement "CEM I 52,5 R Ultracem Italcementi" |  | g        | 225.4 |       |
| Deionized Water                            |  | g        | 92*   |       |
| Superplasticizer "Dynamon Sp1 Mapei"       |  | g        | 2.3   | 1%*   |
| Biochar                                    |  | g        | 4.6   | 2%**  |

\* Regarding the sum of the weight of cement plus that of the biochar

\*\* Starting from a quantity of 230g of cement

w/c ratio =

0.40

NOTE:

No ultrasonic bath

Mixture ok

very fluid mixture

|   | MIXING PROCEDURE  |          |       |  |
|---|---|----------|-------|--|
| First 1.5 minutes:<br>Next 1.5 minutes:<br>fifth minute:<br>sixth minute: | Speed motor 4 - Gradually pour <b>all</b> the cement<br>Speed motor 4<br>Speed motor 6<br>Speed motor 6 |          |       |  |
| Formwork removal  | date:   | 07-09-18 | time: |  |
| Tests 7 days  | expected date:<br>effective date:   | 12-09-18 | time: |  |
|   |   |          |       |  |
| Tests 28 days   | expected date:<br>effective date:   |          | time: |  |





Master degree in architecture for sustainable design