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## CONSTRUCTION HISTORY AND PRESERVATION

## CONSTRUCTION AND BUILDING PERFORMANCE

**BUILDING AND DESIGN TECHNOLOGIES**

# INNOVATIVE CONSTRUCTION MATERIALS: GRAPHENE-BASED SMART COMPOSITES

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## Highlights

Smart concrete represents the latest generation of concrete mixtures. The use of nanoparticles turns the smart concrete sensitive to external stimuli. Graphene, used as nano-additive, is one of the most promising materials for improving the construction sector. For the first time, the use of an industrially scalable method to produce graphene and its use in cement composites is exploited. Different methods of graphene dispersion in water have been evaluated, and the best one, based on the use of a superplasticiser as a surfactant, can improve the flexural and compressive strength of 25% and 65%, respectively, compared to a plain cement paste.

## Abstract

Over the recent decades, the cement composites sector has been characterised by experimentation and production of the so-called intelligent concrete, that can adjust their characteristics by reacting to external stimuli. The ultimate evolution of the concrete performance has been achieved thanks to the use of multifunctional fillers, transforming a traditional concrete into a smart concrete. Graphene, a two-dimensional material, is opening new avenues for innovative applications in the construction sector. This paper reports the first results of using pure crystalline few-layers graphene as an additive in cement composites.

## Keywords

Cement Composites, Innovative Material, Graphene, Smart Concrete, High-Performance Building Elements.

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## 1. INTRODUCTION

The cement composites are among the most widely used categories of construction materials in the last century. [1]. The history of concretes has coincided with the evolution of construction techniques in the building industry. For example, from the classical age (e.g., the Roman domes made using the *betunium*), passing through the 19th century with first pioneering applications of reinforced concrete (e.g., the Weaver building mill, that is one of the first application of Hennebique system), to the maturity of the second half of the 20th century (e.g., the organic shape of the Musmeci bridge). In this century, a rapid technological evolution process led to creating products capable of achieving ever-higher levels of performance according to different construction requirements [2]. Nowadays, the high-level of performance achieved is also associated with "dynamic properties", i.e., the ability to react to environmental conditions changes by adapting its intrinsic characteristics [3].

The cement composites with such peculiarities take the name of “smart concretes”; these materials can respond to external stimuli, for example, thanks to the addition of nano-sized elements, defined as “functionalising” elements. The nano additives inclusion ensures, in addition to high “standard” properties, (e.g., mechanical strength, high durability, low water permeability), a new category of “innovative properties” (e.g., self-sensing, self-heating, photocatalytic). This new evolutionary phase and the consequent introduction of innovative products on the construction market can be considered a possible resource for a sector affected by the crisis at the beginning of 2010, and currently struggling to reverse the trend, restarting [4].

Among the functional nano-sized elements or nano-additives, the two-dimensional (2D) crystals (material with an atomic-scale thickness) are materials that could potentiate the physical-chemical properties of cement composites. Graphene is the best-known member of the 2D crystal family, and its particular properties turn it an exciting material to be tested as a nanofiller in cement composites, e.g., one of the strongest material ever measured with a Young’s Modulus of 1.0 TPa and a tensile strength equal to 130 GPa [5]. Although in recent years seldom applications for construction and building have been reported, using graphene as a functional nanofiller in cement composites [6].

On this basis, in this manuscript are reported applications of graphene used as a functional filler in cement composites, so-called smart concrete, and is explored a dispersion method capable of obtaining an improvement in flexural and compressive strength of 25% and 65% respectively, compared to the bare cement composite.

## 2. STATE OF THE ART

### 2.1. SMART CONCRETE AND GRAPHENE

The first smart concrete’s origin traces back to the experiments in the ’90s of the 20th century with the first “self-diagnostic” conglomerates. For example, the smart concrete can sense when a material reaches the critical breaking point, thanks to the use of carbon and glass fibres embedded in the cement matrix [7]. Over the years, increasing interest in improving the performance of the construction materials, e.g., more safety and durable infrastructures with low-resources consumption and reduced environmental pollution, have triggered the research on intelligent cement composites, resulting in more than twenty categories of smart concretes (e.g., self-healing, self-cleaning, self-adjusting, etc.) [8] (Fig. 1).

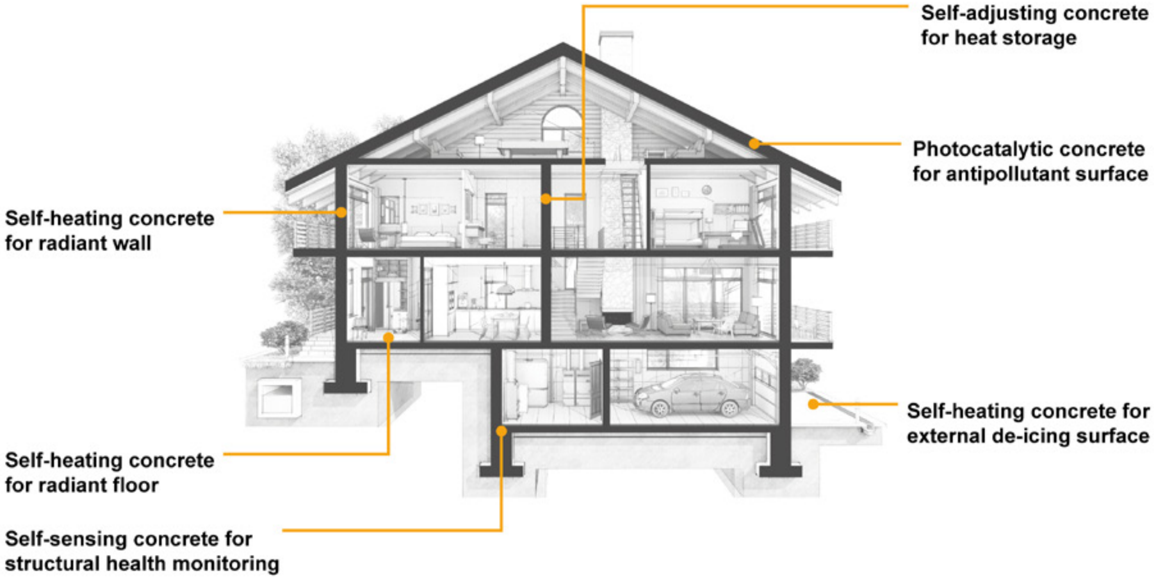

Fig. 1. *Possible applications of the smart concretes in a residential building. (Authors’ elaboration of an image made by Italcementi S.p.A. Source: Ufficio Stampa Italcementi S.p.A.).*

For example, the mixture of graphene and titanium dioxide ($TiO_2$) with cement can induce a photocatalytic response in the concrete reducing the pollution molecules that are in contact with the smart-concrete surface. [9] In this case, the photocatalytic property is due to the insertion of titanium dioxide nanoparticles, which absorb light, inducing the oxidation of the pollutant [10]. This photocatalytic process is activated just for a narrow electromagnetic spectrum, i.e., the $TiO_2$ nanoparticles (bandgap of 3.25 eV) absorb only the UV part of the solar spectrum. The addition of graphene improves the photocatalytic activity, widening the light absorption spectrum range of the $TiO_2$ nanoparticles [11]. The photocatalytic activity is currently exploited for the development of *Cemphene*, a technology already used in construction and architecture, for example, in the Dives in Misericordia Church (Rome) (Fig. 2).

Besides, graphene used as nano-additives induces interesting properties, i.e., self-sensing capabilities, that have a potential application in the field of monitoring the structural integrity of the constructions. Self-sensing is used to monitor *in situ*, exploiting the principle of piezoresistivity, i.e., transforming the mechanical stress into electrical signals, turning the cement into a sensor itself [12]. Similarly, in the so-called self-heating concrete, the graphene improves both electrical and thermal conductivities [13]. The cement material can be heated up exploiting the Joule effect, turning a traditional cement into a radiating element (Fig. 3); an alternative application is also the use in the roads/highways preventing the ice formation without using de-icing salts [13].

It is necessary to ensure an homogeneus dispersion of the additives into the cementitious matrix to fully exploit the enhanced properties of cement composites induced by the nano-additives. In this regard, the homogenisation is one of the main challenges of introducing graphene and other nano additives into smart concretes [14].

Fig. 2. *Richard Meier, Chiesa Dives in Misericordia, Rome, 2003. (Source: photo of Maria Giovanna Senatore).*

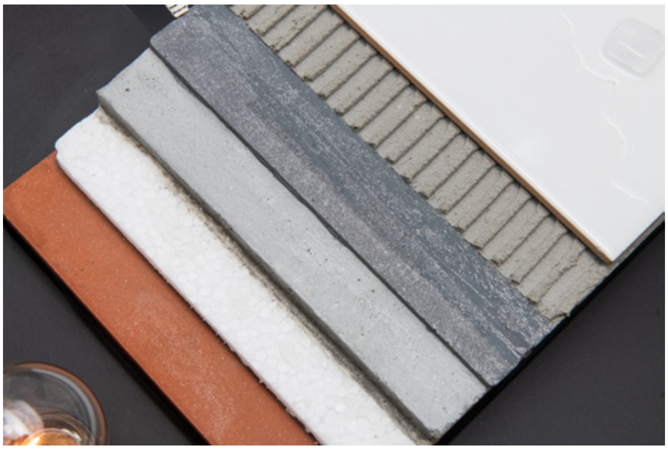

Fig. 3. *The Mock-up in the figure represents an application of a graphene-based self-heating concrete, made by Italcementi for the Mobile World Congress 2019. The graphene-based layer is placed below the adhesive composite for the tiles to obtain a better heating effect on the surface. (Source: Ufficio Stampa Italcementi S.p.A.).*

### 2.2. FILLER DISPERSION IN CEMENT MATRIX

There are several methods to obtain a homogeneous dispersion of nano additives in the composite, e.g., adding nanoparticles as a powder during the mixing phase, pre-dispersing the mixture using ultrasonication with or without surfactants. For instance, in the case of the fibre-reinforced concrete, the use of fly-ash or limestone fillers promotes the formation of cement paste, aiding the integration of the fibre into the concrete [15]. The aggregation of nanomaterials, clusters, can have an undesired effect in the composites' final performance, compromising or reducing the workability, mechanical resistance or flexibility, and other concrete properties [16].

Graphene, similar to the other carbon-based compounds (e.g., nanotubes, nanofibers, fullerene, amorphous carbon), is characterised by its hydrophobicity , wich is detrimental for the dispersion and stability of graphene in the cementitious matrix or paste. Consequently, it is necessary to modify the surface of graphene, rendering it hydrophilic, and thus, dispersible, and stable in the mixing water and cement paste [17]. It is common to use surfactants or dispersants to render the graphene dispersible and stable in water-based dispersions, i.e., they favour the formation of repulsive electrostatic charges on the surface of the flakes that preventing the clustering of fillers [18]. The advantage of using a surfactant relies on the fact that crystalline structure of graphene is not compromised. Consequently, the physical and chemical properties of graphene are preserved [19]. A physical force must be applied to mix the surfactant, and the cement matrix, usually ultrasonic bath, high energy stirring, or pressure homogenisers are used [20].

Over the last few years, the graphene production techniques have undergone continuous evolution, motivated by the search for processes that could combine high-quality control of the nanomaterial with industrial scalability. In over ten years of academic and industrial research, one of the most promising methods for graphene production, capable of combining quality and quantity, is the liquid phase exfoliation (LPE) of graphite, which allows the production of graphene at low cost at an industrial scale [21]. A promising technique to scale up graphene dispersion production is the use of pressure homogenisers, which recently have demonstrated the continuous production of few-layers graphene [22]. The main advantage deriving from this method is that it makes it possible to obtain large quantities of high-quality 2D materials (i.e., ensuring high quality of material, in terms of crystallinity and flakes morphology). Ensuring a massive production that the building sector demands.

Despite the potential application for the graphene-based smart concrete, the composite has not yet been tested in the conglomerate. This work presents the first study on dispersion methods and the effect of graphene mechanical properties of cement paste. In this regard, the use of the graphene obtained by the Wet Jet Milling (WJM) technique used as an additive in cement composites has demonstrated an improvement in flexural and compressive strength of 25% and 65%, respectively, compared to the bare cement composite, confirming the viability of further developments in the construction sector.

## 3. MATERIALS AND METHODS

Graphite flakes (+100 mesh, ≥75% min), N-Methyl-2-pyrrolidone (NMP, 99.5% purity), and Sodium deoxycholate (SDC, ≥97% (titration) and loss on drying < 5.0 %) were purchased from Sigma-Aldrich and used without further purification. Superplasticiser is a powder-based additive on acrylic modified polymer used for concrete with a low water/cement ratio. The cement used is 42.5R CEM II/A-LL, according to the European standard EN:197-1.

### 3.1. PREPARATION OF THE GRAPHENE DISPERSIONS

We exploited liquid-phase exfoliation of graphite [23] to produce graphene materials dispersed in water. In order to assess the best dispersion method in the cement matrix, three different water-graphene dispersions were made: two water dispersion of the few-layers graphene powder exfoliated in NMP, and one obtained using the same method but in an aqueous environment.

**Dispersions of few-layers graphene -FLG- (in the wet-jet milling, WJM) powder in water**. A mixture of 20 g of graphite flakes and 2 L of NMP is prepared. The mixture is placed in the container and mixed with a mechanical stirrer (Eurostar digital Ika-Werke). For the +100 mesh graphite, a 0.30 mm nozzle aperture is used. The processed sample is collected in a second container. The exfoliation process is repeated, passing the sample through the 0.20 mm nozzle and consecutively through the 0.15 and 0.10 mm diameter nozzle. Consequently, a homogeneous black dispersion is obtained. The solvent is then removed utilising a rotatory evaporator (Heidolph, Hei-VAP, at 4 mBar and 80°C), obtaining a dry powder-like material. The graphene in powder form is dispersed (0.25g) in deionised water with 2 g of superplasticiser, SP (G-SP), and without any surfactant (G). At last, the two samples are dispersed using an ultrasonic bath for 120 minutes.

**Dispersion of FLG in water**. A mixture of 20 g of graphite flakes, 2 g of Sodium, and 2 L of distilled water is prepared. The exfoliation process is performed following the same procedure as the case of FLG in NMP. The dispersion (G-SC) is used as produced.

### 3.2. PRODUCTION OF GRAPHENE/ CEMENT COMPOSITES

The three aqueous dispersions G, G-SP, and G-SC have been used as mixing water for the production of graphene-based cement pastes. Their two-dimensional additive loading is 1% by weight of the cement used. A cement water ratio of 0.5 was chosen.

The mixing procedure adopted for the casting of the samples is based on the European standard EN 196-1 modified (i.e., the phase concerning the addition of the sand has been eliminated). The following stages compose this process: water and cement are placed in the mixing bowl, and the addition is completed in 10 s; after, that the water and cement are in contact, the stirrer starts at a low speed (140 ± 5 rpm) and mixed the composite for 60 s, then for additional 30 s at high speed (285 ± 10 rpm); later, during a 90 s of pause, the paste adhering to the wall and bottom part of the bowl is placed in the middle of the container; at last, the stirrer continue the mixing at high speed for 60 s.

The three different mixtures (G/cement, G-SP/cement, G-SC/cement) are poured in polystyrene disk moulds with a 10.1 cm diameter and a thickness of 1.5 cm. After 24 hours, the cement paste disks are removed from the moulds and cured in deionised water for 14 days.

The most promising dispersion method, identified in the first experimental phase, was used to cast the samples to evaluate mechanical properties. The specimens are made of cement paste and had the composition indicated in Table 1.

The cement used is 42.5R CEM II with a water/cement ratio of 0.5. The mixing water had a graphene loading of 0.05%, 0.1%, 0.5%, and 1% by weight of cement used, thus defining six types of samples.

The quantity of surfactant was 30% by weight of graphene used, except for the control sample, where, as indicated by the manufacturer, the superplasticiser is equal to 0.25% by weight of the binder used.

After preparing the mixing of the water using graphene and superplasticiser, the dispersion was subjected to an ultrasonic bath for 120 min. Everything was mixed with the binder according to the speed and times indicated by the European standard EN 196-1 modified, using the abovementioned procedure.

Finally, the samples were poured into cylindrical steel moulds, and after 24 hours, they cured in water for 7 and 28 days. The specimens have dimensions characterised by a ratio between the base diameter and cylinder height of

| *Specimen* | *Cement* | *Water* | *Graphene* | *Superplasticiser* |
|---|---|---|---|---|
| | [g] | [g] | [g] | [g] |
| Control | 240 | 120 | // | // |
| SP | 240 | 120 | // | 0.06* |
| G-SP1 | 240 | 120 | 0.12 | 0.03 |
| G-SP2 | 240 | 120 | 0.24 | 0.07 |
| G-SP3 | 240 | 120 | 1.20 | 0.36 |
| G-SP4 | 240 | 120 | 2.40 | 0.72 |

Tab. 1. *Composition of the type of sample; note: *the quantity of superplasticiser, in this case, is 0.25% by weight of cement.*

two, to have comparable results and avoid results influenced by the slenderness of the sample.

Finally, to ensure more excellent reliability of the results, eight cylindrical specimens were cast for each type of cement paste: four were subjected to bending tests and four to compression one.

### 3.3. DISPERSION OF GRAPHENE AND MECHANICAL MEASUREMENT

The correct dispersion and microstructure evaluation were carried out using investigation techniques such as Raman spectroscopy and scanning electron microscope (SEM).

**Raman Spectroscopy.** Raman measurements were carried out using a Renishaw InVia micro-Raman spectrometer with a 50 × objective (numerical aperture of 0.75), an excitation wavelength of 514 nm line of an $Ar^+$ laser, and an incident power less than 1 mW.

**Scanning Electron Microscopy (SEM).** The cross-sectional morphology of the as-prepared composites was characterised by an analytical (low vacuum) scanning electron microscope using a JSM-6490LA SEM (JEOL) operating at 10 kV acceleration voltage. The acquired SEM images were further analysed using the image processing and analysis software ImageJ.

**Mechanical Measurements.** The flexural strength tests were carried out using an Instron 3365 with a three-point bending clamp and a 2kN load cell. The data are acquired using the Instron Bluehill Universal software. The compressive strength tests were carried out using a Metrocom material testing machines, with a resolution of 0.01 kN and a load cell of 600 kN. The data acquisition software was Metrocom-Dina960xp.

## 4. DISCUSSION AND RESULTS

### 4.1. DISPERSION OF GRAPHENE AND MICROSTRUCTURE

In order to assess the dispersion of graphene inside the cement matrix, three types of cement composite samples are prepared, i.e., G-SP/Cement, G-SC/Cement, and G/Cement, as stated in the materials and methods section. The G/Cement is intended as a control sample where no additives are used, being useful to assess the dispersibility of the graphene. The physical and chemical properties of the graphene obtained by WJM have been shown and discussed in several reports [24, 25].

The cement composites produced are characterised by Raman spectroscopy in mapping mode, giving a view of the particular distribution of the graphene flakes in the cementitious matrix. For this scope, a series of small pieces of cement pastes are sliced from the transversal section of each specimen. The sliced surfaces are analysed by Raman spectroscopy. In general, the most prominent Raman band of graphene obtained by liquid-phase exfoliation of graphite is the G band. Consequently, in the Raman analysis, the G band is used as a reference to assess the presence (or absence) of graphene in the composite. Figure 4a shows the Raman spectra for the three samples, i.e., G/Cement, G-SC/Cement, and G-SP/Cement, for the bare cement paste and the as-produced graphene powder. The Raman signal of few-layers graphene (FLG) flakes is diminished when embedded in the cement matrix. The Raman signal weakening is due to the light scattering induced by the cement. To assess the homogenous distribution or clustering of FLG inside the cement matrix Raman mappings are carried out in all the samples (Fig. 4b, 4c, 4d, 4e). In particular, the Raman mappings illustrate the presence, in red tones, (or absence, in blue tones) of FLG on the cementitious matrixes. To be specific, the mappings are plot following the normalised integral intensity of the G band. The circles indicate the zone where the representative spectra are taken, indicated in different colours. The inset pictures in the Cement, G/Cement, G-SC/Cement, and G-SP/Cement mappings correspond to the areas in the composites where the mappings where taken.

The mapping obtained for the dispersion of FLG without using a surfactant (G/Cement) shows that the graphene inside the cement matrix is not uniformly distributed, showing clusters of FLG (like red islands). Contrary, in the samples where surfactants or dispersants are used, i.e., the G-SC/Cement and G-SP/Cement samples, the red tonalities are distributed in the whole surface sample, indicating a more homogenous distribution of FLG in the cement matrixes.

Scanning electron microscopy (SEM) allowed verifying the Raman analysis. The SEM images (Fig. 5a, 5b, 5c, 5d) confirm the presence of aggregates and dispersed FLG in the cementitious matrixes. In particular, in the sample G/Cement, the presence of aggregates is evident (Fig. 5b).

Contrary, for the G-SC/Cement (Fig. 5c), the FLG displays a homogenous distribution in the cement matrix without forming aggregates. Moreover, the structure of the G-SP/Cement (Fig. 5d): not only the presence of clusters is scarce, but the FLG flakes are incorporated in the matrix with the formation of primary ettringite on the surface of the filler. The presence of this material testifies that the external surface of the FLG is a suitable place for the formation of cement hydration products [6]. Consequently, the nano additive is incorporated within the structural matrix of the cement composite.

Experimental tests have shown that G-SP/Cement and G-SC/Cement guarantee a homogeneous dispersion of the functionalising filler. Even if the presence of aggregates is limited, the use of superplasticiser is characterised by a twofold advantage, i.e., to use a commercially available and cost/effective product (compared to

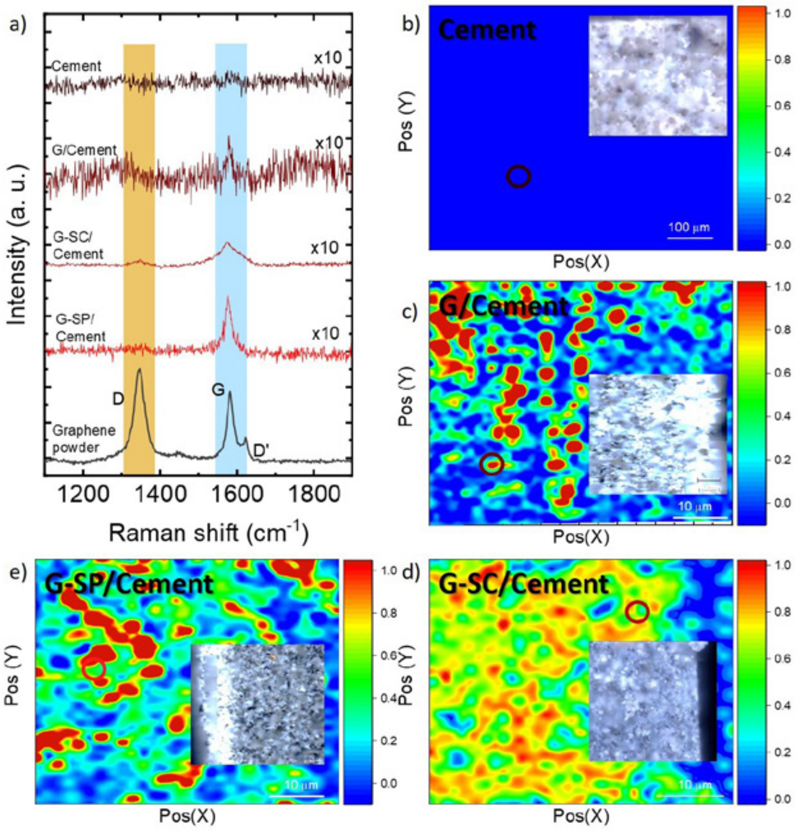

Fig. 4. *In clockwise order: the Raman spectra for the G/Cement, G-SC/Cement, G-SP/Cement samples, the bare cement and the as-produced graphene powder (a); Raman mapping of the bare cement paste (b), G/Cement (c), G-SC/Cement (d) and G-SP/Cement (e) samples.*

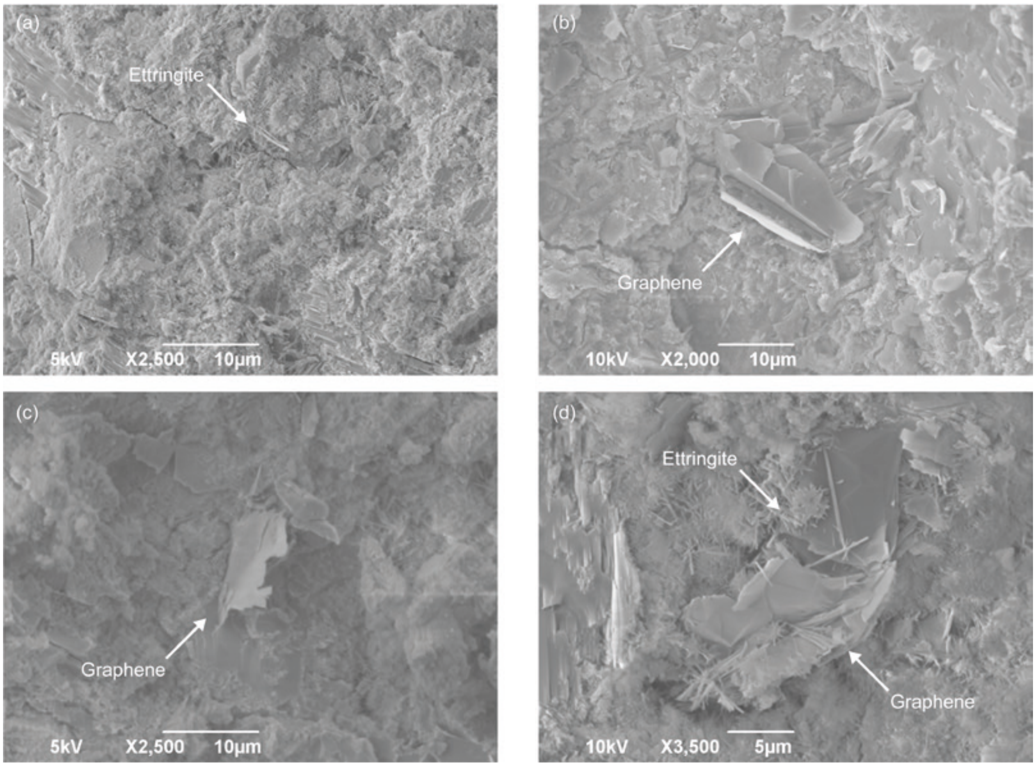

Fig. 5. *Images obtained using SEM microscopy of the fracture section of bare cement paste (a), G/cement (b) G-SC/cement (c), and G-SP/cement (d) samples.*

sodium deoxycholate, the former is ca. 2.56 €/kg [26], and the latter is between 600 and 1300 €/kg [27]).

## 4.2. MECHANICAL PROPERTIES

Mechanical tests are carried out just on the G-SP/Cement samples since it shows the most homogenous distribution among the samples prepared. These tests allow the evaluation of the effect of graphene on the formation of the cement microstructure and verify the possible presence of clusters, affecting the composite performance. Six types of cement composite samples are prepared, i.e., Control, SP, G-SP1, G-SP2, G-SP3, G-SP4, with increasingly graphene loading, as stated in the materials and methods section.

We used the three-point bending test to evaluate the flexural strength, and the results are presented in Fig. 6. The flexural strength results after 7 days of curing in water show that the use of the superplasticiser influences mechanical performance. In this regard, the sample with only the superplasticiser shows a flexural strength value of 10.3 MPa, which is an increase of 33.7% compared to the mechanical performance of the control sample (7.7 MPa). The addition of graphene further improves the flexural resistance; in fact, the G-SP3 sample, with a low load of nano additive, can reach the 11.7 MPa, with an improvement of 51.7% compared to plain cement paste.

After 28 days of curing, when the hardening accelerator effect due to the use of the superplasticiser has been exhausted, the difference in performance between the control samples and graphene ones is evident. Specifically, the samples G-SP3 and G-SP4 show improvements in

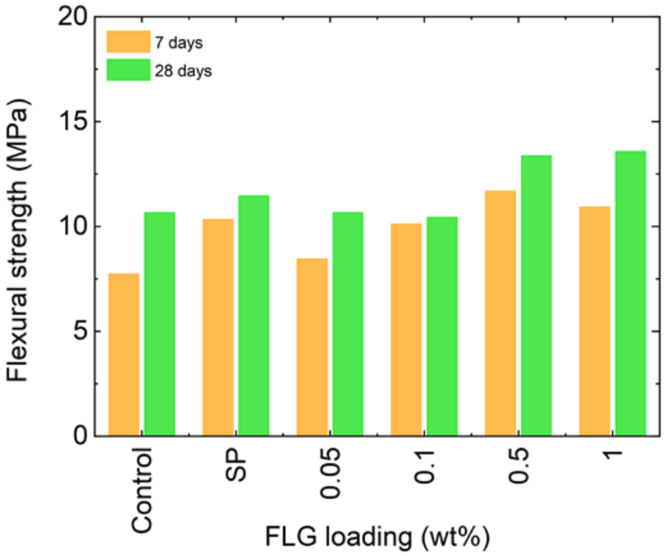

Fig. 6. *Flexural test results with curing periods of 7 and 28 days.*

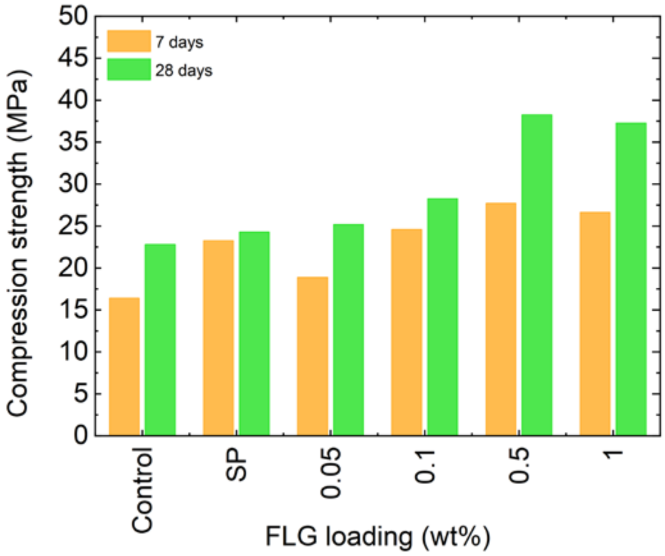

Fig. 7. *Compression test results with curing periods of 7 and 28 days.*

the flexural strength of 25.4% and 27.4%, respectively, compared to the control sample (10.7 MPa).

Subsequently, the graphene-based cement samples were subjected to compressive strength tests.

As seen for flexural strength, the results of compression testing after 7 days show the effect of the combined action of the superplasticiser and graphene. The control SP sample reaches a compressive strength value of 23.3 MPa, with an improvement compared to the Control sample of 41.7%. Also, in this case, the most significant increase in mechanical resistance at 7 days is reached with a graphene load of 0.5%; in fact, the G-SP3 sample has a maximum tensile strength of 27.7 MPa, 68.8% higher than the Control (16.4 MPa).

Finally, the same trend is present for samples after 28 days of curing: the SP-3 and SP-4 samples show an improvement of compressive strength of 67.7% and 63.3%, respectively, compared to Control (22.8 MPa).

The mechanical tests show that the use of the superplasticiser dispersion improves the mechanical performance of the cement paste, evidence of a correct dispersion within the material structure. The beneficial action of graphene is also boosted by the effect of the superplasticiser, which guarantees better performance on the first days of curing compared to bare cement paste. At the same time, it can be seen from the mechanical test of G-SP4, that it is not suitable to use high percentages of graphene. In this regard, the increment in flexural resistance is minimal (G-SP4 shows a flexural strength of 13.6 MPa, a negligible increase compared to G-SP3 value), and an increase in the cost of the composites, due to the higher load of graphene, is not worthwhile. Furthermore, the inferior performance of compressive strength compared to G-SP3, indicates the beginning of a more significant formation of clusters, that affect the mechanical performance of the conglomerate.

## 5. CONCLUSIONS

Nowadays, the conglomerates for construction are in a new evolution phase, developing innovative composites able to react to external stimuli (e.g., photocatalytic, self-sensing, self-heating concrete). These innovative composites are based on the use of nano additives, e.g., graphene. Despite the potential performance of the synergy graphene-cement, there is not yet a full application in the construction sector due to the high cost of the graphene production. Industrially scalable production, combined with the search of low-cost solutions, can contribute to developing innovative products increasingly demanded from the construction industry nowadays.

In this paper, high-quality graphene is used in terms of crystallinity, morphology, and low-production cost. The dispersion process exploits the use of an additive commonly used in the concrete industry, a superplasticiser. In this regard, three different graphene-water solutions were tested to evaluate the best dispersion method to ensure a correct homogenisation of the composite. Raman and SEM analysis have demonstrated that the use of a polymer-based superplasticiser promotes a correct graphene integration in the cement matrix.

The as-produced graphene flakes were used as filler in cement matrixes, evaluating then mechanical performances of the composites. This analysis showed that, for loadings of 0.5% by weight of graphene concerning the cement, increases in flexural and compressive strength of 25% and 65%, respectively, appear, compared to the simple cement paste sample.

In the next phases, the study and evaluation of technological systems' performance will be conducted by realising scale models of building components (e.g., screeds, panels).

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