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# STRENGTH RECOVERY OF NANO-REINFORCED CEMENT MORTARS AS PARAMETER OF SELF-HEALING EVALUATION: A METHODOLOGICAL APPROACH

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**Abstract:** In the present study the strength recovery of nano-reinforced cement mortars due to self-healing mechanism was examined. Graphene oxide nanoplatelets (GOnPs) were added in Ordinary Portland Cement (OPC) mortar mixtures in order to increase their resistance to crack propagation and strengthen the mixtures at the nano-scale. In order to examine the effect of GOnPs addition in the above parameters, a set of reference OPC mortars were also tested. The evaluation of the strength recovery was achieved by a new methodological approach focusing on the quantification of the damage degree and the healing capacity as expressed by the strength recovery after healing. Two different levels of damage (10% and 70% respectively) were induced and their effect on the self-healing capacity was examined. Overall, it was shown that the addition of GOnPs had a beneficial effect on the mechanical properties and allowed the introduction of higher levels of damage (up to 70%) to mortars without leading to specimen fracture. That was not possible for the plain OPC specimens. After autogenous healing the nanocomposites subjected to increased damage demonstrated 63% healing capacity, suggesting a partially recovered microstructure. Specimens damaged by 10% damage degree showed 100% healing capacity i.e. their strength was fully recovered when compared to the strength of reference undamaged specimens.

## 1 INTRODUCTION

During the past decade, research on the self-healing mechanism of cementitious materials has gained a lot of attention, as it could lead to the enhancement of concrete durability and the increase of the structures service-life.

Self-healing capacity is usually expressed by three main methodologies: crack-closing ratio [1], water-tightness [2], and strength recovery [3] after damage has been induced. One of the main issues related to the characterization of self-healing is the introduction of controlled, measurable and reproducible damage [4] as well as the evaluation of the mechanical strength recovery after healing.

Nevertheless, there are not yet specific standard methodologies for the assessment of the self-healing capacity. The interpretation of the results may constitute a real challenge when different parameters, such as the addition of healing agents, healing conditions or different assessment methodologies are to be compared [4].

The incorporation of GOnPs in the binder matrix can affect the material's mechanical properties by increasing resistance to crack propagation [5] and at the same time strengthen the mixtures at the nano-scale by reshaping the microstructure and decreasing the porosity [6].

The enhanced properties of GOnPs reinforced mortars could offer new insight on the self-healing mechanism and the strength recovery capacity of cement-based composites, regarding both damage degree and healing capacity measurements.

In this study the autogenic self-healing capacity of OPC mortars incorporating grapheme oxide nano-platelets (GOnPs) was examined. For this reason, a novel methodology for the assessment of strength recovery due to self-healing was developed and applied in the experimental setup. The results are discussed in comparison to a set of control OPC mixture.

## 2 MARERIALS AND METHODS

### 2.1 Preparation of mixtures

Two mortar mixtures were prepared using ordinary Portland cement (TITAN S.A.) and CEN standard siliceous sand as aggregate. In the first mixture graphene oxide nanoplatelets (GOnPs) were added. Since GOnPs are hydrophobic, their dispersion in a superplasticizer/water mixture was required. In this study the Sika Viscocrete-5500HP superplasticizer was used. The composition of the mixtures is presented in Table 1.

Prior to their addition in the mortar matrix the GOnPs were dispersed into the mixing water containing the superplasticizer. The resulting suspension was ultrasonicated for 1 h at a UIP 1000 hdT Hielser prob-sonicator. Following, the resulting suspension was mixed with the mortar matrix according to the ASTM C305.

For each mixture 24 prismatic specimens were cast (20x20x80 cm) and cured under high humidity controlled conditions (95%RH, $20^{o}$C). The specimens were subjected to controlled damaged after 7 days of curing and where subsequently cured under water for 28 days.

**Table 1**: Mixtures composition

| | OPC | GO | SP | w/c | b/a |
|---|---|---|---|---|---|
| OPC | 450 | - | 3.6 | 0.35 | 1/3 |
| GO | 450 | 0.9 (0.2%) | 3.6 (0.8%) | 0.35 | 1/3 |

### 2.2 Strength recovery assessment

In order to examine the self-healing capacity of the mixtures a new methodological approach was developed. The proposed methodology is based on two concepts: the control and quantification of the damage degree and the quantification of the recovered strength as the self-healing capacity.

Damage degree control: One group of specimens (n=8) were damaged by Hertzian stress (Fig. 1) after 7 days of curing. The same specimens were then tested by 4-point bending test in order to assess their residual strength, $f_d$, (Fig. 2). A second group of undamaged specimens (n=4) was also tested by 4-point bending test and the damage degree was then calculated according to the following formula:

$$D\% = [(f_R^0 - f_d)/f_R^0)]100\% \qquad (1)$$

where:
$D$%= damage degree
$f_R^0$=flexural strength of undamaged specimens

$f_d$=flexural strength of damaged specimens

Self-Healing capacity: Similarly, a third group of specimens (n=8) was damaged using the same levels of Hertzian stress (Fig. 1). This group was then cured under water and tested by 4-point bending test after the designated healing period (28 days) (Fig. 2). Their recovered strength was then contrasted to the measured strength of undamaged specimens (n=4) cured under the same conditions which simulate the full healing capacity (100%).

The healing capacity was calculated by the following formula:

$$H\% = [(f_h/f_R^1)]100\% \qquad (2)$$

where:

H%= healing capacity

$f_R^1$=flexural strength of undamaged specimens cured under the same healing conditions

$f_h$=flexural strength of healed specimens

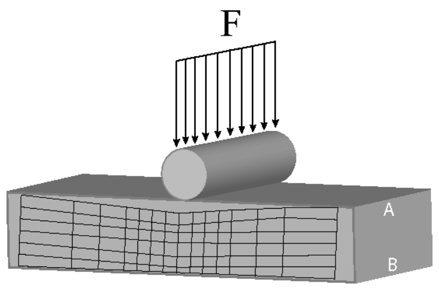



**Figure 1**: Hertzian contact stress.

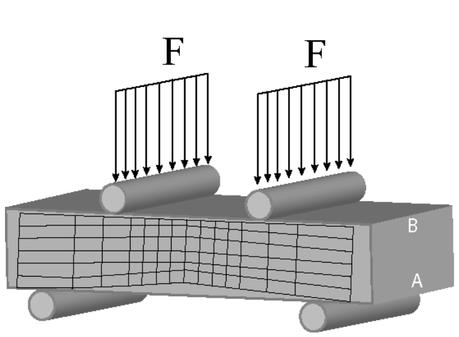



**Figure 2**: 4-point bending test

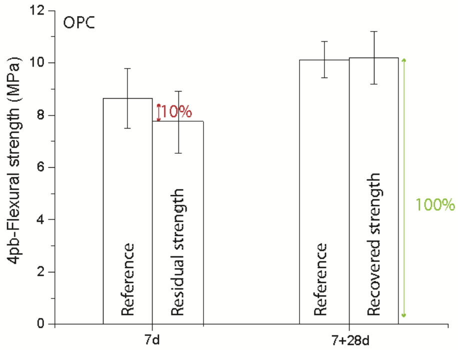
## 3 RESULTS

Mortar specimens with and without GOnPs were firstly damaged by Hertzian stress so as to examine the effect of damage degree on the self-healing capacity. Initially the OPC control specimens were subjected to the Hertzian contact stress test. To assure that the same level of damage was introduced the maximum load was kept constant. It was observed that a constant level of 10 % damage degree was the maximum possible applied, as higher stress resulted in OPC control specimens' structural failure (fracture). On the contrary, two levels of damage degree were induced in the GOnPs nanocomposites, 10% and 70%, respectively. That was a straight suggestion that the incorporation of GOnPs affects beneficially the toughness of the specimens, allowing for a 7 times higher damage degree without the presence of macrocracking (fracture).

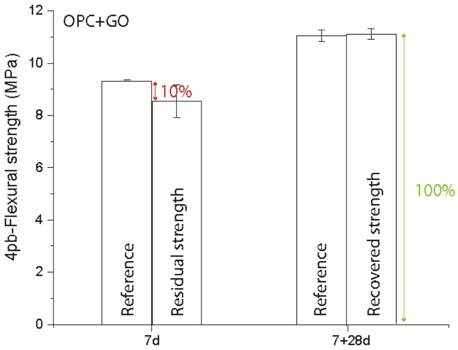
The damaged specimens were subsequently cured under water for another 28 days period to allow their autogenous healing. Their recovered strength was measured afterwards by 4-point bending strength tests. Figs. 3 and 4 depict the results of the samples without any damage at the age of 7 and 28 days (reference), the specimens subjected to controlled damage (residual strength) and the samples after self-healing (recovered strength) subjected to a 10% and 70% damage degree, respectively. Specimens damaged by 10% damage degree showed full strength recovery when compared to the strength of reference undamaged specimens, which is expressed as 100% healing capacity (Fig. 3). The healing capacity in both mixtures was the same irrespective of the presence of the GOnPs.

Nevertheless, when the damage degree was raised to 70% the healing capacity of the GOnPs nanocomposites was 63% (Fig. 4), suggesting a partially recovered micro-structure. It should be noted that for comparison purposes the self-healing period was kept constant in both damage levels. The application of a longer self-healing period is anticipated to further improve the micro-structure of the samples improving their mechanical response.

Considering that the self-healing process (in this experiment) is chemical/binder controlled mechanism, it can be easily concluded and highlighted the beneficial role of GOnPs nano-reinforcement on the damage withstand capacity of cement mixtures (thus providing the necessary time for healing to take place) and consequently on strength recovery and service life extension of damaged mortars.




(a)




(b)

**Figure 3**: Healing capacity of a) reference mortars and b) GOnPs mortars subjected to 10% damage degree.

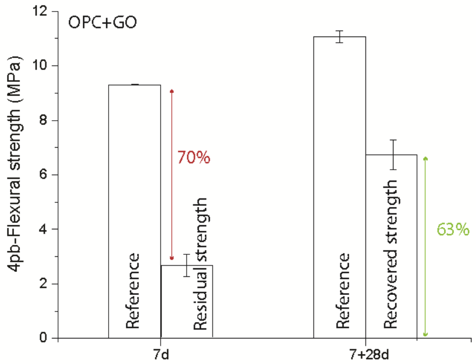



**Figure 4**: Healing capacity of GOnPs mortars subjected to 70% damage degree.

## 4 CONCLUSIONS

In this study the healing capacity of GOnPs nano-reinforced mortar composites was evaluated by the examination of their strength recovery after pre-determined damage has been introduced. A new methodological approach was implemented in order to allow the quantification of both damage degree and self-healing capacity. The main findings of this study are summarised below:

- GOnPs nano-reinforced mortar specimens demonstrated higher toughness, allowing the introduction of a 7-times higher damage degree than the OPC control specimens.
- Low damage degree (10%) resulted in a 100% strength recovery in all mixtures, irrespective of the presence of GOnPs.
- Higher damage degree (70%) was only possible on GOnPs nano-reinforced mortar specimens and resulted in 63% strength recovery.

Overall, it was shown that the incorporation of GOnPs in the cement matrix improves the mechanical properties of mortars and allows for higher damage degree without leading to specimen fracture. Moreover, it was shown that the damage degree is the main factor that controls the level self-healing efficiency at specific time-frame.

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