The Effect of Graphene Oxide on Cement Mortar

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Abstract. This paper investigates the effect of water dispersed- and powder Graphene oxide (GO) nanoparticle on fresh cement mortar, microstructure and mechanical strength after 3,7, and 28 days of curing. These properties were studied by treating the cement mortar with 0.03 wt%, 0.05 wt%, and 0.2 wt% GO of the cement weight combined with 0.8wt % polycarboxylate superplasticizer. The results show that the workability decreases as increasing the content of water dispersed GO. The heat of hydration is increased for both types of GO systems. The percent air content in 0.03 wt% and 0.05 wt% GO is almost constant, but increased from 3.2 % to 4.9 % in 0.2 wt% water dispersed GO. The increased air content has effect on poor compaction and workability. GO has the potential of accelerating the hydration process and enhance the early mechanical strength (3 and 7 days), but the workability seems to diminish the mechanical strength after 28 days of curing, particularly for the highest content of water dispersed GO. No distinct influence of GO on the microstructure. The overall results showed that the impact of water dispersed GO was found out to be higher than the powder GO.

1. Introduction
In recent years, the application of nanotechnology (1-100nm) has shown an impressive results in solving several engineering related problems in electronics and biomedicine. Through chemical and physical processes, nanomaterials improve material properties such as thermal, mechanical, electrical, lubricity and rheological. Comparing with the micro/macrosized particles, the surface area to volume ratio of the nanoparticle is higher Amanullah et al, (2009) [1]. Nanomaterials improve cement properties (Ruhal et al, 2011 [2], Ershadi, et al (2011) [3], Li, et al (2003) [4], Rui et al. (2015) [5]. Among others, Graphene is known for its superb material properties and has shown a great potential in other industries, such as electronics and polymer composites. Graphene is about 200 times stronger than steel [6]. Graphene Oxide (GO) is a derived from graphene. Wang et al 2015[7] have reported the influence of GO impact of of graphene oxide on the microstructure and mechanical strength of cement, which has potential of providing a nano-reinforcement. However, the research on GO is still at an early stage. This paper presents the effect of GO in the conventional cement mortar. The investigation is through various experimental studies.

2. Experimental works
Wang et al. 2015[7], reported enhanced mechanical properties with the GO 0.03 wt.% GO and 0.05 wt% of the cement weight. In this paper, 2.00 wt.% GO concentration were also evaluated to investigate the performance of high concentration on the mortar properties. The reference (control) cement mortar was formulated without nanoparticle additive. The impact of GO nanoparticle on the reference system
were studied by adding a 0.03 wt.%, 0.05 wt.%, 0.2 wt % GO nanoparticle. This section presents the description of cement mortar additives, cement mortar formulation, measurement and characterization

2.1. Materials and test methods.

Table 1 shows cement mortar ingredients used in the reference and GO nanoparticle treated cement mortar. The mix design was according to the procedure described in NS-EN 196-1:2016 – Methods of testing cement [8]. The reference cement mortar was formulated by mixing 450gm standard cement, 1350gm CEN Reference Sand and 3225gm Water. The GO treated mortar was formulated by adding GO powder and GO water dispersed in the reference mortar system. The cement mortar ingredients were mixed with a Hobart N-50 mechanical mixer for 5 min.

Cement mortar formulation

1. Reference (Ref.) = 450gm cement, + 1350gm Sand + 3225gm + Water +0.8 wt.% Superplastizer
2. Graphenea (Water dispersed) = Ref + [0.03 wt.%, 0.05 wt.%, 0.2 wt.%] GO
3. Graphitene (Fine powder) = Ref + [0.03 wt.%, 0.05 wt.%, 0.2 wt.%] GO

| Materials                | Quantity [g] |
|--------------------------|--------------|
| Standard cement          | 450 ± 2      |
| CEN Reference Sand       | 1350 ± 2     |
| Water                    | 3 225 ± 1    |
| MasterGlenium ACE 434    | 0.8 wt.% of the cement weight |
| Superplasticizer (SP)    |              |
| Graphene Oxide (GO)      | [0.00, 0.03, 0.05, 0.2] wt.% of the cement weight |
| Graphenea & Graphitene   | [0.00, 0.03, 0.05, 0.2] wt.% of the cement weight |

The experimental test methods was based on NS-EN 196-1:2016 – Methods of testing cement “Part 1: Determination of Strength” [9]. The prime objective is to investigate the effect of graphene oxide (GO) on:

- Fresh cement mortar (Workability, Air content, Density, and Heat of hydration)
- Mechanical properties (Flexural strength, Compressive strength, Splitting Tensile Strength and Ultrasonic Velocity)
- Microstructure (The interfacial transition zone & the bulk cement paste)

2.2. Results and discussion.

The effect of GO on the fresh mortar and on the cement specimen was investigated through the methods presented in the previous section. The results are summarized as follows.

2.2.1. Effect of GO on the fresh cement mortar

Workability

A reduced workability is one of the challenges with graphene oxide. Consequently, it can cause poor compaction and therefore influence the mechanical properties. The impaired workability can be explained by a reduced amount of free-water since GO has a significant high specific surface which can result in a high water absorbency. In literature it is documented that the reduced workability has been solved by adding superplasticizer (SP). However, the potential side-effects due to chemical reaction between SP and GO are unknown to the investigators.

The results from the mini-flow test are presented in Table 2. Results show that for the 0.03wt.%, 0.05wt% and 0.2wt.% concentration of the Graphenea (water dispersed), the mini-flow diameter of the fresh mortar decreased by 5.5%, 18.9% and 60.6%, respectively. On the other hand in the Graphitene
(fine powder) system, the mini-flow diameter decreased by 3.5%, 2.8% and 3.5%, respectively. From this test results one can observe that there is a correlation between the content of Graphenea (water dispersed) and the reduced flow diameter. For Graphitene (fine powder), there is only a small change in the flow diameter with increasing content of GO, but no correlation between the percent GO and the flow diameter. The cement mortar with Graphenea 0.20% & SP 0.8% had a stiff plastic consistency after mixing. The flow diameter was measured to only 10 cm, which equal to zero flow since the bottom diameter of the cone is 10 cm, see Figure 1.

According to Gong et al. (2015) [10], the mini-flow diameter is reduced by 34.6 % with a GO content of only 0.03 wt%. Similar results are also reported by Pan et al. (2015) [11], Shang et al. (2015) [12], and Tang et al. (2014) [13]. Shang et al. (2015) [12], also observed that the mini-flow diameter decreases with an increasing content of GO. The mini-flow diameter is reduced by 36.2 % with a GO content of 0.08 wt%. According to Chuah et al. (2014) [7] [14], similar results are also observed with other nanomaterials, such as nano silica and carbon nano tubes.

![Figure 1. Mini-Flow Test - 0.2% Graphenea & 2.0% SP](image)

**Air content**

The air content results are presented in Table 2. Results show that for the 0.03wt.%, and 0.05wt% Graphenea increased by 0.2% concentration and in 0.2% increased by 1.7%. This can be explained by the stiff plastic consistency that was observed during the mini-flow test. On the other hand, as the Graphitene (fine powder) concentration increases (0.03wt.%, 0.05 wt.% and 0.2wt.%), the air content decreased by 0.7%, 0.7% and 0.3%, respectively.

The densities of all the mortar are within the range of 2.22 to 2.3 kg/m³. The small reduction in density for Graphenea 0.2% seems to be a result of the reduced workability, which was observed during the mini-flow test.

**Heat of hydration**

According to the author’s knowledge, the influence of GO on the heat of hydration is not clarified well in previous research. For instance, Zhao et al. (2017) [8] [15] reported that GO increases the maximum cement hydration exothermal rate, while Wang et al. (2015) [6] observed the opposite.

Figure 2 presents the temperature developments of different mortar compositions. Graphene oxide (GO) treated cement mortar shows an earlier temperature development and a higher maximum temperature. These results indicate that graphene oxide might have chemically reacted with the cement mortar components, and hence results in increasing the exothermal heat development. The maximum (peak) temperatures with respect to the reference mortar are presented in Table 2. The results also indicate that the impact of water dispersed GO (Graphenea) on the temperature development is higher than the GO in powder form (Graphitene). For instance, in the 0.05wt.% GO additive systems, the water dispersed GO (Graphenea) and powder form (Graphitene) increased temperature development by 5.4% and 2.9%, respectively. Similarly, in the 0.2wt.% the temperature development increased by 2.6% and 0.7%
respectively. One can observe a non-linear effects with respect to concentration. However, more studies required to come to a conclusion. There is also a correlation between the content of the water dispersed GO (Graphenea) and maximum (peak) temperature.

Figure 2. Temperature development during 3 days of curing

Table 2. Fresh mortar properties

| Material                  | GO [wt.%] | SP [%] | Mini-Flow Test [cm] | Air content [%] | Density [kg/m³] | Temperature Max(peak) [°C] |
|---------------------------|-----------|--------|---------------------|-----------------|----------------|--------------------------|
| Reference                 | 0         | 0.8    | 25.4                | 3.2             | 2299           | 40.8                     |
| Graphenea                 | 0.03      | 0.8    | 24.0                | 3.4             | 2264           | 42.3                     |
| Water dispersed           | 0.05      | 0.8    | 20.6                | 3.4             | 2262           | 43.0                     |
|                           | 0.20      | 0.8    | 10.0                | 4.9             | 2223           | 43.4                     |
| Graphitene                | 0.03      | 0.8    | 24.5                | 2.5             | 2278           | 41.3                     |
| Fine powder               | 0.05      | 0.8    | 24.7                | 2.7             | 2278           | 42.0                     |
|                           | 0.20      | 0.8    | 24.5                | 2.8             | 2261           | 41.1                     |

2.2.2 *Effect of GO on Mechanical Properties:*

*Flexural Strength:*

The flexural strength of the GO treated and untreated mortar were measured at three, seven and 28 days of curing and the results are presented in Table 3. Graphene oxide seems to improve the early flexural strength after three and seven days, but have no effect at 28 days. No distinct variation between the content or type of graphene oxide and the flexural strength is observed. The percent flexural strength development from three to 28 days is also lower compared to the reference mortar.
The improved flexural strength after three and seven days can be a consequence of the increased heat of hydration. This indicates that graphene oxide has a potential of accelerating the hydration process which increases initial flexural strength development.

**Strain Distribution – Verification of the Three-Point Bending Test**

The typical strain distribution during the three-point bending test is presented in Figure 3A. The red area indicates that the strain arises and propagates in a very concentrated part of the specimen. The prism ends are not subjected to substantial strain during the three-point bending test, and therefore, validates the method of using the prism ends to determine the compressive strength.

![Figure 3: Three-Point Bending (A) and Compressive (B) Test Transverse Strain distribution in x-direction](image)

**Compressive strength**

The compressive strength after three, seven and 28 days of curing is presented in Table 3. Graphene oxide increases the early compressive strength after three and seven days, but have no effect after 28 days of curing. In addition, the compressive strength for Graphenae 0.2% is reduced by 8 % after 28 days, while compressive strength for Graphenea 0.2% & SP 2.0% is improved by 7 % after 28 days. This indicates that the reduced workability has a considerable effect on the compressive strength and can result in a variation of 15 %.

**Strain Distribution – Verification of the Compressive Strength Test**

The strain distribution during the compressive strength test is presented in Table 3. The red zones, which indicate a relatively higher substantial strain compared to other parts of the specimen, are first initiated in the middle of the specimen, as illustrated in Figure 3B. The strain distribution propagates then towards the bottom and the top part. The observed strain distribution and the propagation is equivalent to a traditional compressive strength test with cubes, and the selected method of determining the compressive strength can be considered as valid.

**Splitting Tensile Strength Results**

The splitting tensile strength results compared to reference mortar are presented in Table 3. The results show an enhanced splitting tensile strength after three and seven days of curing for the cement mortar containing Graphenea. The highest enhancement is 19 % for the cement mortar containing 0.2 wt.% Graphenea and after three days of curing. Moreover, this enhancement seems to decline during the hydration process, and at 28 days the splitting tensile strengths are almost equal or less than the reference mortar. The results are almost equivalent to the reference mortar for the cement mortar containing 0.03wt.% Graphitene. For the other mortar compositions containing Graphitene, the splitting tensile strength is reduced by 6 % and 8 % at 28 days. The results indicate that graphene oxide produced by Graphena can accelerate the strength development in the early stages of the hydration process. The strength development seems to decline after three days of curing and no improvements are obtained at 28 days.
Strain Distribution – Verification of the Splitting Tensile Test

According to the theoretical cylinder stress distribution, the top and bottom part of the cylinder face are subjected to compression, while the middle is exposed to tension. Figure 4 presents the horizontal strain evolution on the cylinder face during the splitting tensile test. The image shows no substantial strain near the edges of the cylinder, while the red areas in the middle indicate strain. The strain evolution arises first in the middle of cylinder face and propagates outwards to the cylinder edge. These observations can be associated with the theoretical cylinder stress distribution. However, it is important to emphasize that the presented theory of the splitting tensile test is related to stress through the whole cylinder, and the images express the actual strain distribution observed on the cylinder face.

Figure 4: Strain evolution ($\varepsilon_{xx}$) during the splitting tensile test

Three virtual strain gauges were arranged as illustrated in Figure 5. The corresponding strain evolutions are presented in Figure 6. The virtual strain gauge positioned in the center of cylinder face (red) show substantial larger strain compared to those located close to the edges (blue & green). Moreover, the bottom strain gauge (green) shows a negative strain in the earlier stages of the strain evolution. These results indicate that the selected setup for the splitting tensile strength test provides equivalent stresses as for a conventional splitting tensile test with a larger cylinder and different loading rate.
Ultrasonic Velocity Test
The ultrasonic velocity test is non-destructive method, which determines a special type of modulus of elasticity, and should not be confused with the more conventional E-modulus or Young’s Modulus (i.e. the ratio of stress to strain). The sonic velocity is determined from the travel time and the length of cement plug. The modulus of elasticity is calculated as [16]:

\[
M = \left( K + \frac{4}{3}G \right) = V_p^2 \cdot \rho \tag{1}
\]

\[
Volume = \pi r^2 L
\tag{2}
\]

\[
\rho = \frac{mass}{volume}
\tag{3}
\]

\[
V_p = \frac{L}{\Delta t} = \frac{10^{-6} \cdot L}{1000 \Delta t} \tag{4}
\]

Where \( M \) is the modulus of elasticity, \( K \) is the bulk modulus and \( G \) the is shear modulus, \( \rho \) is density and \( V_p \) is compressional sonic velocity.

The Modulus of Elasticity (\( M \))
The modulus of elasticity (\( M \)) after seven and 28 days of curing is presented in Table 3. The results show a reduced modulus of elasticity for the mortar cylinders containing Graphenea, especially at 28 days of curing.

Detecting embedded air voids through sonic velocity
The ultrasonic velocity test results can also indicate how the reduced workability influences the amount of embedded air voids in the mortar. Since the sonic sound is not transmitted through air bubbles and voids, the sonic travel time will increase if large air bubbles or voids are located in the sonic travel path [4]. Reduced sonic velocity indicates possible embedded air voids. The sonic test results take the density into account, and can therefore not be used to quantify possible air voids.

Sonic Velocity
The sonic velocity is reduced for the mortar cylinders containing Graphenea, which indicates a higher content of embedded air bubbles and voids. Furthermore, the results from the density of hardened mortar and the mini-flow test indicates both directly and indirectly similar observations.

### Table 3. Mechanical hardened mortar properties

| Material          | I % Graphenea | Flexural Strength [MPa] | Compressive Strength [MPa] | Splitting T. strength [MPa] | Hardened Density [kg/m³] | E modulus [GPa] |
|-------------------|---------------|-------------------------|---------------------------|-----------------------------|--------------------------|------------------|
| Days              | 3 | 7 | 28 | 3 | 7 | 28 | 3 | 7 | 28 | 7 | 28 |
| Reference         | 0 | 6.1 | 6.9 | 7.8 | 42 | 48 | 61 | 3.2 | 3.9 | 4.9 | 2364 | 44.2 | 46.5 |
| Graphenea         | 0.03 | 7.2 | 7.4 | 6.8 | 45 | 52 | 58 | 3.4 | 3.9 | 4.7 | 2348 | 42.7 | 43.6 |
| Water disp.       | 0.05 | 6.6 | 7.3 | 7.9 | 45 | 51 | 59 | 3.4 | 3.9 | 4.5 | 2340 | 43.7 | 41.6 |
| Graophitene       | 0.20 | 6.9 | 7.5 | 7.7 | 43 | 48 | 56 | 3.8 | 4.1 | 5.0 | 2282 | 41.6 | 42.5 |
| Fine powder       | 0.03 | 6.6 | 7.2 | 7.6 | 42 | 51 | 60 | 3.1 | 3.8 | 4.9 | 2362 | 44.9 | 47.6 |
|                   | 0.05 | 7.1 | 7.3 | 7.9 | 43 | 51 | 61 | 3.0 | 3.6 | 4.7 | 2361 | 45.2 | 48.4 |
|                   | 0.20 | 6.6 | 6.8 | 7.5 | 43 | 50 | 57 | 3.0 | 3.7 | 4.5 | 2362 | 41.6 | 48.6 |

2.2.3 Effect of GO on the Microstructure – SEM & EDS

Graphene oxide sheets were only observed by the scanning electron microscope (SEM) in a sample taken from the cement mortar containing 0.05wt.% Graphenea. The low dosages of GO (0.03 wt.%, 0.05 wt.%, and 0.2 wt.% of cement weight) and the small sample size can explain why graphene oxide sheets were not observed in the other mortar compositions. The graphene oxide sheets were verified by analyzing the bright objects with the Energy Dispersive X-ray spectroscopy. The analyzed bright objects were selected because they did not look like traditional hydration products and because of their particular shape.

The graphene oxide sheets shown Figure 7 are observed at different locations on the sample. The size of the graphene (width or length) varies approximately from 0.865 μm to 16.7 μm (diameter of a human hair is approximately 17 to 181 μm). Figure 7 shows a wrinkled graphene oxide sheet located next to a small air bubble. The percent element composition is presented in Table 4. The element analysis shows a carbon content of 64.61 % and 16.34 % oxygen. The element composition indicates with a high probability that the observed object is a graphene oxide sheet or sheets. Figure 8 and Figure 9 illustrate similar observations as in Figure 7 with element analyses (Table 5 & Table 6). Moreover, the GO sheet observed in Figure 8 has a more distinct polyhedron shape compared to Figure 7. The smallest observed graphene oxide sheet is illustrated in Figure 9. Moreover, similar wrinkled-shaped graphene oxide sheets have also been observed in previous research, but the element analysis showed a larger content of calcium (Ca).

No distinct variation in the interfacial transition zone (ITZ) between the different samples were observed, except for the graphene oxide sheet located in the ITZ for the cement mortar containing 0.05wt.% Graphenea. This is illustrated in Figure 8.
Figure 7. SEM picture of cement specimen I

Table 4. Element Analysis

| Element | wt.% | At % |
|---------|------|------|
| C K     | 64.61| 76.30|
| O K     | 16.36| 14.50|
| NaK     | 6.30 | 3.88 |
| MgK     | 0.74 | 0.43 |
| AlK     | 0.73 | 0.38 |
| SiK     | 3.00 | 1.52 |
| S K     | 1.34 | 0.59 |
| CaK     | 6.27 | 2.22 |
| FeK     | 0.65 | 0.16 |
| Total   | 100.00 | 100.00 |

Figure 8. Graphene Oxide Sheet located in the ITZ & Spectrum II

Figure 9. The smallest observed Graphene Oxide sheet. Spectrum III is located in the center of the bright object
Table 5. Spectrum II - Element Analysis

| Element | wt. % | At % |
|---------|-------|------|
| C K     | 73.89 | 83.03|
| O K     | 11.89 | 10.03|
| NaK     | 5.95  | 3.49 |
| MgK     | 0.98  | 0.54 |
| AlK     | 0.97  | 0.48 |
| SiK     | 0.88  | 0.42 |
| S K     | 2.73  | 1.15 |
| CaK     | 1.97  | 0.66 |
| FeK     | 0.74  | 0.18 |
| Total   | 100.00| 100.00|

Table 6. Spectrum III - Element Analysis

| Element | wt. % | At % |
|---------|-------|------|
| C K     | 55.74 | 70.05|
| O K     | 18.57 | 17.52|
| NaK     | 5.40  | 3.54 |
| MgK     | 0.90  | 0.56 |
| AlK     | 1.19  | 0.67 |
| SiK     | 4.97  | 2.67 |
| S K     | 1.60  | 0.75 |
| CaK     | 10.31 | 3.88 |
| FeK     | 1.32  | 0.36 |
| Total   | 100.00| 100.00|

3. Summary
In this paper, the effects of graphene oxide (GO) on the cement mortar were studied through experimental work. The overall results showed that the impact of water dispersed GO was found out to be higher than the powder GO. The main results are summarized as follows.

- The workability is reduced by the increasing content of water dispersed GO, but is less affected by fine powder GO.
- The consistency of fresh mortar with the highest content of water dispersed GO (0.2 wt.%) is categorized as stiff plastic.
- GO has almost no effect on the content of air in the fresh mortar, except for the cement mortar with the highest content of water dispersed GO (0.2 wt.%).
- The 0.2 wt.% water dispersed GO and powder GO decreased the density of the mortar by 3.5% and 1.7% respectively.
- The 0.2 wt% water dispersed GO increased the heat of hydration than the other concentration.
- GO enhances the flexural strength after three and seven days, but has no effect at 28 days. The results show no correlation between the content of GO and the flexural strength. Moreover, the highest improved flexural strengths were in the range of 9-17% and observed after three days of curing.
- The effect of GO on the compressive strength corresponds to the flexural strength. Except for 7% improvement for the cement mortar with 0.2 wt.% water dispersed GO and a content of polycarboxylate which provides an equivalent workability as the reference mortar (additional mortar composition).
- Water dispersed GO increases the splitting tensile strength after three days of curing, but the enhancement decreases after seven days, and no effect at 28 days of curing. In addition, the powder GO has almost no influence on the splitting tensile strength at three, seven and 28 days.
- No distinct variations in the dynamic modulus of elasticity are detected after seven days of curing. At 28 days of curing the water dispersed GO reduces the dynamic modulus of elasticity, and the opposite effect is observed for fine powder GO. Moreover, the reduced sonic velocity for cement mortar with water dispersed GO indicates an increased content of air voids.
- SEM and X-ray detector have shown that Graphene oxide sheets size varies from 0.865 μm to 16.7 μm. The element analysis showed a high content of carbon (55.74 - 73.89 wt.%) and oxygen (11.89 – 18.57 wt.%). Furthermore, no distinct change in the interfacial transition zone or the bulk cement paste have been observed, except for the presence of GO sheets.

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