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# EFFECT OF GRAPHENE OXIDE ON THE MECHANICAL STRENGTH AND MICROSTRUCTURE OF CEMENTITIOUS MATERIALS

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## ABSTRACT

Cement-based concrete is used commonly for numerous constructions. Even though, cement has superior properties and great performance, its inherited brittleness is a major setback that needs more investigation for improvement. To eliminate this problem different types of additives, fibers, supplementary cementitious materials and nanomaterials have been used. One of these promising nanomaterials is graphene. This study is to investigate the feasibility of using graphene as a nano-additive to the cement based materials combined with a common used nano-additive such as silica fume as a dispersant. To conduct the study, eleven different mortar mixes were cast to investigate microstructure and mechanical properties of the cement mortar materials which were compared to benchmark samples. The studied parameters were ratios of both silica and graphene. Based on compressive and tensile strength, the results show that graphene can be thought of having the potential to be used as nano-reinforcement to improve the mechanical properties of cement based materials, provided that a proper amount of the dispersion agent is used.

***Keywords:*** Nanomaterial; Graphene oxide; Silica fume; Additive; Cement.

## 1. Introduction

Despite the fact that cementitious materials have shown great properties, they are also semi-brittle materials with very low tensile strength and reduced strain capacity [1]. To overcome these defects, different types of additives, fibers, supplementary cementitious materials, and nanomaterials have been used [2-5]. The studied nanomaterials included the three principal shapes; 0D nanoparticles, such as Nano silica; 1D nano fibers, such as carbon nanotube; and 2D Nano sheets, such as graphene and graphene oxide (GO) [6].

Pure graphene is a monolayer of carbon atomic crystal in two-dimensional plane arranged in a hexagonal lattice. Theoretically the tensile strength of a single graphene sheet of 130 GPa and it has a theoretical modules of elasticity of 1 TPa. At this scale the material produced has superior mechanical properties. It is nearly 300 times stronger than steel. Moreover, it is a light material weighting about 0.77 milligrams/m$^2$, and can be considered as a giant molecule with a large surface area available for chemical modification. It also has a good dispensability in water especially graphene oxide (GO) [7].

GO is manufactured in labs by Hummer's method. In this method the strong oxidation of graphite weakens the π- π stacking between successive layers, where oxygenated functional groups are inserted onto the surface of the produced GO sheet. Due to its oxygen containing hydrophilic functional groups, it can easily disperse in water than any other carbon nanomaterials, such as carbon nanotubes [8]. These functional groups physically adsorb the metal ions released from cement hydration products [9], and also form strong bonds with the cement matrix and reduce the probability of sliding under the effect of mechanical loads [10]. Moreover, the wrinkled morphology of the GO sheets offer nucleation sites for the cement hydration products at early ages [6]. The excellent mechanical properties of the GO sheet are also advantageous that can be exploited in improving the produced cement composite. Such intrinsic properties makes it has the potential for being a perfect reinforcing material to bridge cracks [11].

Recent studies have shown that introducing a mild percentage as 0.05% of GO by weight of cement could increase both the compressive strength and the flexural strength of the cement paste by an amount ranges from 15% to 33% and 41% to 59%, respectively. However, for the hardened mortar the increase in the compressive strengths were 43.2% , 33% and 24.4% at the ages of 3 ,7 and 28 day, respectively, while that of the flexural strengths were 69.4% , 106.4% and 70.5% at the ages of 3 ,7 and 28 days, respectively [12]. The same improvement in mechanical properties of cement paste could be achieved by incorporating another carbon nanostructure; such as carbon nano tubes, with the GO. A very small portion of 0.025% by weight of cement for each of the two materials could enhance both flexural and compressive strength of cement paste by up to 24.21% and 21.13%, respectively [13].

Incorporating GO into cement paste has been also reflected on the stress strain curve of the GO-cement composite; a mild slope in the post peak zone minimizing the probability of sudden failure [7]. That is the 2D GO sheets minimize crack propagation, and restrict sub-micro cracks from transformation to micro crakes [14]. The enhancement has been also reflected on the damping capacity and crack propagation [15].

The GO enhances also the transport properties and increases the long-term durability of concrete structures with only a small fraction of 0.01 to 0.03% by weight of cement. The GO-cement composite can effectively hinder the ingress of chloride ions and aggressive elements. This is due to its catalytic effect on the hydration process which results in decreased pore volume, and hence improves the transport properties and the corrosion resistance of the cementitious materials [16, 17]. Mercury intrusion (MIP) tests also showed that the addition of GO agglomerates shifts the differential pore volume peak towards finer pores [11]. Results also point that the addition of GO to Portland cement paste increases non-evaporable water and calcium hydroxide contents, consequently optimizing the degree of hydration in cement paste [18].

The effectiveness of a nanomaterial in enhancing mechanical properties is directly affected by the quality of its dispersion in the cement matrix. To serve this purpose, silica fume (SF) was incorporated to mechanically spread the GO nano sheets away from divalent calcium ions in cement past. The results of both the microstructure analysis and the mechanical testing prove that the dispersion of the GO nano sheets was improved by adding a proper amount of SF (2-10) % to the cement matrix. However, using large amount would have a reversing effect on the mechanical properties of the GO cement composite. This is caused by the blocking effect between the GO and the hydration products [19]. A recent study suggests that the adequate amount of the SF should roughly have the same specific surface area of the GO, but it did not provide a guideline to estimate such value [20].

This study is concerned with the possibility of using GO as a nano additive to cementitious materials. The effects of incorporating GO into cement mortar on the workability, hydration process, mechanical properties and microstructure is investigated. The SF is used to enhance the GO dispersion in the cement matrix. To serve this purpose, eleven different mixes have been prepared and tested on the ages of seven, twenty eight and ninety days. The results were then compared to benchmark samples to understand how GO and SF might work to enhance the properties of the cement matrix.

## 2. Experimental work

### *2.1. Procedure*

A number of eleven mixes was prepared as shown in Table 1, using filler to binder ratio of 2.75:1 and water to cement ratio of 0.485 according to ASTM C109/C109-16a. The GO was used as an additive with percentages of 0.03 %, 0.05 %, 0.07 % and 0.09 % by weight of cement. The SF was used as a partial replacement of cement by weight with percentages of 5 %, 7 % and 9 % to enhance the dispersion of the GO, without using any surfactant or superplasticizer. For each mix 3 samples were tested on the ages of 7 days, 28 days and 90 days. Samples were tested for flexural strength according to ASTM C348-14 and then tested for compression using portions of prisms broken in flexural just after flexural test according to ASTM C349-14 to determine the compressive strength.

SF was added to disperse the GO dilution to avoid agglomeration and then they were mixed together. The mixing process was done mechanically using an ELE automatic mixer with designated speeds and fixed mixing sequence. After mixing, a portion of the mixture was used for the flow table test, according to ASTM 1437-15, while the rest of the mixture was used to cast the specimens. The mixes were compacted by tamper for 15 times on two layers and then installed in a mechanical vibrator where the mould was firmly fixed. The mould was being raised and dropped under its own weight and performs 60 strokes to avoid aggregation. Finally the specimens was covered with a non-permeable flat surface until their removal from moulds 24 hours later and then cured in water until the time of the test.

### *2.2. Equipment*

The machine used for mechanical testing was a MEGA 10-1000-50 DM1-S with a maximum capacity of 500KN for compression, 10KN for bending, and a bending roller length of 100 mm. The load was automatically increase through control via digital controller DIGIMAXX® C-20 with servo valve in closed loop system with permanent comparison of nominal value and actual value.

To examine the fracture surface of the hardened cement composite a QUANTA FEG 250 Field Emission Scanning Electron Microscope (FE-SEM) was used. The Energy Dispersive X-ray Spectroscopy (EDS), attached to the microscope, was used to identify the GO and SF, and also to demonstrate the dispersion of GO nanosheets.

### *2.3. Materials*

Graphene oxide was prepared using Hummer's method in Nano tech Inc. labs. TEM imaging of the GO dispersion samples shows some zones darker than other indicating the obtained sheets are not fully monolayer but a stack of few layers as shown in Fig. 1. The cement used in this study was CEM I 42.5N, while the sand was standard with silica content not less than 90 %. The sand is passing from standard sieve number 10 (2 mm), retained on standard sieve number 200 (74 micron) and conforms to ASTM C778. The average grading of the used sand is listed in Table 2. The SF was obtained from KIMA for chemical industries with specification shown in Table 3.

**Table 1.**
Mixing proportions of test specimen.

| Mix number | Symbol | W/C | C:S | SF | GO |
|---|---|---|---|---|---|
| 1 | S0G0 | 0.485 | 1:2.75 | 0 | 0 |
| 2 | S0G3 | 0.485 | 1:2.75 | 0 | 0.03% |
| 3 | S0G5 | 0.485 | 1:2.75 | 0 | 0.05% |
| 4 | S0G7 | 0.485 | 1:2.75 | 0 | 0.07% |
| 5 | S0G9 | 0.485 | 1:2.75 | 0 | 0.09% |
| 6 | S5G0 | 0.485 | 1:2.75 | 5% | 0 |
| 7 | S5G3 | 0.485 | 1:2.75 | 5% | 0.03% |
| 8 | S7G0 | 0.485 | 1:2.75 | 7% | 0 |
| 9 | S7G3 | 0.485 | 1:2.75 | 7% | 0.03% |
| 10 | S9G0 | 0.485 | 1:2.75 | 9% | 0 |
| 11 | S9G3 | 0.485 | 1:2.75 | 9% | 0.03% |

**Table 2.**
Sieve analysis of sand.

| Sieve number (mm) | 2.00 | 1.19 | 0.60 | 0.30 | 0.15 |
|---|---|---|---|---|---|
| Average passing (%) | 100 | 99 | 97 | 29 | 2 |

**Table 3.**
Chemical analysis of SF as acquired from KIMA ferrosilicon plant Aswan.

| *Chemical compound* | *Chemical abbreviation* | *Percentage* |
|---|---|---|
| Silicon | $SiO_2$ | 93.4% |
| Iron dioxide | $Fe_2O3$ | 1.28% |
| Aluminum oxide | $Al_2O_3$ | 0.8% |
| Magnesium oxide | MgO | 0.75% |
| Calcium Oxide | CaO | 0.32% |
| *Chemical properties* | | |
| Alkalinity | | 1.10% |
| Chloride | CL | 0.0025% |
| Carbon | C | 1.62% |
| Sulphate | $So_4$ | 0.003% |

**Table 4. (Cont.)**

| *Physical properties* | | |
|---|---|---|
| Moisture | | 0.14% |
| Loss on ignition at 1000°C | | 1.40% |
| Bulk density | | 325 $Kg/m^3$ |
| Color | | Pale Gray |
| *Screen analysis* | | |
| Over 50Micron | | *1% Max* |

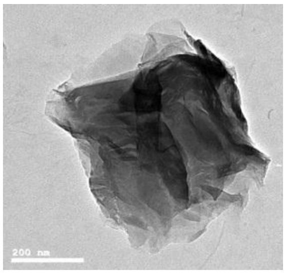


**Fig. 1.** TEM analysis of GO platelets acquired from Nanotech Inc.

## 3. Results and discussion

### *3.1. Workability*

The average diameters of the slumped cone in the flow table test were recorded as shown in Fig. 2. A gradual decrease in the cone diameter of the slumped mortar can be noticed as the GO content increases, which implies a reduction in the workability of the cement mortar. The highest reduction in the slumped cone diameter was 8 % for specimen S0G9. Such reduction in workability is generally attributed to the large specific surface area of the GO nano sheets which consumes the free water in the mixture. Comparable results have been reported before in the literature [11, 18, 21].

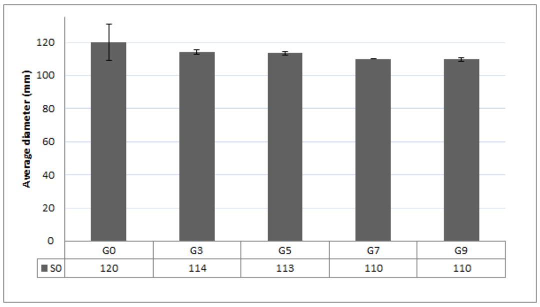


**Fig. 2.** Effects of adding GO on the flow table test results of cement mortar.

### *3.2. Mechanical strength of cement mortar incorporating GO*

After cured samples were extracted from water, they had been tested at surface of dry condition for flexural at constant loading rate of 0.05KN/S until fracture. The broken samples were then tested for compression according to ASTM C349-14 at a constant loading rate of 2.4 KN/S. The corresponding strength values for the compression and flexural tests were finally calculated and depicted as shown in Figs. (3 to 8).

Figure 3 shows the compression strength of the GO-cement composite using variable GO percentages at different ages. The results show that incorporating a very small percentage of

GO gives a significant increase in early compressive strength. For example, using 0.03% GO by weight of cement (specimen S0G3) increased the compressive strength by 29% at 7 days. Although, increasing the percentage of the GO to 0.05 % and 0.07 % as in specimens S0G5 and S0G7, respectively, the enhancing effect of the GO was minimized due to the aggregation. This aggregation might occur in the absence of a surfactant agent, resulting in larger pore sizes and hence reduced strength. Contrarily, the enhancement in sample S0G9 was notable, with an increase of 23%. This implies that the GO in this mix might be monolayer. On the other hand, the addition of GO had a minimal enhancement to the flexural strength; an improvement of 6% was obtained when a percentage of 0.09% GO was incorporated, as shown in Fig. 4.

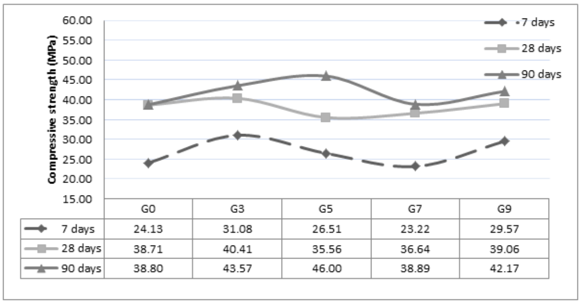


**Fig. 3.** Effect of GO content on compressive strength at different ages

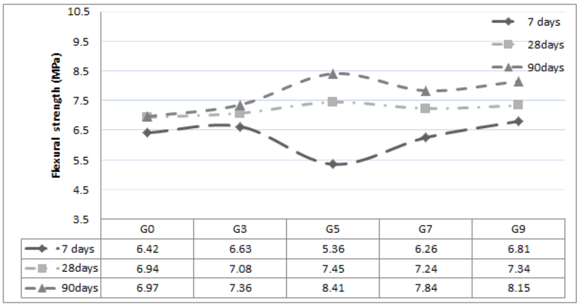


**Fig. 4.** Effect of GO content on flexural strength at different ages

After 28 days, the incorporation of 0.03% GO reflected a slight improvement of 5% in the compressive strength as in specimen S0G3. As the GO content was further increased, without using dispersant agent, to 0.05% (specimens S0G5) the strength enhancement was negatively affected. Regarding the flexural strength, the best improvement was about 7% in specimen S0G5. This improvement has not been achieved in the other mixes as the percentage of the GO was increased. However, the mix S0G9 showed the same enhancing tendency shown in 7 days, indicating that the probability of GO to be monolayers is high. Such discrepancy in the strength results has been reported before in the literature [20, 22]. That is attributed to the aggregation of the GO caused by the high alkalinity and the presence of the $Ca^{++}$ ions in the cement paste which tend to interact with the GO functional groups causing its aggregation.

At the age of 90 days, both compression and flexural strength continued to develop with the hydration age and in the same context, and the improvement became more obvious. For the compressive strength, noticeable improvements of 13% and 19% in specimens S0G3 and S0G5 respectively, was achieved. This improvement is related to the continuous stimulation effect of the GO, although it didn't occur in the fallowing mixes as the GO percentage increased due to the absence of a proper dispersion agent. Regarding the flexural strength the highest improvement was in specimen S0G5 with 21%, while the enhancement in specimen S0G7 was 17%.

It is worth mentioning that the expected enhancement results are directly related to the quality of the GO used. That is the activity of GO to stimulate the hydration process increases as the number of stack layers of the GO decreases. The GO used in this study was not actually monolayer, but in fact a stack of few layers, as shown before in Fig.1.

### *3.3. Mechanical strength of cement mortar incorporating GO with SF*

The upcoming results is a comparison of two sets of samples where the GO percentage was kept constant, while the percentage of SF as a surfactant agent was changing to study its effect on the mechanical strength of the GO-cement composite at different ages.

Figure 5 illustrates the compressive strength of the test specimens at 7 days. It shows that the highest compressive strength was obtained by adding 0.03% GO with 5% SF (specimen S5G3), where the compressive strength increased by 43%. Also, for specimen S7G3 the compressive strength enhancement was 38%. That is the stimulation in the hydration process resulted in more dense and interlocked hydration products. However, in specimens incorporating SF only, the best enhancement was in specimen S7G0 by 34%.

With further increasing the SF content, to 9%, as in specimen S9G3, the improvement descends to 34%. A level at which the effect of the SF was decreasing implying the blocking effect of the SF after reaching certain content. Thus, a certain pattern can be noticed where the strength enhancement in the GO specimens increases as the SF content increases, to a certain limit, then gradually descends to form a crest-like trend. A comparable results has been obtained in a recent study [20], where incorporating a 0.02% of GO and 2% of SF at water/cement ratio of 0.4 was able to enhance the compressive strength of the cement past by nearly 13% at 7 days. However, it was found that increasing SF content negatively affected the strength of the GO-cement composite, giving lower strength values than the counterpart specimens which incorporated SF only. This indicates that the GO could enhance the strength of mortar if it is incorporated with a proper content of SF at a certain water/cement ratio in the mixture.

The peak strength in the crest-like trend refers to a denser microstructure. That is the SF helped in dispersing effectively the GO and therefore decreasing the amount of the relatively large harmful pores and the total porosity which is positively reflected on the mechanical properties. However, any further increase of SF content decreases strength enhancement, as the excess amount of SF may shield the GO from the cement hydration process making the GO acts only as filler [19].

The flexural strength results at 7 days also emphasize that. As illustrated in Fig. 6, the enhancement achieved in specimen S5G0, containing 5% SF by weight of cement, could be accomplished by only using 0.03% of GO as an additive as in specimen S0G3. The enhancing effect of incorporating both SF and GO could be also seen in specimen S7G3 compared to S7G0 which only contains SF. Nevertheless, these improvements were rather small due to the low binder to filler ratio used in this study. Also noticeable, the flexural strength results nearly followed the same behavior of the compressive strength results, where a certain peak is clear, after which the strength tends to decrease.

At 28 days, the best improvement in the compressive strength was of 28% in specimen S5G3 followed by an improvement of 23% in specimen S7G3. The least enhancement by 22 % was in specimen S9G3, as shown in Fig. 7. These results to great extent follow the same trend, with a peak, for the 7-day results, but with lower enhancement ratios compared to the benchmark specimen. This implies the stimulation effect of the GO for the hydration process at early ages [23]. It should be mentioned that the highest improvement in the counterpart group incorporating SF only was just 17% when 5 % SF replacement was used in specimen S5G0, while using GO alone had a limited improvement on the compression strength at 28 days. This might be attributed to the aggregation of GO when used in the absence of a dispersing agent like SF. These aggregations causes weak zones and stimulate cracks [20].

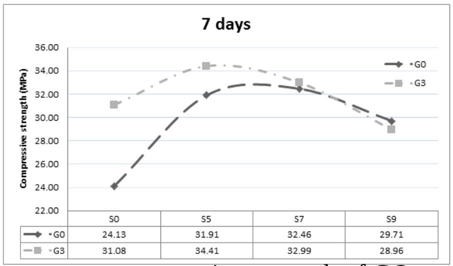


**Fig. 5.** Effect of SF content on compressive strength of GO-cement composite at 7 days

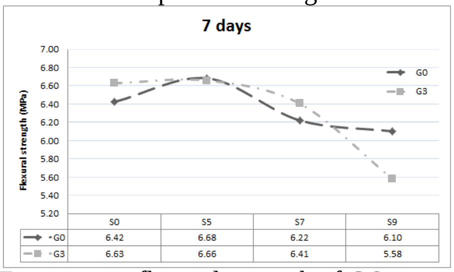


**Fig. 6.** Effect of SF content on flexural strength of GO-cement composite at 7 days

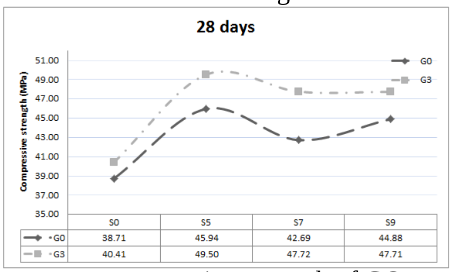


**Fig. 7.** Effect of SF content on compressive strength of GO-cement composite at 28 days

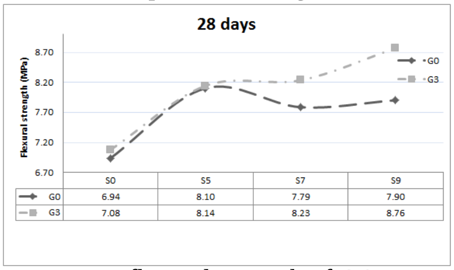


**Fig. 8.** Effect of SF content on flexural strength of GO-cement composite at 28 days

Figure 8 shows flexural strength results after 28 days. The best improvement in flexural strength was 26% for specimen S9G3. This is due to the dispersing effect of the SF allowing GO to interact with cement hydrates. The enhancements in the other specimens were 19 % for specimen S7G3 followed by 17 % for specimen S5G3. However, the best improvement in flexural strength using SF only was 17% for specimen S5G0.

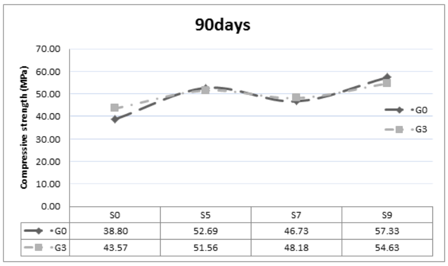


**Fig. 9.** Effect of SF content on compressive strength of GO-cement composite at 90 days

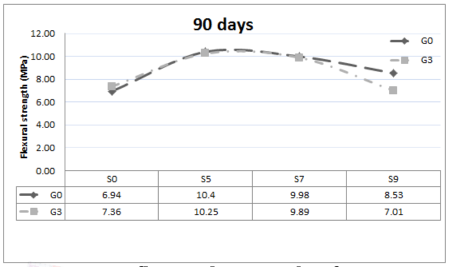


**Fig. 10.** Effect of SF content on flexural strength of GO-cement composite at 90 days

Figures 9 and 10 illustrate the compressive and flexural strength values after 90days. The figures show that using a percentage of 0.03 % GOES by weight of cement along with different percentages of SF has a limited effect on the strength improvement on the long term. This implies that GO at this percentage when incorporated with SF has a greater improving effect on the early strength than on the long term. However, using GO alone with the same percentage (0.03 % GO) could improve the compression strength by 13% and flexural strength by 6%, as shown in Fig. 9 and Fig. 10, respectively.

### *3.4. Microstructure of the GO/SF cement composite*

Figure 11 shows the SEM image of the hydration crystals formations in the plain mortar at 28 days. The figure clearly shows a high percentage of needle-like morphology (Ettringite) which is poorly dense and distributed throughout the matrix. This structure is mainly responsible for the brittleness of the cement matrix.

The SEM images of specimen S5G3 are shown in Figs. 12 (a, b). The images show a denser microstructure where some dense plate-like CH hydrates appeared, and a cloud-like CSH gel was formed. Also noticeable, is the wrinkled semi-transparent morphology of some small edges of GO covered with CSH gel and bridging a micro crack. The needle-like ettringite still appears but in much smaller amount showing that the hydration process was stimulated by the addition of GO and SF. That is the free lime content (CH) reacts with the added SF forming more CSH gel, due to the incorporation of GO which promoted the hydration reaction and hence led to a dense microstructure [15]. Thus, these images are consistent with the enhancement effect noticed in mechanical properties in the previous section.

Recent study has also presented the scanning of GO-cement composites with a larger GO content at the age of 10 min. [24]. The imaging showed that the GO wrinkled sheets covered the cement particles and that the presence of the GO stimulated the cement particles to begin hydration as whole in the same time, and increased the volume of the produced CH which promotes the overall rate of hydration. On the other hand, the imaging of the benchmark samples showed that the hydration of the cement particles was much less consecutive and smaller in size than those with the GO addition.

Figure (12.a) shows a trace of flowerlike crystals (marked in circles in the Figure) similar to those reported in previous studies [12, 21]. That is the incorporation of GO nano sheets offers growth points on its surface for the hydration process [25]. It is also noticeable that there was not any aggregations for the GO in the images of the selected samples, indicating that the GO was well dispersed in these samples this is meanly due to the presence of the SF [10]. Furthermore it is reasonable to say that the addition of the SF to the GO improves its dispersion in the cement matrix no matter how small the GO percentage is [19].

In conclusion, GO reduces the dormant period of cement hydration, accelerates the formation of the C3S phase, increases the formation of the CH and the creation of intermixture multiphases, increasing the overall hydration process and therefor improving the mechanical properties [26]. It was also reported that the GO forms strong interfacial adhesion with the negative calcium divalent ions in the CSH gel and that is the reason for the enhancement in mechanical properties [16].

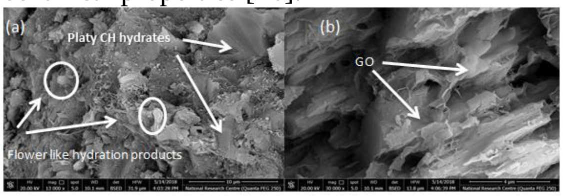


**Fig. 11.** SEM image of sample S0G0 (plain mortar) at the age of 28 days, showing the needle-like morphology of Ettringite.

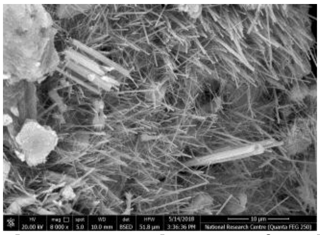

**Fig. 12**. SEM image of sample S5G3 at the age of 28days (a) the CH hydrates and the flowerlike hydration products (b) a trace of the wrinkled semi-transparent morphology of GO covered with hydration products.

## 4. Conclusion

This study investigates the effect of using GO as a nano-additive to cementitious materials. The study also investigates the incorporation of the SF as a dispersing agent for the GO nano sheets with the cement matrix. The following conclusions on the mechanical properties and the microstructure of the produced composite could be drawn:

i. Incorporating GO within cement mortar, without using dispersing agent, may reflect a limited improvement to the mechanical strength. The aggregation of the GO results in weak zones and triggers cracking. However, the incorporation of SF as a surfactant helps in dispersing the GO, resulting in an improvement in both compression and flexural strength at different ages.

ii. The use of a proper amount of SF promotes the dispersion of the GO within the cement matrix. However, using large amount has a reversing effect on the mechanical properties of the GO-cement composite due to the blocking effect between the GO and the hydration products, making the GO acting only as a nano-filler.
iii. The percentage improvement of the GO to the mechanical strength at early ages is higher than its contribution to the long term strength. So, it could be said that GO stimulates the hydration process and hence improves the mechanical properties at early ages.
iv. The SEM imaging showed that the wrinkled morphology of the GO sheets could help in bridging micro cracks and offer additional interlocking mechanism. This is reflected on the improvement of the mechanical properties.

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## تأثير أكسيد الجرافين علي الخواص الميكانيكية و التركيب المجهري للمواد الأسمنتية

### الملخص العربي:

تستخدم الخرسانة المصنعة من المواد الأسمنتية في العديد من الأنشاءات و بالرغم من المميزات العديدة للمواد الأسمنتية ألا أن مشكلة القصافة العالية لها ما زالت تحتاج الي حلول. للتغلب علي هذه المشكلة تم أستخدام العديد من الأضافات و الألياف و بعض بدائل المواد الأسمنتية و المواد النانومترية، و يعد الجرافين أحد أهم هذة المواد النانومترية. هذه الدراسة تبحث أمكانية أستخدام الجرافين كإضافة للمواد الأسمنتية مع أستخدام نوع اخر من الأضافات الشائعة مثل غبار السليكا لتحسين خواص المواد الأسمنتية. لأجراء هذه الدراسة تم تحضير أحدي عشر خلطة أسمنتية مختلفة لدراسة خواصها الميكانيكية و التركيب المجهري و مقارنة هذه النتائج بالعينات المرجعية (بدون أي أضافات)، و قد شملت العوامل المتغيرة التي تم دراسة تأثيرها نسب الجرافين و السليكا. تشير نتائج أختبارات الضغط و الأنحناء علي العينات المختبرة أنه يمكن أستخدام الجرافين كأضافة تحسن الخواص الميكانيكية و ترفع من جودة تفاعلات الأماهه للمواد الأسمنتية شريطة أن يتم توزيعها جيدا داخل عجينة الأسمنت.