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The effects of Graphene Oxide flakes on the mechanical properties of cement mortar

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Abstract. This paper discusses a study of cement mortar reinforced with Graphene Oxide (GO) flakes carried out at the University of Plymouth. Over 60 specimens were prepared and tested to obtain the tensile, compressive and flexural strengths of cement mortar with/without 0.5% GO flakes by weight of cement. The dispersion of the GO flakes and the effect of the use of polycarboxylate ether superplasticizer (0.2% by weight of cement) on the material strength are discussed. Images of the particle sizes of GO are presented from the transmission electron microscopy analysis. In addition, the images from the field emission scanning electron microscope analysis are also presented to show the difference of the microscopic structure of cement mortar with/without GO. The results of the strength tests are presented. It is shown that the inclusion of the GO flakes in general led to positive results, which suggest that GO improved the tensile, compressive and flexural strengths of cement mortar.

Keywords: Graphene Oxide flakes; mechanical properties; strength; cement mortar; microscopic structure

1. Introduction

Cementitious materials are, in general, brittle and weak in tension in comparison with their compressive strength. To improve the poor tensile strength, various reinforcements such as steel, carbon or glass fibres have been added. The fibres greatly improve strength as a whole and also delay the propagation of macroscale cracks in cement mortar. However they do not prevent brittle cement from cracking (Lv *et al.* 2013). This is because cracks in cement based materials initiate at the nanoscale where macro and micro fibres are not effective (Horsezczruk *et al.* 2015).

Recently, nanomaterials like Graphene Oxide (GO) has been added to cement based materials. GO has exceptional mechanical, thermal, optical and electrical properties, a high surface area-to-volume ratio and unique atomic structure (Horsezczruk *et al.* 2015). Research (Babak *et al.* 2014; Chuah *et al.* 2014; Du and Pang (2015); Gong *et al.* 2014; Lu *et al.* 2015; Lv *et al.* 2013; Pan *et al.* 2015; Sedhagat *et al.* 2014) showed that the exceptional intrinsic characteristics of GO improved tensile strength, reduced porosity and improved heat dissipation when incorporated in the cement matrix, while 2D Graphene nanoplatelets could be used for structural health monitoring (Le *et al.* 2014) in cement composites and improved the resistance of concrete to chloride ion and water penetration (Du *et al.* 2016). GO also reduced the scale of cracks (Babak *et al.* 2014). Table 1 summarises the existing research into cementitious materials reinforced with GO and shows comparisons of the strength and stiffness of cement mortar/paste without GO. The laboratory experiments have yielded positive results, however the inclusion of GO has highlighted problems with its dispersion into the cement matrix.

GO is easily dispersible in water thanks to the presence of oxygen groups, however they are not sufficient to properly disperse carbon nanoparticles in cement mortar. In addition, due to its large aspect ratio, GO absorbs significant amount of water, hampering the hydration of cement paste (Babak *et al*. 2014; Chuah *et al*. 2014). It has been observed that due to the strong attractive van de Waal's forces and the presence of hydrophilic groups in GO, agglomerates are formed over time (Babak *et al.* 2014; Chuah *et al*. 2014). The preliminary studies carried out by the authors also showed agglomeration of GO in the cement matrix. To enhance dispersion of GO, surfactants such as polycarboxylate superplasticizer are commonly used, often followed by ultrasonication. The superplasticizer not only reduced agglomeration, but also improved the workability of GO cement (Babak *et al.* 2014). Alternatives to superplasticizer include the use of ultrasonication (Horsezczruk *et al*. 2015), a high-speed shear mixer (Gong *et al*. 2014) and a hand-mixer (Pan *et al*. 2015).

As shown in Table 1, in most cases adding more GO results in higher strength gain. However, for a certain water-to-cement (w/c) ratio, there is always a limit, beyond which the examined parameter starts decreasing (marked with ↓). For example in the study by Babak *et al*. (2014) when w/c ratio of 0.4 was used, gradual increase of GO content has resulted in tensile strength gain until the optimal and the biggest increase of 48% was obtained using 1.5% of GO by the weight of cement. Surprisingly, when 2% GO was added to the mixture, the tensile strength decreased by

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16.3%. The researchers explained that this was caused by significant absorbance of water by GO flakes, causing the formation of clusters and hence creating zones of weaknesses. To overcome this problem, they changed w/c ratio to 0.5. As a result, 24.7% increase in tensile strength was recorded. Another strength drops (marked with ↓) were observed in the studies by Lv *et al*. (2013) and Lv *et al*. (2014b).

Research into GO reinforced cement mortar is still in its infancy, with only handful of studies carried out worldwide. Although some positive results have been obtained, as shown in Table 1, the quantities of GO within the cement matrix and the dispersion of GO remain varied. More research is required for this innovative product to be widely used in the construction industry.

This paper discusses an investigation of the effect of GO flakes on the strength of cement mortar. The dispersion of the GO flakes and the effect of the use of polycarboxylate ether superplasticizer on the material strength are also discussed. The microscopic structures of the cement mortar with/without GO are examined to observe the dispersion of the GO flakes into the cement matrix.

2. Experimental work

A total of 69 test specimens were prepared in the Materials Laboratory at the University of Plymouth. Table 2 shows the visual representation of the specimens. The test specimens were cast in the quantities of 3 parts of CEN Standard sand (BSI, 2016), 1 part of CEM1 Portland Cement and a water-cement ratio of 0.5. Single layer Graphene Oxide (GO) flakes of an average of 1.3 μm equivalent diameter, 0.5% by the weight of cement, were used to compare their effects on strengths. The GO flakes were prepared using the environmentally friendly proprietary method by BGT Materials and added to water at a rate of 1g at one time and sonicated in an ultrasonic bath for 5 minutes. Once concentrated, it was centrifuged at 1500 rpm for a period of 10 minutes before adding cement. A polycarboxylate ether superplasticizer was used at 0.2%, by the weight of cement, to facilitate the dispersion of the GO flakes and also its effect on strengths.

Transmission Electron Microscopy (TEM) analysis was carried out to observe GO flakes dispersed in water, determine particle size and quantify the visual nature of the material in its raw form. The GO flakes were dispersed in water and sonicated for 10 minutes. Subsequently, a drop of the GO solution was dropped onto a hydrophilic grid and left to air dry. The sample was then placed into a sample holder and inserted into the JEOL JEM-1400 Transmission Electron Microscope and images were recorded.

Three sets of strength testing were carried out: tensile, compressive and flexural testing. Fig. 1 shows the test specimens during and after testing. Each set had at least three representative samples with/without GO flakes. The tensile strength testing was carried out in accordance with the American Society for Testing and Materials (2012). A total of twenty-four briquettes were tested at 2, 7 or 28 days after casting.

Compressive strength testing was carried out using six 40mm cubes and six 38mm diameter, 38mm high cylinders. The cubes and cylinders were tested at 29 days after casting.

A total of twenty-one 40mm by 40mm, 160mm long beams were cast to determine their flexural strength. The beams were divided into three groups: a) control samples without GO, b) samples with 0.5% GO and c) samples with a half depth without GO and the other with 0.5% GO. The latter were cast with ordinary cement mortar up to the half depth of the moulds and then topped with GO incorporated mortar. This was to investigate the effect of GO on the flexural strength of a beam when GO were added on its tension side only. The presence of GO was visible as its bottom half depth was darker in colour, as shown in Fig. 1 (c). In addition, three beams were filled with ordinary cement mortar up to the half depth and left to cure with eight grooves spaced across the surface at regular intervals. The grooves were to stimulate roughness to improve the bonding strength between the different mortar types. The three beams were filled with GO incorporated mortar on the following day. The beams were tested at 28 or 29 days after casting. The latter three beams whose top halves were 29 days olds at testing.

Field Emission Scanning Electron Microscope (FESEM) analysis was carried out to observe the difference in the microstructures of the specimens with/without GO flakes. After the tensile testing, the briquettes were sliced approximately 20mm long, 10mm wide and 2mm thick. The slices were mounted onto a specimen holder with double sided carbon tape and the edges painted with colloidal sliver solvent to dissipate electron beam energy. They were then sputter coated with a 20nm thick layer of Chromium in the Quorom Q150T to make them more conductive. The samples were inserted into the chamber of the JEOL JEM 7001F to be depressurized and images were recorded.

3. Results and discussion

3.1 Tensile strength

The tensile test results are shown in Table 3. It was found that the average tensile strength decreased by 9% at 2 days when 0.5% GO flakes were added to cement mortar. The decrease could be due to thermal crack induced by the temperature rise during the hydration process. However the tensile strength was increased by 11 % at 7 days and up to 17% at 28 days, compared with the specimens without GO flakes.

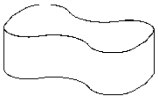
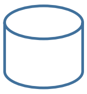
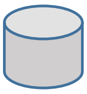
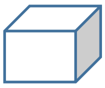
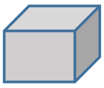
The incorporation of 0.2% polycarboxylate ether superplasticizer was necessary to facilitate the dispersion of the GO flakes in the cement matrix and its effect on the increase in strength at 28 days is as much as 8%. However, it was found that the dispersant appeared to have little influence on the cement mortar without GO flakes.

The 17% increase at 28 days after casting is less than the 27% gain reported by Babak *et al*. (2014) and significantly less than the 78% reported by Lv *et al*. (2013). The difference could be because of different exfoliating techniques used. Lv *et al*. and Babak *et al*. used the

Table 1 Summary of the existing research into cementitious materials reinforced with Graphene Oxide

Researchers	Matrix	GO (by cement weight, %)	w/c ratio	Dispersion method	Number of samples tested for each test	Specimen dimensions	Comparison with specimens without GO (%)			
							Compressive strength	Tensile st rength	Flexural strength	Young's m odulus
Babak *et al*. (2014)	mortar	0.1	0.4	Polycarboxylate superplasticizer 0.5% of cement, sonication for 40 mins	3	Briquette moulds with width and depth of 25 ± 0.5 mm at the waist line.	-	2.2	-	-
		0.3					-	12.6	-	-
		0.5					-	27.0	-	-
		1					-	38.9	-	-
		1.5					-	48.0	-	-
		2					-	-16.3 ↓	-	-
		2	0.5				-	24.7	-	-
Horszczaruk *et al*. (2015)	mortar	3	0.6	Sonication for 3 hours	N/S	N/S	-	-	-	Increase from 1–10 GPa to 5-20 GPa
Lv *et al*. (2013)	mortar	0.01	0.4	Polycarboxylate superplasticizer 0.002% of cement	5	Compression & flexural: rectangular shape - 40x40x160mm. Tensile: dumbbell shape with length of 200mm, which middle section is a rectangle with a size of 100x70x70mm and the two ends of samples are rectangle with a size of 50x70x70mm.	13.4	47.0	51.7	-
		0.02					27.6	59.5	32.9 ↓	-
		0.03					38.9	78.6	60.7	-
		0.04					42.2	36.6 ↓	30.5 ↓	-
		0.05					47.9	35.8 ↓	30.2 ↓	-
Wang *et al*. (2015)	mortar	0.05	0.37	Polycarboxylate superplasticizer 0.008% of cement	N/S	For both tests (compressive & flexural) rectangular shape - 40×40×160 mm.	24.4	-	70.5	-
Lv *et al*. (2014b)	paste	0.01	0.3	Polycarboxylate superplasticizer 0.02% of cement	3	For both tests (compressive & flexural) rectangular shape - 40×40×160 mm.	17.4	-	39.6	-
		0.02					31.2	-	54.8	-
		0.03					46.1	-	66.5	-
		0.04					55.7	-	67.1	-
		0.05					57.4	-	53.1 ↓	-
		0.06					58.5	-	42.9 ↓	-
Gong *et al*. (2014)	paste	0.03	0.5	Shear mixer at 100-200 rpm for 15 seconds	3	For both tests (compressive & tensile) cylindrical specimens - 23.5x47 mm.	46.5	53.3	-	-
Pan *et al*. (2015)	paste	0.05	0.5	Hand-mixer at 2000 rpm for 5 mins	3 compression 4 flexural	Compression test: 15x15x15mm cubes. Flexural test: 15x15x80 mm prisms.	33.0	-	59.0	6.3
Lv *et al*. (2014a)	paste	0.01	0.3	Polycarboxylate superplasticizer 0.2% of cement	5	For both tests (compressive & flexural) rectangular shape - 40×40×160 mm.	10.0	-	15.6	-
		0.02					15.2	-	21.2	-
		0.03					20.1	-	27.3	-
		0.04					25.6	-	30.8	-
		0.05					27.5	-	30.7 ↓	-
		0.06					29.5	-	30.7 ↓	-

Table 2 Visual representation of the test specimens

Specimen type	Specimen visualisation	Composition	Dimensions (mm)
Briquettes		Control sample	ASTM standards (2012) 44.5x77x25.5
		Cement mortar with 0.5% GO	
Cylinders		Control sample	Diameter: 38 Height: 38
		Cement mortar with 0.5% GO	
Cubes		Control sample	40x40x40
		Cement mortar with 0.5% GO	
Beams		Control sample	40x40x160
		Cement mortar with 0.5% GO	
		A half depth on the tension side only has 0.5% GO	

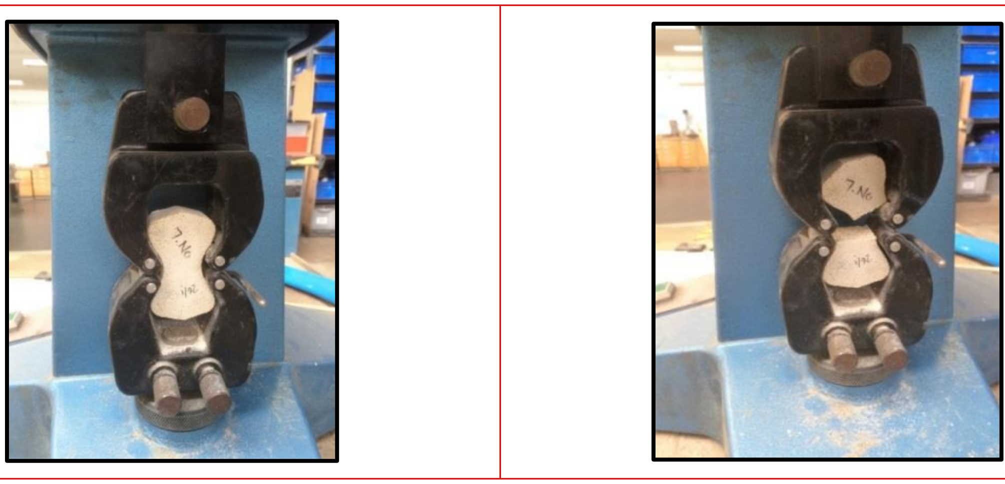
modified Hummers method to exfoliate GO flakes, while the GO flakes used in this research were exfoliated using environmentally friendly techniques and hence no harmful chemicals were used. Hence, it is possible that the intrinsic tensile strength performance of GO reinforced cement is dependent upon the exfoliation techniques utilised and the resultant variability of products produced from such methods.

3.2 Compressive strength

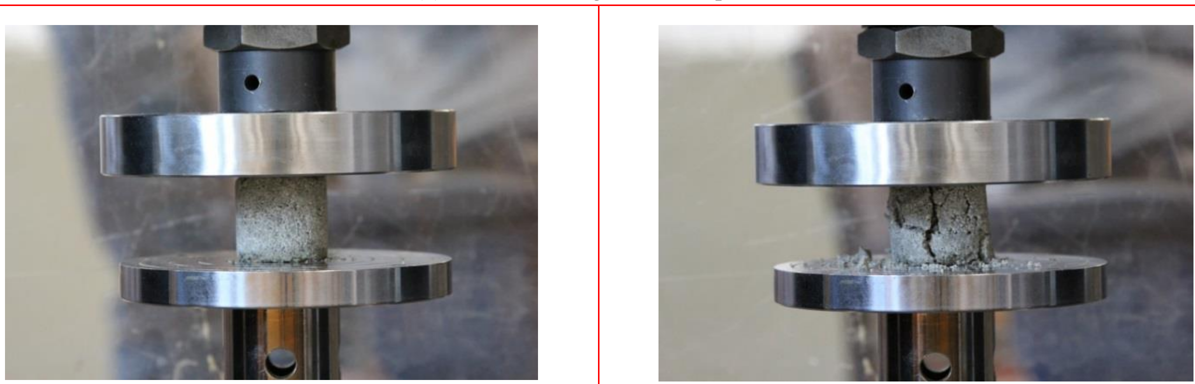
Six 40mm cubes and six 38mm diameter, 38mm high cylinders were tested at 29 days after casting to determine their compressive strength. The test results are presented in Tables 4 and 5. It was found that the average cube and cylinder strengths were increased by 10% and 29% respectively when 0.5% GO flakes were included.

3.3 Flexural strength

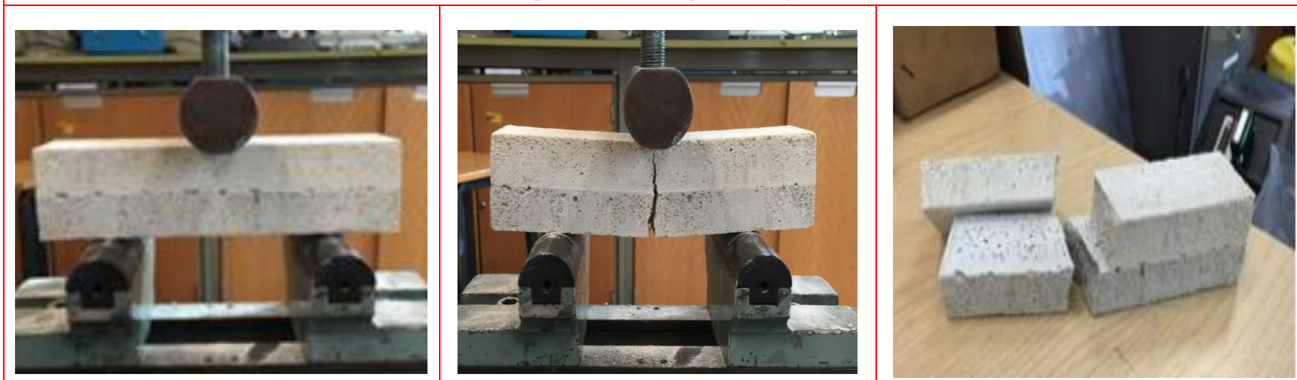
Fig. 1 (c) shows the setup for a three-point bending test and the failures of the beams. As shown in Table 6, 0.5% GO flakes enhanced the flexural strengths of the beams by 3% and 19% with and without 0.2% superplasticizer respectively. The samples with GO flakes on the tension side only gave mixed results. The beams whose two halves were cast on the same day were separated before they reached the full strength, leading to bonding failure. While, when the two halves were cast on two consecutive days with grooves between the two halves, it appears to have achieved the full bond strength and hence resulted in 12% increase in flexural strength. However, it should be noted that the bottom half was 29 days old at testing.



(a) Tensile testing of the briquettes



(b) Compressive testing of the cylinders



(c) Flexural testing of the beams

Fig. 1 Test specimens during and after testing

The incorporation of 0.2% superplasticizer resulted in 6% and 23% increase in the flexural strength of the specimens with and without GO flakes respectively. Unlike in the tensile strength testing, the superplasticizer influenced the beams with GO less than those without GO. Moreover, it should be noted that all specimens with the superplasticizer were tested at 29 days after casting.

3.4 TEM and FESEM analysis

The images presented in Fig. 2 are obtained from the TEM analysis carried out on GO flakes dissipated in water after 10 minutes ultrasonication. The images captured were of material that looked most similar and appeared more abundantly across the grid. The captures are two dimensional and some appear to be agglomerated GO flakes, where fractures within the material can be identified.

The images presented in Fig. 3 were captured from the FESEM analysis and clearly distinguish difference in the microstructure of cement mortar with/without GO flakes. As shown in Fig. 3(a), needle-like crystals were visible across the entire surface of the GO specimens. These needle-like crystals were also observed in the work by Lv *et al*. (2013). These formations are regarded to be calcium-silica-hydrate gel. In comparison with the GO specimens, crystals were more sparsely spread and rounded with no rod like crystals visible across the surface of the specimens without GO flakes examined, as shown in Fig. 3(b). This clearly indicates that the GO flakes influences the microstructure of cement mortar. The forming of these crystalline structures cross link pores is considered to be contributing to the toughness of the matrix and durability (Lv *et al*. 2013).

Table 3 Tensile strength of the 24 briquettes

Specimen Number	GO flakes (0.5%)	Superplasticizer (0.2%)	Age (Days)	Tensile strength (N/mm^2)	Average strength (N/mm^2)	Comparison
1	No	No	2	2.29	2.70	Control samples
2	No	No	2	2.77		
3	No	No	2	3.06		
4	Yes	No	2	2.46	2.47	-9%
5	Yes	No	2	2.38		
6	Yes	No	2	2.56		
7	No	No	28	3.24	3.77	Control samples
8	No	No	28	3.20		
9	No	No	28	4.67		
10	Yes	No	28	3.55	4.03	+7%
11	Yes	No	28	4.67		
12	Yes	No	28	3.87		
13	No	Yes	7	3.78	3.46	Control samples
14	No	Yes	7	3.34		
15	No	Yes	7	3.27		
16	Yes	Yes	7	3.96	3.85	+11%
17	Yes	Yes	7	3.34		
18	Yes	Yes	7	4.25		
19	No	Yes	28	3.22	3.75	Control samples
20	No	Yes	28	3.82		
21	No	Yes	28	4.20		
22	Yes	Yes	28	4.25	4.37	+17%
23	Yes	Yes	28	3.98		
24	Yes	Yes	28	4.89		

Table 4 Compressive strength of the 40mm cubes

Specimen Number	GO flakes (0.5%)	Superplasticizer (0.2%)	Age (Days)	Compressive strength (N/mm^2)	Average strength (N/mm^2)	Comparison
1	No	Yes	29	38.72	35.41	Control samples
2	No	Yes	29	37.61		
3	No	Yes	29	29.92		
4	Yes	Yes	29	39.04	38.92	+10%
5	Yes	Yes	29	37.97		
6	Yes	Yes	29	39.76		

Table 5 Compressive strength of the 38mm diameter, 38mm high cylinders

Specimen Number	GO flakes (0.5%)	Superplasticizer (0.2%)	Age (Days)	Compressive strength (N/mm^2)	Average strength (N/mm^2)	Comparison
1	No	Yes	29	31.27	28.09	Control samples
2	No	Yes	29	26.88		
3	No	Yes	29	26.13		
4	Yes	Yes	29	42.30	36.25	+29%
5	Yes	Yes	29	37.48		
6	Yes	Yes	29	28.97		

Table 6 Flexural strength of the 40mm by 40 mm, 160mm long beams

Specimen Number	GO flakes (0.5%)	Superplasticizer (0.2%)	Age (Days)	Flexural strength (N/mm^2)	Average strength (N/mm^2)	Comparison
1	No	No	28	7.08		
2	No	No	28	6.63	6.39	Control samples
3	No	No	28	5.48		
4	Yes	No	28	7.59		
5	Yes	No	28	7.61	7.61	+19%
6	Yes	No	28	7.62		
7		No	28	5.84		
8	Bottom half depth only	No	28	5.74	5.92	-7% Bond failure
9		No	28	6.18		
10		No	29/28*	6.81		
11		No	29/28*	7.73	7.13	+12%
12		No	29/28*	6.83		
13	No	Yes	29	7.41		
14	No	Yes	29	7.88	7.85	Control samples
15	No	Yes	29	8.27		
16	Yes	Yes	29	8.57		
17	Yes	Yes	29	7.83	8.09	+3%
18	Yes	Yes	29	7.87		
19		Yes	29	7.44		
20	Bottom half depth only	Yes	29	7.73	7.81	-0.5%
21		Yes	29	8.25		Bond failure

*The top and bottom halves are 29 and 28 days old respectively on the day of testing and grooves were used between the two halves

5. Conclusions

Experimental work was carried out, involving a total of 69 test specimens of cement mortar with or without 0.5% GO flakes at the Materials Laboratory at the University of Plymouth. The inclusion of the GO flakes in general led to positive results. Compared with the test samples without GO flakes,

- the tensile strengths of the briquettes were improved by 11% at 7 days and up to 17% at 28 days after casting. However, the strength at 2 days decreased by 9%. The decrease could be due to thermal cracks induced by the temperature rise during the hydration process. More research is needed to clarify this.
- The average strengths of the 40mm cubes and 38mm diameter, 38mm high cylinders were increased by 10% and 29% respectively.
- The flexural strengths of the 40mm by 40mm, 160mm long beams were enhanced by up to 19%. However, beams with GO flakes on the tension side only gave mixed results. It was found that the bond between the two halves of the beams was important to avoid a premature bonding failure.

The incorporation of 0.2% superplasticizer was necessary to facilitate the dispersion of the GO flakes and enhanced the tensile and flexural strengths of the specimens. The images from the FESEM analyses clearly indicated that the GO flakes influenced the microstructure of cement mortar.

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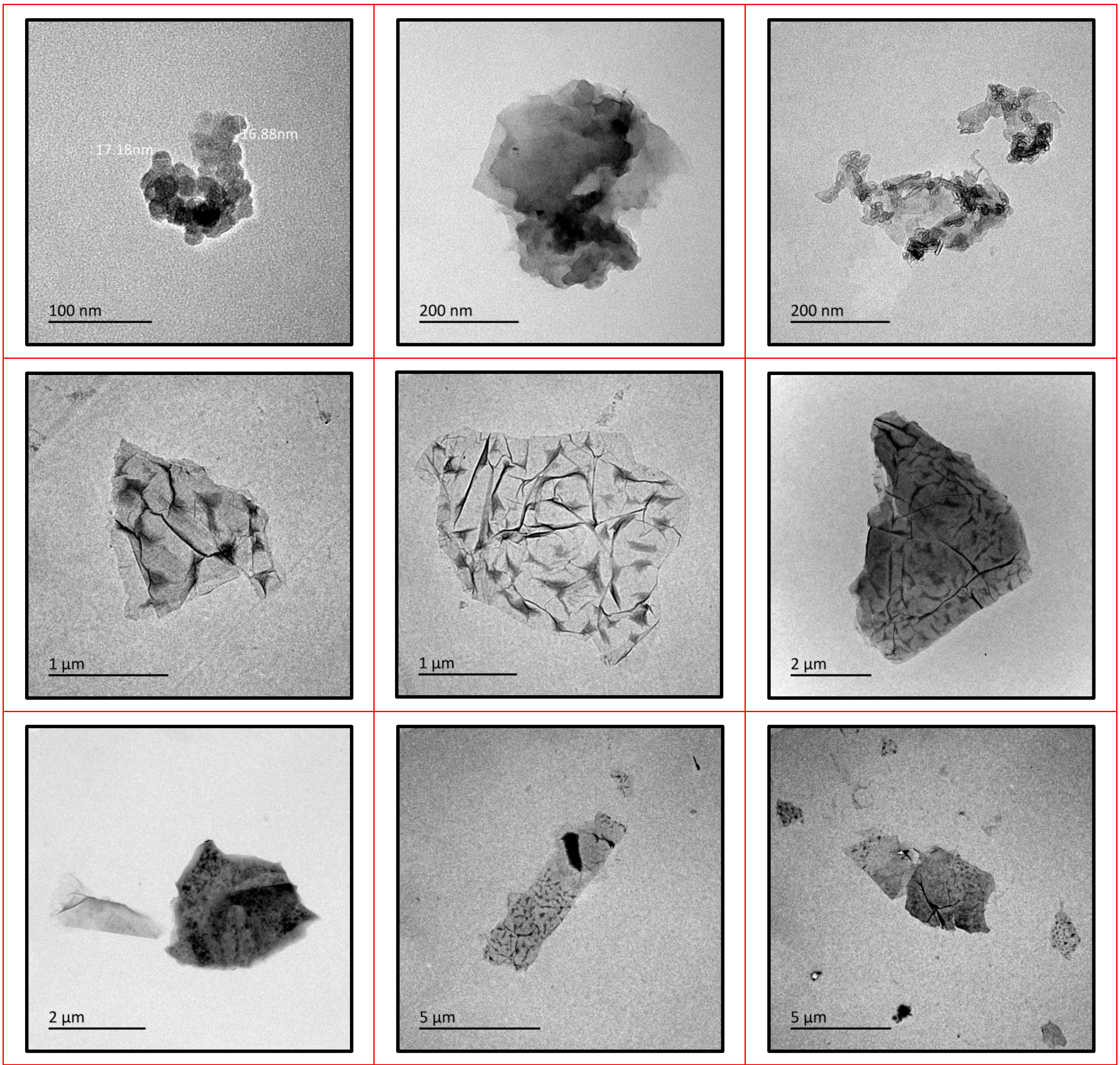
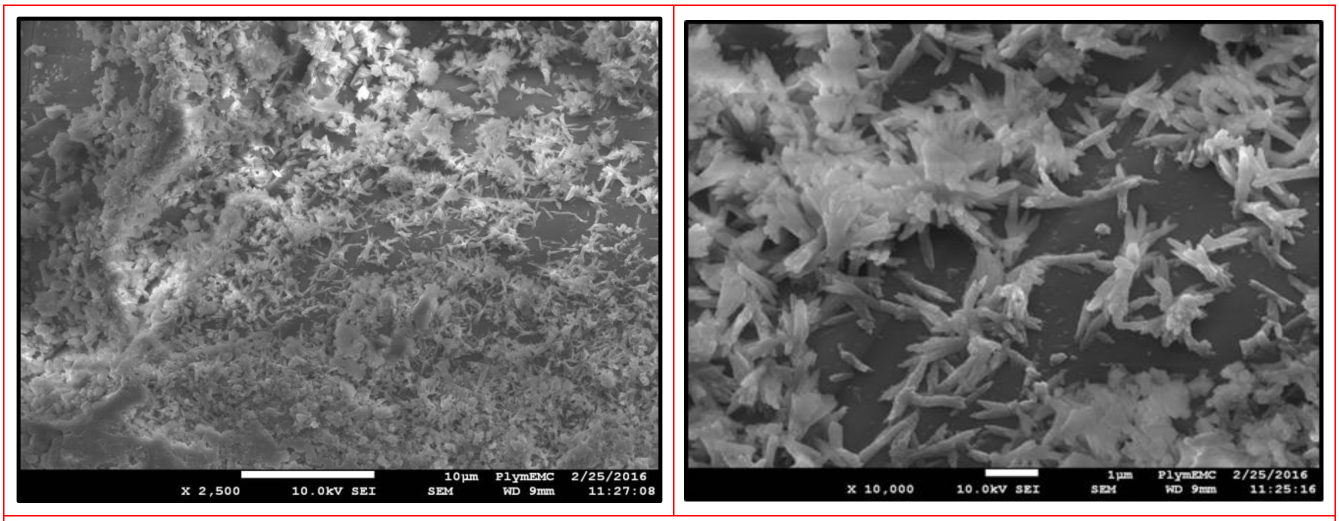
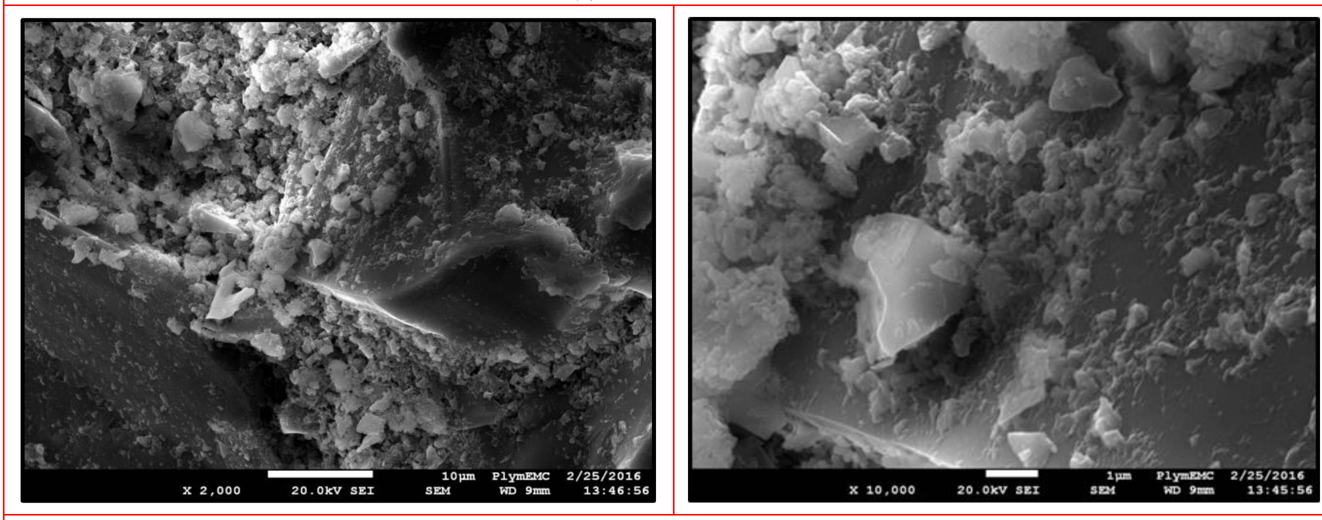


Fig. 2 Images from the TEM analysis of GO flakes dispersed in water



(a) with GO flakes



(b) without GO flakes

Fig. 3 Microstructure of the cement mortar with or without GO flakes

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