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EFFECT OF GRAPHENE OXIDE NANOSHEETS FROM GRAPHITE, PETROLEUM COKE ON CEMENT PROPERTIES

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ABSTRACT: The thickening time and strength of cement are important factors in the cementing process in the oil and gas industry. When the cement slurry hardens rapidly, it thickens before reaching the target. In addition, strong cement withstands the pressure of casing and formation. Additives are commonly used to enhance the cement's thickening time and strength. GONs have attracted attention as promising additives for cement. GONs were synthesized from graphite and petroleum coke using the liquid mechanical exfoliation method. FTIR spectroscopy revealed that the synthesized samples contain C–H, C=O, and O–H functional groups, and UV-Vis spectrometry showed absorbance at wavelengths of 233 and 261 nm. They were added to the cement at different concentrations (0.01%, 0.02%, 0.03%, 0.04%, 0.05%, 0.06%) to investigate their effect on the cement thickening time and strength. The thickening time of the samples increased with GONs concentration; the samples containing 0.06% GONs exhibited a maximum thickening time of 155.52 min. In addition, the compressive strength and shear bond strength of the samples increased linearly with GONs concentration, reached a maximum at a GONs concentration of 0.05% (32.78 and 6.21 MPa, respectively), and decreased with further addition of GONs. SEM revealed 1.5-3.8 μm cracks in the cement without GONs, where the sample with 0.05% GONs exhibited very small cracks approximately 0.4-1.0 μm. X-ray diffraction analysis showed that the cement with GONs contained plazolite, resulting in a high-strength, dense, and strong cement matrix. These results demonstrate that GONs effectively improve cement thickening time and strength.

Keywords: Graphene Oxide, Graphite Petroleum Coke, Strength, Thickening Time

1. INTRODUCTION

Cementing is an important factor in drilling operations. In the petroleum industry, cement powder and water are mixed into a slurry and then pumped into the wellbore through casing pipes into the annulus along the drilling holes. This process, which is termed cementing, is performed to ensure zone isolation, hold casing, and prevent loss of circulation. When cementing is incomplete, it cannot be pushed completely (easily hardened) because it dries before reaching the annulus. When the cement slurry dries early, pre-hardening shrinkage occurs, i.e, the material shrinks before hardening, which can affect its application and cause problems, such as too early hardening [1]. Early hardening of the cement poses a challenge in cementing very deep wells, which requires a long time for the slurry to reach the annulus [2].

The thickening time is the duration required for the cement slurry to reach 70 unit consistency (UC). This value is the maximum amount of cement slurry that can be pumped according to API standards [3]. The thickening time can be increased by adding retarders, such as lignosulfonate and carboxymethyl hydroxyethyl cellulose (CMHEC), which slow the drying of cement [4]. Calcium lignosulfonate contains hydroxyl, carboxyl, carbonyl, and other active groups that act as adsorbents [5]. CMHEC contains anionics and hydrocolloids that enhance cement adsorption [6].

The hardened cement can become brittle due to low tensile stress [7]. The presence of pores in the drilling cement results in its low strength [8]. The strength of the cement decreases over time, resulting in cracking. Cracks usually occur because of the pores between cement molecules, which collapse when a load is applied. Therefore, squeeze cementing is required, which is expensive. Cracks can be prevented by increasing the strength of cement by adding additives, which can reduce the number of pores and defects in the cement base material [9,10]. Silica flour (SF) is a common cement additive used for strength improvement [11,12]. Two types of strength are usually considered for cement: compressive strength (CS) and shear bond strength (SBS). According to the American Petroleum Institute (API), the minimum CS required for continuous drilling operations is 1000 psi, and the shear strength should not be less than 100 psi for the casing to bond firmly. An SBS of less than 100 psi can cause the formation of a micro-annulus between casing and cement or between cement and formation, resulting in poor zone isolation, causing production decline. Therefore, it is necessary to optimize cement slurry design before cementing [13]. The CS of cement up to API standards is sufficient to provide structural support to the casing, prevent formation movement, and protect the well from collapse. Cement with low

CS cannot withstand the mechanical loads casing, formation, and internal/external pressure. Cement casings with low CS are not able to withstand pressure changes during operations, such as secondary cementing or production. Therefore, the cement may collapse when the operating pressures increase, and it be comes difficult to remediate the well in case of failure [14,15].

Recently, nanomaterials have attracted significant interest as drilling cement additives. Graphene oxide nanosheets (GONs) are highly promising retarders for cement and can increase the strength of drilling cement. GONs are two-dimensional (2D) nanomaterials containing sp2 and sp3 hybrid carbon atoms, which act as insulators. The GONs layer has a thickness of approximately 1.1 nm ± 0.2 nm [16]. GONs are hydrophilic and easily disperse in water; thus, they can be integrated into water-based cement slurries. The oxygen and -OH functional groups present on GONs surfaces promote their interactions with cement and improve the interfacial bonding with the cement base material.

Graphene oxide has been widely synthesized from natural graphite found in metamorphic rocks, igneous rocks, and coal seams. Ahmed added synthetic graphite to H-grade cement to improve its thickening time and CS. Wang and Shuang examined the effect of graphene nanoplatelets (GNPs) on the properties, pore structure, and microstructure of cement and reported an optimum CS of 4.3% for the sample with 0.05 wt% GNPs [17]. Chintalapudi and pannem, investigated the effect of graphene oxide on the strength of cement composites, and an optimum CS of 46% was obtained at a dose of 0.03% [18]. Tabatabaei investigated the effect of graphene oxide on cement durability. The sample with 0.2 vol% graphene exhibited a thickening time value of 1 h 55 min and an optimum SBS of 75 kN/mm [19]. Cody varied the concentration of GNPs in H-Class cement suspensions and reported that GNPs modified the microstructure of hydrated cement suspensions dried at 90°C and 95% relative humidity (RH) by strengthening the pore space. Thus, the mechanical properties, including Young's modulus and axial peak stress, increased significantly with increasing GNPs contents[20].

In the oil and gas industry, petroleum coke, also known as green coke, is produced by delayed coker units in petroleum processing complexes. This product is associated with various environmental issues, which render its value relatively low. The Dumai oil refinery in Indonesia produces petroleum coke with a production capacity of 330,000 tons per year [21]. The value of this product can be increased by using it for the synthesis of carbon products, such as graphite, petroleum coke, and GONs, which have high technological value.

In this study, we employed GONs derived from graphite petroleum coke as additives to increase the cement thickening time and strength.

The synthesized GONs were characterized by Fourier transform infrared (FTIR) and ultraviolet-visible (UV-Vis) spectroscopy. Furthermore, we explored the effects of the GONs on thickening time, CS, and SBS of drilling cement. The microstructures and pore sizes of the cement samples were analyzed by scanning electron microscopy (SEM). Finally, the minerals in the drilling cement with and without GONs were determined.

2. RESEARCH SIGNIFICANCE

This study focuses on the synthesis of GONs from graphite petroleum coke using the liquid mechanical exfoliation (LME) method and its effects on the thickening time and strength of drilling cement. Another nanomaterial-based drilling cement additive is nanosilica (SiO_2), but it is not as good as GONs in improving strength and thickening time. GONs have a very strong and stable two-dimensional structure, which can improve the strength of drilling cement by increasing the bond between cement particles.

3. MATERIALS AND METHODS

3.1 Materials and Equipment

The first material used is graphite petroleum coke as an additive mixture for making cement samples. Second, class 'G' cement has an API 10 standard with a high sulfate resistant (HSR) type as the basic cement in well cementing with a depth of up to 8000 ft with a temperature of up to 900℃. Third, distilled water is used as a mixture in cement suspension. The last material is grease which functions to coat the cubic mold so that the cement is easily released from the mold.

The equipment used in making samples are digital scales, constant speed mixers to mix cement, water and additives, atmospheric consistometer used to circulate slurry with bottom hole circulate temperature (BHCT) temperature, measuring cups, stopwatches, hydraulic press used to measure the cracking pressure strength, water bath used to control the temperature to remain constant in testing the hardness of the sample, cubic molds in testing cement samples for CS, cylindrical molds in testing cement samples for SBS, and vernier calipers.

3.2 Synthesis and Characterization of GONs

Graphite petroleum coke was used as the base material for the synthesis of GONs using the LME method [22]. This method combines liquid exfoliation (LE) and mechanical exfoliation (ME). It is easy and cheap, and it does not require explosive chemicals. This method is cheap because it only uses a mixer and sonication equipment, compared to the chemical vapor deposition (CVD) is a graphene synthesis method using SiO_2 substrate that requires expensive and high-tech equipment [23]. To synthesize GONs, 1 g of graphite and a linear alkylbenzene sulfonate (LAS) surfactant solution were mixed. The surfactant was used to weaken the van der Walls bonds and widen the distance between the graphene sheets. Next, the solution was then blended for 5 min. The blender was set at fixed speed that could separate the graphene sheets. The solution was then sonicated at a frequency of 40 kHz to improve GONs [24]. Because the synthesis was performed in the liquid phase, the resulting GONs contained a large amount of oxygen. Furthermore, the solution was centrifuged and dried at 100˚C to produce dry GONs. The GONs samples were characterized using FTIR (Nicolet IS10 Spectrometer) in the range of 4650-450 cm^{-1} to identify the functional groups present in the samples. UV-Vis Spectroscopy (SHIMADZU UV-1800) was performed to measure the excitation energy of the samples the absorbance of radiation in the UV-Vis electromagnetic wave region.

3.3 Cement Supply and Testing

Class G cement was used to prepare drilling cement according to the API 10A classification. To prepare cement slurry, distilled water was added to a mixer cup and mixed at 400 rpm for 15 s. The cement powder and GONs were then added to mixer cup and mixed at 1200 rpm for 35 s. The concentration of GONs was varied, as listed in Table 1.

Table 1. Composition of the cement slurry

Sample	Mass (gr)		
	Cement Powder	Water	GONs
0%	791.237	348.223	0
0.01%	790.574	348.406	0.079
0.02%	789.789	348.613	0.158
0.03%	789.073	348.770	0.236
0.04%	788.345	349.000	0.315
0.05%	787.629	349.156	0.394
0.06%	786.957	349.330	0.472

The thickening time of the samples was measured using an atmospheric consistometer. The cement slurry was first poured into container to the height limit on the boundary line and then covered with a greased lid. The container was placed in the atmospheric consistometer at a temperature of 140 °F. The thickening time was recorded when the consistometer showed a scale of 70 units of consistency (UC).

The CS and SBS of the samples were determined using a hydraulic press. CS tests were performed using cubic samples, where SBS tests were performed using cylindrical samples. The cement slurry was poured into the molds and then immersed in a water bath at 140 °F for 24 h. After hardening, the samples were loaded until they cracked, and the CS was recorded as the maximum pressure at crack. If the pressure exceeds 3000 psi, initial loading is not required. The maximum loading was achieved between 20 and 80 s. The CS and SBS were calculated with correction constant K using the following equations:

$$CS = K \times P \times (A1A2) \quad (1)$$

$$SBS = K \times P \times (A1\pi Dh) \quad (2)$$

SEM with energy-dispersive spectroscopy (SU3500) was conducted to analyze the topographies and elemental compositions of the samples, and X-ray diffraction (XRD Bruker D8 Advance) was performed to determine contained in the cement samples.

4. RESULTS AND DISCUSSIONS

4.1 Characterization of GONs

4.1.1 FTIR

FTIR spectroscopy was performed to investigate the presence of oxygen functional groups bound to the samples. Fig. 1 shows the FTIR spectra of the GONs. The analysis was based on the excitation of molecular bond vibrations through the absorption of infrared light with a wavelength range from 4650 to 450 cm^{-1}. The first peak was observed at a wavelength of 3203.90 cm^{-1}, which is attributed to O–H stretching (carboxylic group). The peak observed at 1634.72 cm^{-1} is attributed to C=O stretching. The C–H stretching group was observed at a wavelength of 680.04 cm^{-1}. These results indicate the presence of hydroxyl, carboxylate, and carbonyl groups in the samples, confirming that GONs were successfully synthesized from graphite petroleum coke. In addition, GONs formed a chemical structure of conjugated aromatic rings [25].

4.1.2 UV-Vis Spectroscopy

Fig. 2 shows the UV-Vis spectra of the GONs. The absorbance of GONs causes the excitation of electrons form the ground state of the excited state.

Two peak absorbance waves are observed at 228 and 261 nm, which are attributed to the electron transition in the π–π^* orbital of the C–C and C=C bonds in the sp2 hybrid area and the n–π^* transition of the C–O bond [26]. The energy gap of the GONs was 5.33 eV. which was calculated using the following Planck equation:

$$E_g = \frac{hc}{\lambda} \tag{3}$$
$$= \frac{(6.626 \times 10^{-34}\, J.s)(3 \times 10^{8}\, m/s)}{232 \times 10^{-9}}$$
$$= \frac{1.9878 \times 10^{-25}\, J}{2.33 \times 10^{-7}}$$
$$= 8.53 \times 10^{-19}\ \mathrm{J}$$

Converting joules to electron volts:

$$1\ \mathrm{eV} = 1.602 \times 10^{-19}\, J$$

So, Eg in eV:

$$E_g = \frac{8.53 \times 10^{-19}}{1.602 \times 10^{-19}}$$

$$= 5.33\ \mathrm{eV}$$

Where, E_g is the energy gap, h the Planck's constant, c the speed of light, and λ the wavelength.

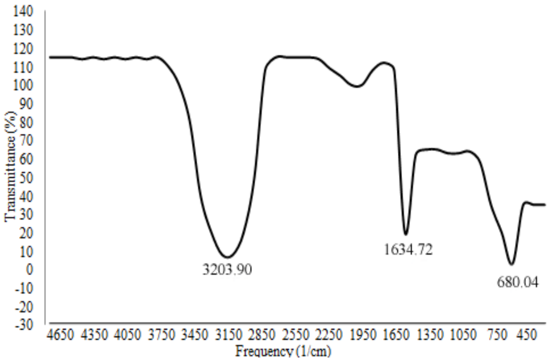

Fig 1. FTIR Transmittance of the synthesized GONs

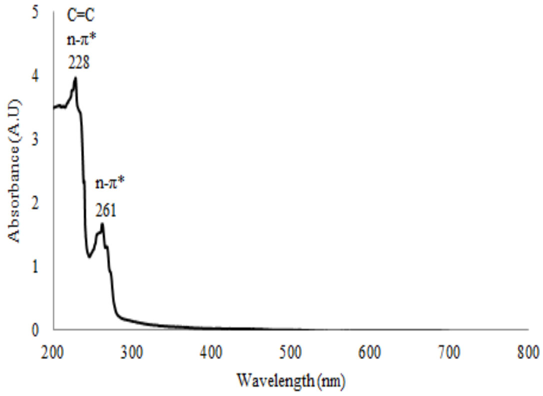

Fig 2. Ultraviolet-visible spectra of GONs

4.2 Cement Testing

4.2.1Thickening time

Table 2 lists the thickening time of cement samples with and without GONs, and Fig. 3 shows the variation in the thickening time with the GONs concentration.

Table 2. Thickening time versus GONs concentration at 70 UC

No	Additive GONs (%)	Thickening Time at 140°F (second)
1	0	6873
2	0.01	7637
3	0.02	7642
4	0.03	8081
5	0.04	8360
6	0.05	8714
7	0.06	9352

The thickening time of the cement slurry without additives was approximately 6873 s. When the cement was mixed with water, the constituents were hydrate as Fig. 4. The compounds formed are calcium silicate hydrate (C–S–H) gel, calcium hydroxide (CH), strengite, and monosulfate, which promoted the binding and strength the of cement [27].

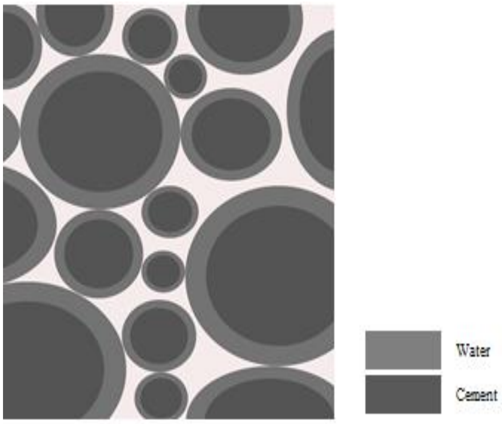

Fig 3. Cement slurry (cement + water)

The thickening time of the cement slurry increased linearly with the GONs concentration. Compared with the sample without GONs, the thickening time of the sample 0.01% GONs increased by 11.19%, and that of the sample with 0.06% GONs increased by 36.03%. This is attributed to hydrophilicity of GONs [28], owing to the presence of hydroxyl and carbonyl functional groups at the edges of the structure, as shown in Fig. 4. Because of its hydrophilicity, when mixed with cement slurry, GONs can attract water molecules and form water layers around the cement particles (Fig. 5). Such layers reduce the dehydration rate of cement and slow the bonding among particles, thus reducing the cement hardening time. These results demonstrate that GONs are good retarders for cement slurry, which can delay cement hardening time, ensuring that the cement slurry reaches the target before hardening.

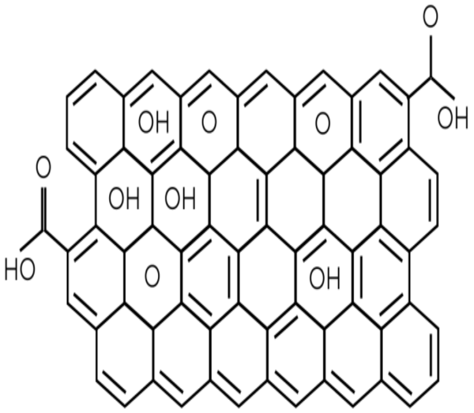

Fig 4. Chemical structure of GONs

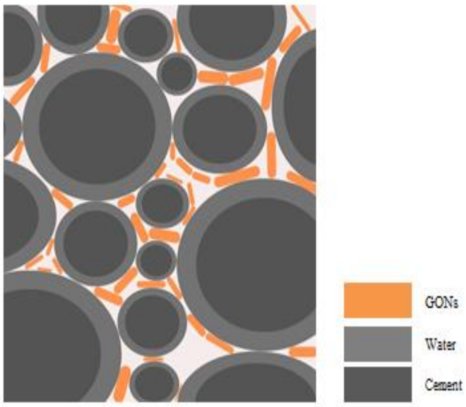

Fig 5. Cement slurry with GONs

4.2.2 Compressive Strength and Shear Bond Strength

The addition of GONs to cement slurry increased its CS and SBS, as shown in Figs. 7 and 8. The CS and SBS of the sample with 0.01% GONs increased by 3.99% and 4.69%, respectively, indicating that even at a very small concentration, GONs can significantly increase the strength of cement.

The CS and SBS of the samples continuously increased with increasing GONs concentration. The sample with 0.05% GONs exhibited the maximum CS (32.78 MPa) and SBS (6.21 MPa), which were recorded at 0.05% GONs, representing 41.40% and 39.88% increases, respectively, compared with those of the samples without GONs. This increase in strength is attributed to the fact that GONs act as fillers in the cement matrix, thereby filling the space between the cement particles and reducing the pore size, resulting in a denser and stronger cement matrix [29]. In addition, the GONs improve the pore structure of cement slurry, thereby reducing the absorbency of the cement composite and increasing its strength. Graphene oxide aggregates as fibers in the cement matrix, thereby increasing its strength, toughness, and crack resistance.

With further addition of GONs up to 0.06%, CS and SBS of the samples decreased. However, the values were still significantly higher than those of the sample without additives and were way above the API standard. This result indicates that excessive GONs in cement result in their accumulation due to intermolecular van der Waals forces, thereby reducing their dispersion in the cement slurry [30]. At very high GONs concentrations, the water demand increased significantly, affecting the fluidity of the slurry. These results demonstrate that GONs can effectively improve cement strength.

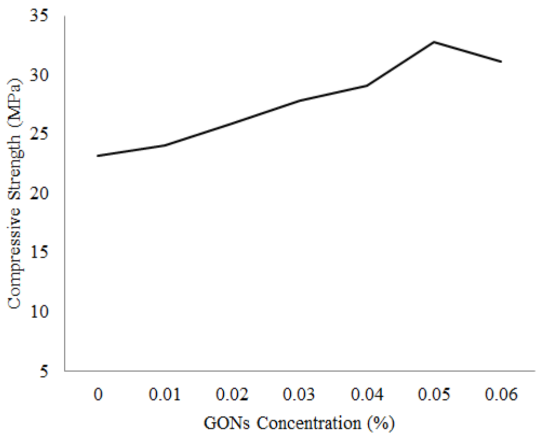

Fig 6. Variation of the CS cement samples with GONs concentration

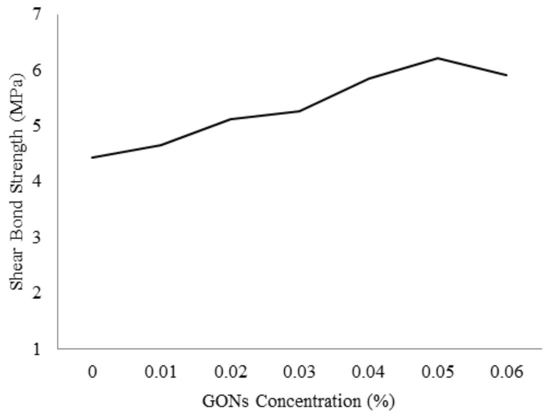

Fig 7. Variation of the SBS cement samples with GONs Concentration

4.3 Pore testing

4.3.1 SEM

We employed SEM to characterize the topography of the cement samples without GONs and with the optimum GONs concentration of 0.05%. Table 4 lists the microcracks and strength of the samples.

Table 3. Microcracks and strengths of samples with and without GONs

Sample	Microcrack (μm)	CS (MPa)	SBS (MPa)
A	1.500 – 3.833	23.17	4.44
B	0.447 – 1,005	32.76	6.21

The topography of the cement without GONs with 2000x magnification large and long gaping microcracks with gap of 1,500 – 3,833 μm (Fig. 8). These microcracks can reduce the CS, and casing stability of the slurry, and cause leakage of formation fluid. If microcracks in cement are not detected or repaired, they can cause problems in the next stage of drilling, including difficulty in isolating the productive zone or preventing contamination between the formations.

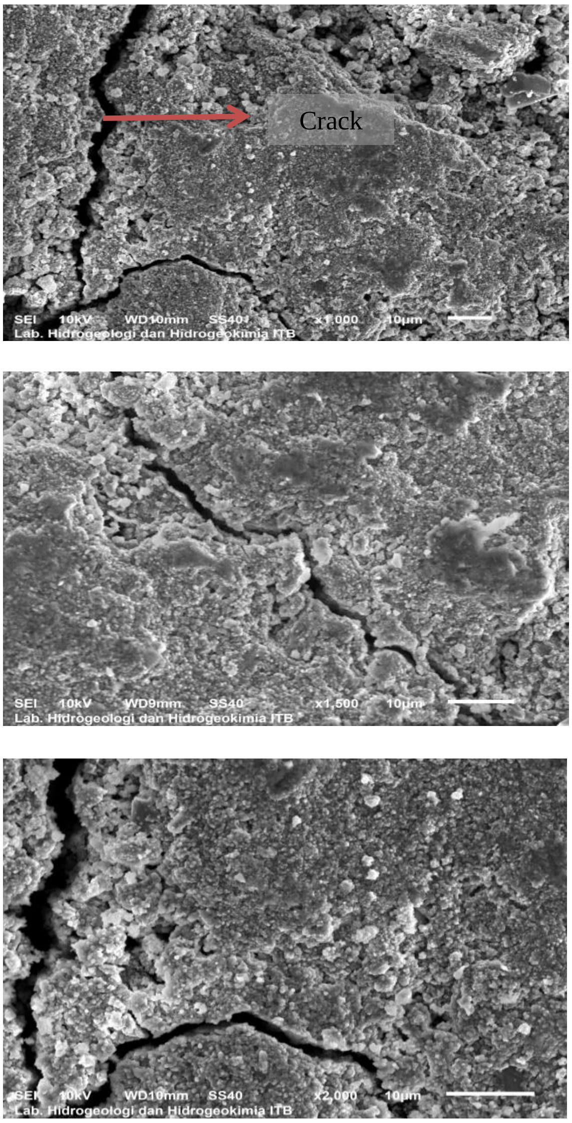

Fig. 8 Scanning electron microscopy (SEM) of cement sample without GONs at 1000×, 1500×, and 2000×

Fig. 9 shows the topography of the cement sample with 0.05% GONs, at 2000× magnification. The pore size ranged from 0.447 to 1.005 μm, which is much smaller than that of the sample without GONs. This reduction in pore size is attributed to the fact that GONs filled the space between the cement particles and reduced the formation of cracks, resulting in a denser and sturdier matrix and increased cement strength. The addition of GONs to the base cement reduces or prevent the spread of cracks and the diameter of the pores in cement samples, thus improving the mechanical properties of the cement composites. Nanosized and lightweight GONs can be widely distributed in the cementitious material matrix [31].

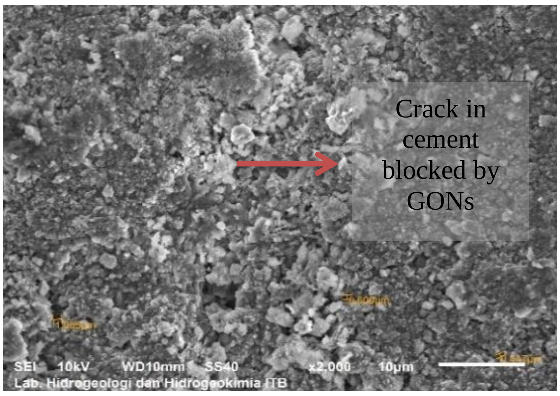

Fig 9. SEM of cement sample with 0.05% GONs

EDS mapping was used to determine the morphology of the GONs in the cement matrix by detecting the carbon and oxygen content. The EDS spectra of the sample without GONs are shown in Fig. 10, and Fig. 11 shows those of the sample with 0.05% GONs. The samples contained several element: carbon (C), oxygen (O), sodium (Na), magnesium (Mg), aluminum (Al), silicon (Si), sulfur (S), potassium (K), calcium (Ca) and iron (Fe).

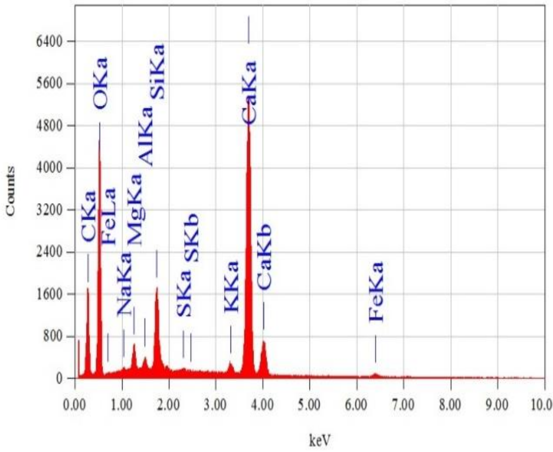

Fig 10. Energy dispersive spectra (EDS) of the cement sample without GONs

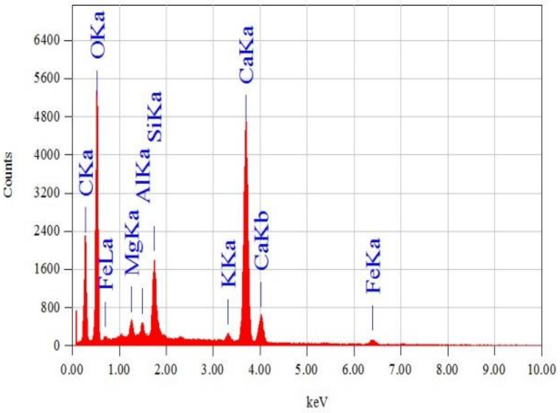

Fig 11. EDS results of the cement sample with 0.05% GONs

The concentrations of the elemental constituents of the samples were determined using the ZAF standardless quantitative analysis method (Table 4). The sample without GONs contained 16.85% carbon and 48.26% oxygen. The carbon and oxygen contents increased to 20.78% and 50.17%, respectively, with the addition of 0.05% GONs. This is because GONs molecules are composed of carbon atoms with oxygen functional groups at their edges, thereby contributing to the content of both elements in the cement composite. In addition, the hydration products contributed to the increase in the carbon and oxygen contents.

Table 4. EDS results for cement samples without GONs and with 0.05% GONs

Elements	Cement without GONs	Cement with 0.05% GONs
	Mass (%)	Mass (%)
O	48.26	50.17
Ca	27.61	22.35
C	16.85	20.78
Si	3.43	3.09
Mg	1.13	0.76
Fe	1.12	1.61
K	0.78	0.64
Al	0.47	0.59
Na	0.20	-
S	0.14	-
Total	100	100

4.3.2 XRD

XRD was employed to determine the minerals in the cement samples. As shown in Figs 13 and 14, the sample without GONs contained brown millerite ($Ca_2(Al,-Fe)_2O_5$), ferrite ($Ca_2Fe_{15.588}O_{25}$), portlandite ($Ca(OH)_2$), strengit ($Ca_6(Al(OH)_6)_2(SO_4)_3(H_2O)_{26}$), lardite ($Ca_2(SiO_4)$, stishovite ($Hg_4Sb(OH)_3O_3$), pentlandite ($Fe_{1.63}Ni_{1.82}Co_{5.6}S_8$), and calcite ($CaCO_3$). In contrast, the samples with 0.05% GONs exhibited high calcite content because the calcite particles were well bound and evenly distributed in the cement matrix. The mass of calcite in the cement increased because of the better structural support of the GONs enriched cement matrix. In addition, no peaks of stishovite and pentlandite were observed, but a new compound, plazolite ($Ca_3Al_2(SiO_4)_{1.53}$) was observed, indicating that GONs changed the crystal structure of the cement. The plazolite filled the voids and substantially changed the porous structure to a dense and compact one, thereby increasing the strength of the sample. Plazolite is chemically similar to calcium hydrosilicates, and hydrogranates produce a dense and strong cement matrix [32].

Table 5. Mineral contained in the cement without GONs and with 0.05% GONs

Minerals	Cement without GONs	Cement with 0.05% GONs
	Mass (%)	Mass (%)
Brownmillerite	37	21
Ferrite (Ca-bearing)	22	2
Portlandite	20	29
Ettringite	10	7
Larnite	6	7
Shakovite	3	-
Pentlandite	1.7	-
Calcite	0.3	16

5. CONCLUSION

GONs were successfully synthesized from graphite petroleum coke LME. FTIR revealed (C–H, C=O, and O–H) groups at wave-numbers of 680.04, 1634.72, 3203.90 cm^{-1}. The UV-Vis spectra of the sample show two absorbance peaks at 233 and 261 nm. The cement thickening time was increased by 36% with the addition of 0.06% GONs. This is attributed to the hydrophilicity of GONs, which promotes the cohesion of water molecules to form water layers around the cement particles, thereby reducing reducing the dehydration rate of the cement and slowing down the bonding of the particles and thus the thickening time of the cement. The addition of GONs also increased the cement strength. The sample containing 0.05% GONs exhibited the maximum CS and SBS of 4754.71 and 901.20 psi, which represent approximately 41% and 39% increases, respectively, compared with those of the sample without GONs. These results indicate that GONs improved the cement pore structure. GONs filled the space between the cement particles and reduced the pore size of the cement sample from 3,833 to 1,005 µm (approximately 73%), resulting in a denser and stronger cement matrix.

The increased strength can make the well structure more resistant to high pressure and extreme environments underground. Longer thickening time gives the operator additional working time to pump the cement into the well before it hardens, ensuring a more even spread of cement between the casing and the rock formation. Under high temperature and pressure (HPHT) conditions, GONs reinforced cements are more resistant to thermal and chemical degradation.

6. ACKNOWLEDGEMENTS

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