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ORIGINAL RESEARCH PAPER

GO/C_2S Nanocomposite as an environmentally- friendly Material for Sustainable Development of Cement Structure

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ABSTRACT

In the present work, Graphene Oxide (GO) nanoparticles with 0.1, 0.2, 0.4 and 0.8 weight percentages (wt.%), synthesized by Hummer method entered into the Di-Calcium Silicate (C_2S) matrix (synthesized with using Pechini method and their nanostructural and mechanical characteristics studied by XRD (X-ray diffraction), FTIR (Fourier transform infrared), GPS 132 A, 4- probe techniques and emission factor analyses.

To study and examine GO-C_2S nanocomposites as an environmentally- friendly Material for Sustainable Development of Cement Structure 4- capacitor form, with Metal (Al) /GO-C_2S composite/ Si substrate, is fabricated. The quality factor and dissipation factor were measured by GPS 132 A tool. The obtained results show that the sample with 0.4% wt. of GO nanoparticles has a 0.625 fraction emission factor and higher quality factor (32.4), measured at two frequencies of 120 KHz and 1 KHz. Therefore C_2S/ GO nanocomposite with 0.4 wt.% GONs can be therefore introduced as a good additive to reduced emission factor for production of cement and dissipation factor.

Keywords: *Di-Calcium Silicate, Graphite, Nanocomposites, Quality Factor*

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INTRODUCTION

Some issues such as oxidation of fossil fuels deforestation (and another land- use changes) and carbonate decomposition are threatening the people life and environment. On one hand, the global production of cement and cement-based materials have entered in undesirable living conditions and increased more than 30- fold since 1950 and almost 4- fold since 1970 [1-3]. The reason could be due to rapid development in some countries (China as an example) [1]. Van [2] reported that cement production in China has grown by a factor of 12 since 1990. Andrew [1] estimated that India is the second- largest producer of cement. These points indicate that most contamination and environmental problems caused by cement production in developed and developing countries. It means that thinking and finding a solution to decrease environmental problems are so important. In this regard, researchers have conducted many studies on cement structure to improve cement-based material's characteristics with noting to some aspects of cement productions: (i) The chemical reaction with some additives, such as Graphene Oxide nanoparticles (GONs) and Di-Calcium Silicate (C_2S); (ii) crystallite phases and mechanical characteristics with higher quality factor (Q_F) and lower dissipation factor (D_F).

For this purpose, Graphene oxide (GO), an oxidized form of graphene has been candidate, so that the oxygenated groups were on both sides of the plate and graphene nanoparticles and/or crystallites were placed on their plate's edges for getting hydrophilic effect and uniform distribution

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of this substance in the ceramic and concrete industry. Di-Calcium Silicate (C_2S) compound is also one of the most important parts of these composites. In Refs. [4-9] the effect of GONs nanoparticles on the calibration reaction rate of calcium silicate compounds was studied. The use of nano-additives to improve the physical, electrical and mechanical properties of dielectric composites is an essential work. Adding GO to dielectric composites leads to significant improvement in mechanical strength, electrical, nanostructural characteristics, and a decrease in growth of a crack in composite structures (see more details in our previous work, Ref. [9].).

In this regard, people feel that any type of development must be nondestructive, biocompatible and sustainable [10-15]. Therefore, cement structure needs to be improved and modified with enhancing the quality factor of the samples. In the present work, GONs, synthesized by the Pechini method, as additives were added in the C_2S matrix. The Pechini method is a convenient method for low temperature and high purity synthesis. Then, the effect of GONs on the structure of C_2S and type of its bond were studied using the FT-IR (Fourier-transform infrared spectroscopy) graph, XRD (X-ray Diffraction) analysis information. The obtained results from GPS 132 A and emission factor analysis show that adding 0.4 in wt.% of GONs to C_2S leads to a more uniform distribution of GONs and lower dissipation factor, and higher quality factor which is necessary for Sustainable Development of Cement Structure.

EXPERIMENTAL PROCEDURES AND DETAILS

As stated above, we extracted the Oxygengraph nanoparticle from graphite oxide by using the pechini method and then synthesized the nanocomposites in order to trap GO in a dicalcium silicate network. In the latter case, the Hummer method is used for the synthesis of graphene [8], in that 50 ml of concentrated sulfuric acid, 2g of graphite and 2g of sodium nitrate were mixed together at room temperature and then 140 ml of distilled water and 16 ml of oxygen, 3.7g of potassium permanganate were gradually added into the above samples and stirred for more 12 hours to prevent its explosion (reaction mixture temperature was increased up to 35°C). Finally, the reaction was stopped. To produce single-layer oxide graphene, the resulting suspension was ultrasonic in 40 kHz for 30 minutes. The produced graphene was then centrifuged, washed with 3% HCl solution, and filtered 3-4 times with using Buchner funnel (and in the end, they were dried in vacuum oven at 40 °C for 24 hours).

Moreover, the calcium nitrate tetrahydrate $(Ca(NO)_3)_2$. 4 H_2O. Sigma- Aldrich and silicon dioxide (40% suspensions soluble in Sigma-Aldrich) were solved and strung for 30 minutes at RT. In the next step, Citric acid ($C_6H_8O_7.H_2O$: Fisher) was entered into the above solution, in that the produced GONs portion reduced with tetra-ethoxylatedsilane. To make a uniform solution ethylene gigolol (EG. C_2H_6O Sigma- Aldrich) with molar ratio EG: CA = 2) was used and immediately after that solution stirred to full liquid evaporation at 80 to 100°C and dried for 24h in the oven. The resulting suspension is filtered and calcined for 3 h at 800 to 850°C. GO was finally formed in the structure of the trapped C_2S and GO/ C_2S composite.

Portland cement Type II from Peyvand Golestan Cement Company, Iran complying with ASTM C150 standard [14] after that was used in this investigation. Table (1) presents chemical and Table (2) shows the physical properties of cement (For more details, see our recent works [15-17]. And GO synthesized can be found in our work [18].)

The mix proportions and sample preparation are the same procedure as reported in our recent works [17,18] and performed according to ASTMC109 standard [19]. For improving and enhancing the quality factor, C_2S with 0.1, 0.2, 0.4

Table 1: Chemical compositions of cement (wt. %).

Items	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	LOI	C_3S	C_2S	C_3A	C_4AF
PC	21.06	5.22	3.68	63.90	1.38	1.81	2.40	54.57	19.51	7.61	11.19

* PC: Portland cement.

Table 2: Analysis of the physical properties of cement.

	Time (min)	Time (min)	C.S	C.S	C.S	C.S
Blaine (cm^2/g)	Initial setting	Final setting	3 days	7 days	28 days	Autoclave (%)
3115	170	195	21	33	46	0.12

C.S: Compressive Strength (MPa)

and 0.8 wt.% GONs were synthesized and mixed with Portland cement (2- fold weight order). On the other hand, process emission from cement production reached a peak in 2016 at about 1.46 CO_2. After China (63%), India, USA, Iran, Turkey, and Brazil account for 59.31, 6.86, 1.90, 1.76, 1.75 and 1.71% of all cement produced in the world, respectively [1]. In addition to cited countries, the cement production in terms of million ton CO_2, in South Korea (Brazil) increased from 16-30 (5-35) during 1990-2015 (1970-2010), whilst European countries have no tendency to produce cement due to negative environmental effects caused by its production [20-24]. However, cement plays an important role in the building, bridge, and so on, which demonstrated that researchers could not put cement aside.

In our previous work [18], we pointed out that one ton of CO_2 was emitted in the atmosphere in the cement production process. It is clear from below chemical reaction and FTIR analysis, the environmental prolusion and contaminations are due to cement production.

$$O_2 + C \rightarrow CO_2 \quad (1)$$

$$CaCO_3 \rightarrow CO_2 + CaO \quad (2)$$

To produce cement and concrete with relatively lower pollution, the effect of different nano particles additives for improvement in the characteristic of cement based materials and industry have been studied. These nano particles including montmorillonite [10], clay [25], SiO_2 [26], Fe_2O_3 [26, 27], Al_2O_3 [27], TiO_2 [28], CNT (Carbon nano tube) [15, 29], ZrO_2 [30], ZnO_2 [31] and SiC [17].

Therefore, for sustainable development and maintenance of environments, in the present work, nanocomposite with lower stress, better quality factor and lower dissipation factor has been studied.

DISCUSSIONS

Some relevant techniques and analyses were applied to study the chemical reaction, mechanical, and nanostructural characteristics. The chemical structure of GO and belite with an Infrared system of Thermo Scientific Company shown in Fig. 1. The FT-IR spectra of GO and C_2S (Fig. 1(a) and (b)) show three vibrational regions: the vibrations of the O-H bond of the graphene-adsorbed water molecules (at about 2800 Cm^{-1}), the vibration C-O and the bonding vibration C-OH (at in 1227 and 1440 (Cm^{-1})) and the relatively large width of 1720 (Cm^{-1}), vibrational tensile bond without carbonyl or carboxylic C = O, such as carboxylic anhydrates and ketones [10]. In addition to the above regions, the other significant regions should be taken into account. The carbon network C = C, with changeable intensity due to the presence of the bound surface of the water peak around the wave number of 1585(Cm^{-1}), dominated peak at the wave number of 3452 (Cm^{-1}), O-H transplants, asymmetric tensional vibration bonds in FTIR spectra (Fig. 1), the Si-O vibrational bonds, and

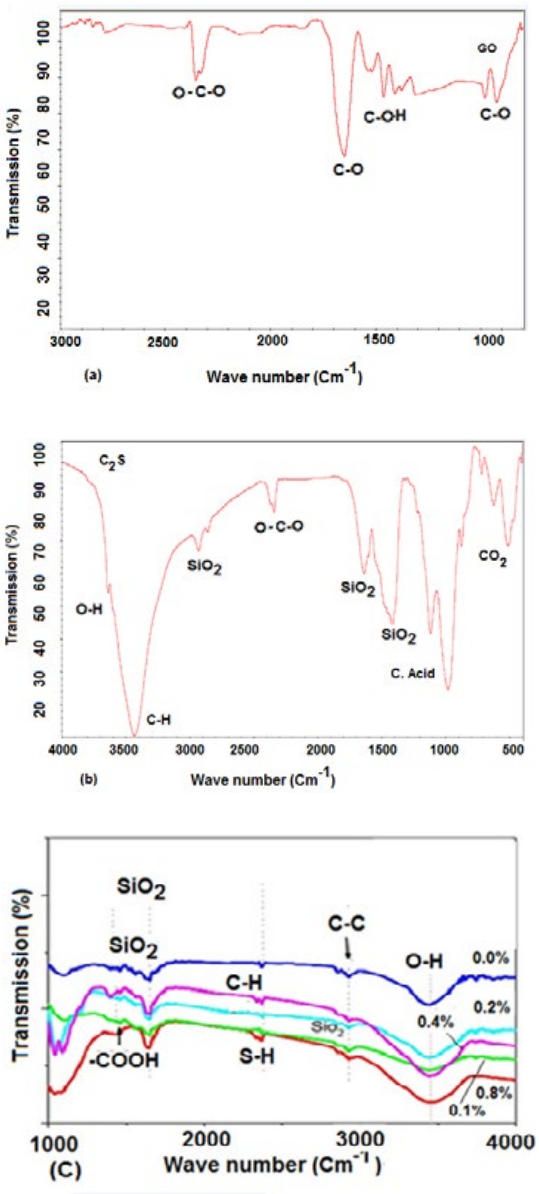

Fig. 1. (a) FT-IR pattern of GO, (b) FT-IR pattern of dicalcium silicate and (c) C_2S/ GO with 0.1, 0.2, 0.4 and 0.8 in wt.% GO.

the shape of the peaks located at the wave number 2950, 1649, 1460, and 1110 (Cm^{-1}), are related to the amorphous SiO_2 are of importance.

The mild peaks in the range of wave numbers; at wavenumber 2842 and 2330 (Cm^{-1}) are also characteristic of the C-O and C-H groups. It also reveals that the tension bond S-H (at wave number 2353(Cm^{-1})) and absorption peak of the region 1649 (Cm^{-1}) attributed to flexural vibrations of carbon dioxide, wide and moderate peak around the wave number 2330 (Cm^{-1}) which confirmed the presence of O = C = O carbon dioxide should be analyzed with an emission factor [1]. Of course, there is another sharp peak (at 1400 (Cm^{-1})) which demonstrated that calcium silicate composite with GONs related to carboxylic acid groups is successfully formed. In the range of 968 to 971 Cm^{-1}, it is related to stretch vibration for $SiO(\upsilon_3)$ functional group [45-47] which is no significant change between samples containing nanomaterials and the control sample.

To study the effects of particle size on sample surface morphology, chemical structure and quality factor of the present samples and sustainable development potential of nano C_2S/ GON, as environmentally – friendly material substitutes, X-Ray Diffraction (XRD) patterns with Philips X-Ray Diffraction PW1730 was used in order to determine C_2S phases and final compounds of GO/C_2S composite (Fig. 2).

XRD spectra exhibit GO/C_2S nanocomposites, synthesized by Pechini method with weight percentages of 0. 1, 0. 2, 0. 4, and 0. 8 wt.% GONs have different crystallites phases and structures, as also analyzed with using the Williamson- Hall approach [10]. The data in Fig. 2 and Tables 3,4 and 5 indicate that different crystallite's phases with 0.1, 0.2, 0.4 and 0.8 wt.% GONs have different dimensions, so that the belite, synthesized in one stage can be considered as a control sample and stored in dry conditions. The dimensions of the composite crystallites are found using Scherer equation as following [10]:

$$D = \frac{k\lambda}{\beta_{hkl} cos\theta} \quad (3)$$

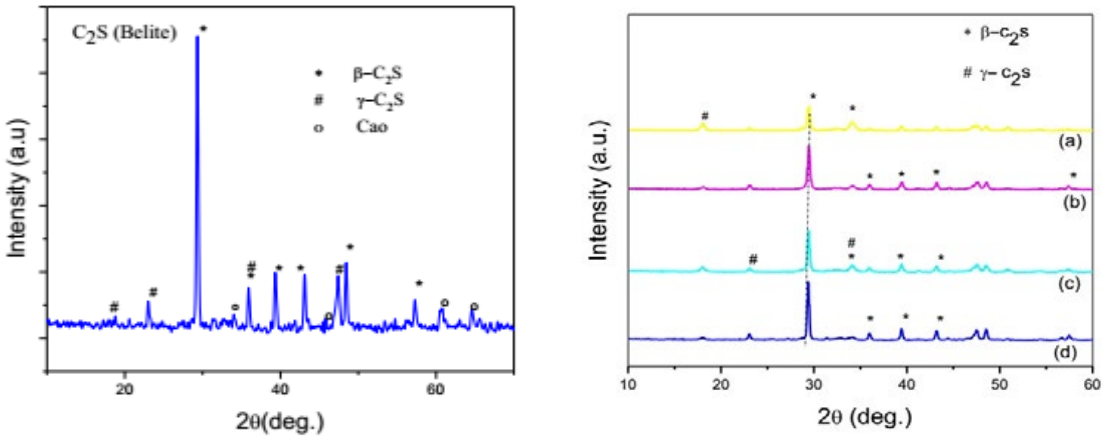

Fig. 2. XRD pattern of (Left): GO/ C_2S composite and (Right): dicalcium silicate synthesized by Pechini method

Table 3. Different phases of C_2S and their particle sizes calculated with X-powder software and Scherer equation

Dielectric	Crystallite phases	Crystallite Size (nm) Scherer	Crystallite Size (nm) X- Powder
$\beta - C_2S$	Monoclinic	9	11.7
$\gamma - C_2S$	Monoclinic	9	12.1

Table 4. Dimensions of dicalcium silicate nanocrystals

2θ(deg.)	23.0	31.4	35.2	43.1	48.4	53.4	57.2	72.8	83.4	89.1
Intensity (Arb.)	185	53.9	432.1	490.0	62.1	199.2	54.0	78.0	46.2	49.2
FWHM (deg.)	0.23	0.35	0.25	0.28	0.29	0.23	0.23	0.35	0.23	0.28
Size (nm)	20	9	31	36	36	31	28	20	10	19

In Eq. (3); D, β, and θ is the size of nano-crystallite, full width at half maximum (FWHM), and Bragg's angle, respectively. The shape factor, k, (equal to 0.94) and λ (the wavelength of incident radiation, Cu_{ka} = 1.5406 Å) are known from Refs. [4, 10, 18]. In Table 3, it is clear that the stated sample has two distinct structures of gamma-dicalcium silicate and beta-dicalcium silicate, which shows a good dispersion of GON's size regardless of the network interaction.

In addition, another key factor which causes environment's contamination is due to occurring of the stresses (caused by defects), crystalline defects. Williamson- Hall relation is therefore used for estimating strain in the samples [18].

$$\beta_{hkl}\cos\theta = \frac{k\lambda}{D} + 4\epsilon \sin\theta \qquad (4)$$

Fig. 3 shows FWHM of the dominated peak found with Scherer equation and X- Powder software. The network stress (Fig. 4) indicates that stresses in dicalcium silicate and C_2S with 0.4 %wt. GONs are directly related to the crystal size (see Fig. 3 and Table 5). The slope of line for Beta-dicalcium silicate in Fig. 4 is positive, meaning that stresses are tensional for GO/C_2S composite with C_2S /GO (with 0.1%, 0.2%, 0.4%, and 0.8% wt. GO nanoparticles) show that nanocomposite with 0.4 in wt% GO nanoparticles can significantly fill C_2S cavities. This point demonstrated that the asymmetric presence of GO in the C_2S plates could increase the reactivity intensity of the gel C-S-H vibration peak.

Moreover, as reported in Ref. [18], known as Hall- Petch relation, there is an inverse relationship

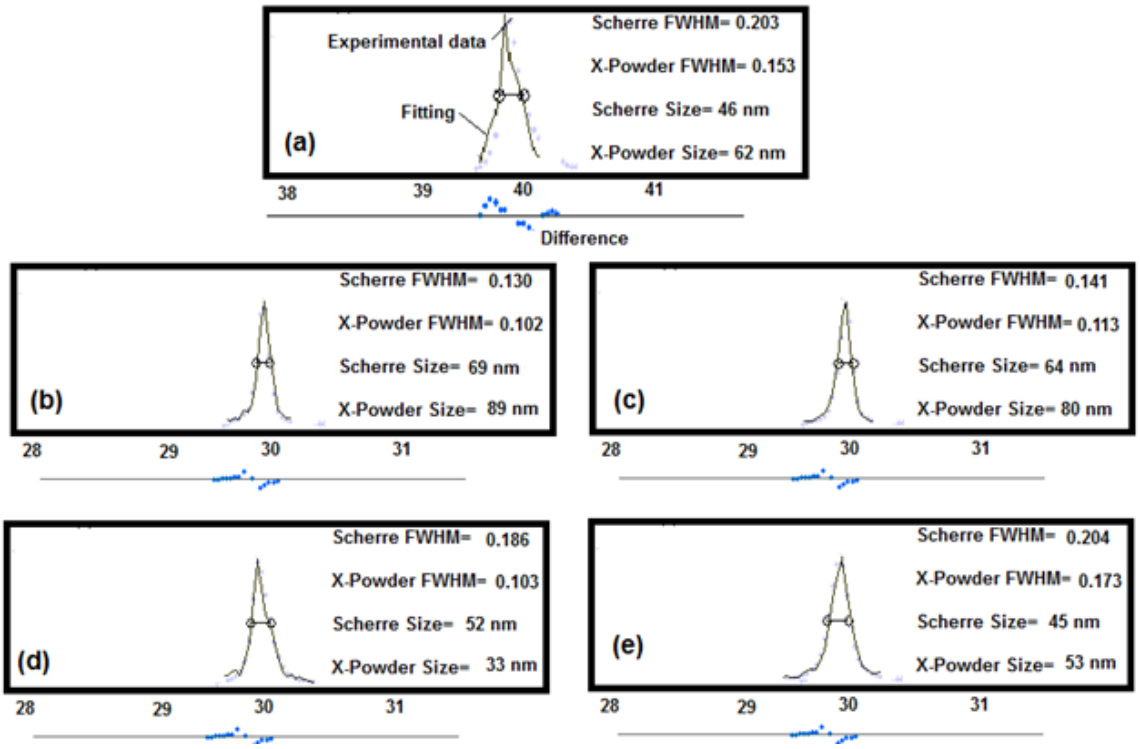

Fig. 3. X- Powder measurement of crystallite phase and β (FWHM): (a) C_2S, (b) C_2S+ 0.1 wt.% GONs, (c) C_2S+ 0.2 wt.% GONs, (d) C_2S+ 0.4 wt.% GONs and (e) C_2S+ 0.8 wt.% GONs

Table 5. Dimensions of dominated peak at 30° for C_2S+ 0.1, 0.2, 0.4 and 0.8 wt.% GONs nanocrystals

sample	FWHM (Scherer)	FWHM (X- powder)	Size (nm) (Scherer)	Size (nm) (X- powder)
C_2S+ 0.0 wt.% GONs	0.203	0.153	46	62
C_2S+ 0.1 wt.% GONs	0.186	0.103	52	33
C_2S+ 0.2 wt.% GONs	0.141	0.113	64	80
C_2S+ 0.4 wt.% GONs	0.130	0.102	60	89
C_2S+ 0.8 wt.% GONs	0.204	0.173	45	53

of mechanical strength of sample with the square root of average grain diameter (d) (Eq. 5). The smaller size of the sample, the stronger mechanical structure of the sample will be formed.

$$\sigma_y = \sigma_0 + \frac{k_y}{\sqrt{d}} \quad (5)$$

where σ_y, σ_0, k_y are the yield stress, materials constant for the starting stress for dislocation movement, the strengthening coefficient, respectively.

In parallel to mechanical studies, the emission factor for the production of cement, E_{cement}, from [1], 4- capacitors with C_2S- GONs (sandwich between Al and Si) were fabricated. The wafer capacitor surface and wafer thickness is A = 1.13 cm^2 and d=100 nm and$\varepsilon_0 = 8.85 \times 10^{-12}\left(\frac{C^2}{Nm^2}\right)$ and the capacitance of the samples, measured by GPS 132 A tool. The dielectric constant (k) is calculated using the equation $= \frac{cd}{\varepsilon_0 A}$.

The other parameters such as quality factor (Q_F), and dissipation factor (D_F) were also measured.

$$Q_F = 2\pi \frac{Energy\ stored}{energy\ dissipated\ per\ cycle} \quad (6)$$

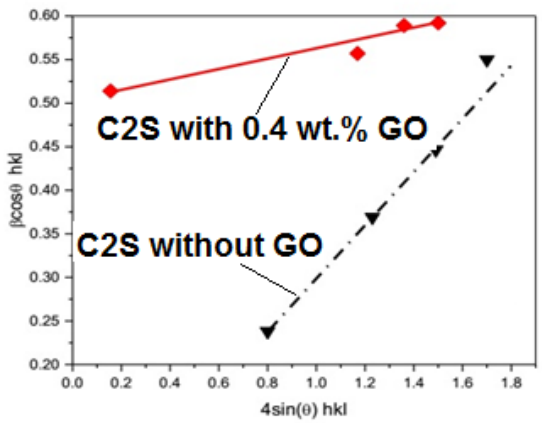

Fig. 4. The linear relationship between βcosθ and 4sinθ in the Williamson-Hall diagram (according to the XRD spectra of nanocrystallites of GO/C_2S silicate, synthesized by Pechini method).

$$D_F = 1/Q_F \quad (7)$$

The results show that the maximum value of the dielectric constant for C_2S +0.4 %wt. GO at the frequency of 120 and 1 kHz is k=62 and k = 30, respectively (Table 6). Higher (lower) Q_F (D_F) value indicates a lower rate of energy loss (loss-rate of the energy of the capacitor) with respect to the stored energy of the capacitor as demonstrated in Eq. (6).

The estimated the fraction emission factor, f, (Eq. 8) (by looking at XRD patterns in Figs. 2 and 3) for samples in Table 6 at 120 kHz for C_2S with 0.2, 0.4 and 0.8 wt.% GONs is about 0.790, 0.625 and 0.643, respectively.

$$E_{cement} = f_{Cement}^{CaO} \frac{M_r^{CO_2}}{M_r^{CaO}} \quad (8)$$

where E_{cem} is emission factor [1], f is the fraction of CaO in cement, $M_r^{CO_2}$ and M_r^{CaO} is the molecular weight of CO_2 (44.01), and the molecular weight of CaO (56.08), respectively. The obtained result is better (and lower) than that reported in Ref. [1]. Andrew [1] reported that fraction emission factor with the average CaO content of cement is about 0.635.

CONCLUSIONS

In the present work, GONs were synthesized using the Pechini method and then added to the C_2S matrix, as nanocomposite materials mixed to cement (Composite/cement: ½ in wt. ratio). The presence of SiO_2 groups in the chemical structure of GO has been confirmed using the FT-IR spectrum. The variety in the size of the GO/C_2S of nanocrystals of composite structure can increase the effective contact surface of the composite matrix and leads to a strong bond between the network and the nanoparticle.

In order to get a uniform distribution, GO particles of raw graphite were synthesized by Hammer method and then added using Pechini method in the steps of the synthesis of belite. In the synthesize procedure, due to the presence of functional groups, GO was trapped in the belite

Table 6. C, k, Q_F and D_F of GO/ C_2S obtained by using GPS 132 A technique where wafer capacitor surface and wafer thickness is A = 1.13 cm^2 and 2 mm, respectively for frequency=1 kHz and 120 kHz

Samples	120 kHz	120 kHz	120 kHz	1 kHz	1kHz	1kHz
	Q_F	D_F	k	Q_F	D_F	k
C_2S +0.1 %wt. GO	2.5	0.41	11	2.7	0.36	14
C_2S +0.2 %wt. GO	6.5	0.11	34	8.6	0.11	12
C_2S +0.4 %wt. GO	32.4	0.03	62	25.9	0.03	30
C_2S +0.8 %wt. GO	23.1	0.04	56	4.3	0.23	8

network. The results of the XRD analysis indicate keeping crystallization of nanoparticles and the presence of GO phase in the network. The emission factor for sustainable development is estimated, in that C_2S+ 0.4 wt.% GONs with 32.4 quality factor and 0.03 dissipation factor and 0.625 fraction emission factor respected with other present samples has suffix-ability to reduced cement contamination as well as decreasing environmental pollution can be suggested for the future cement-based material industries.

CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

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