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Graphene Oxide-Reinforced Cementitious Concrete Composites Incorporates Silica Fume And Fly Ash

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Abstract

In recent years, nano-sized graphene oxide (GO) has come to the fore as a promising material for enhancing the mechanical and durability performance of cementitious composites. On the other hand, partial substitution of industrial wastes and by-products into cementitious composites attracts the attention of researchers in order to ensure long-term sustainability. In order to combine these two aspects, the main focus of this study is to examine the effect of 0.05% and 0.1% GO-reinforcement on the slump, 7 and 28-day compressive and flexural strength and 28-day rapid chloride permeability test (RCPT) properties of cementitious composites. In this context, mixtures with three different binder combinations, a control, a silica fume (SF) substitution, and a fly ash (FA) substitution, were designed. The results showed that GO-reinforcement reduced the slump values of the mixtures between 5-15 mm, while the 28-day compressive strengths increased in the range of 9.82%-13.61% with 0.05% GO-reinforcement, and in the range of 17.02%-20.68% with 0.1% GO-reinforcement. The 28-day flexural strength of the mixtures increased by about 10% on average as a result of 0.1% GO-reinforcement. According to the RCPT anaylses, it was observed that the chloride permeability of the mixtures decreased up to 18.85% with 0.1% GO-reinforcement.

Key Words

Graphene-oxide, nanomaterial, cement, mechanical properties, RCPT.

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1. Introduction

Cementitious composites are widely used to build civil structures (Chakraborty et al., 2013). Although these are used a wide range of structural applications, they are brittle and have poor tensile strength. There is a need to minimize the potential consequences of deterioration. Because brittleness and poor tensile strength deteriorate the hardened concrete on exposure to harsh environmental conditions in time. Important structures such as dam, skyscraper may be exposed to harsh environments and severe loading conditions. Hence, the durability of structures is one of the most important properties, particularly for structures exposed to harsh environments (Neville, 1995; Mindess et al., 2003; Mehta and Monteiro, 2006). In this way, increasing the performance of cement-based materials is of big significance for durable and environment-friendly structures. The durability properties of concrete structures are mightily affected by its permeability properties to the harmful agents such as water, CO_2, chloride, etc. (Mehta, 2001; Sahmaran et al., 2007). As it is known, different fibers or steel bars have been applied to limit the propagation of micro-cracks to develop the mechanical, permeability properties of plain cementitious materials (Nemkumar and Gupta, 2006; Sahmaran et al., 2009, Sahmaran et al., 2013).

Today, nanomaterials have been extensively used to cementitious materials as nanofillers owing to progress in nanotechnology (Jo et al., 2007; Stefanidou and Papayianni, 2012). It is worthy widely the use of nanomaterials in cementitious materials, enabling such materials with higher mechanical strength, durability, toughness, multifunctional for the next generation buildings and structures. During recent years, there has been increasing attention in the use of nanomaterials in construction materials to increase mechanical, permeability, durability performances and to create multifunctional capabilities (Sobolev and Ferrada-Gutierrez, 2005; Metaxa et al., 2012; Chen et al., 2012; Kawashima et al., 2013; Jiang et al., 2021). These nanomaterials fill the gaps in the cementitious composites, causing high compactness and higher strength (Metaxa et al., 2012; Chen et al., 2012; Kawashima et al., 2013). So far, nanomaterials such as graphene nanoplatelets (GNP) (Jiang et al., 2021), carbon nanotubes (CNTs) (Chaipanich et al., 2010; Konsta-Gdoutos et al., 2010; Azhari and Banthia, 2012; Sobolkina et al., 2012; Al-Dahawi et al., 2016), carbon nanofibers (CNFs) (Tyson et al., 2011; Konsta-Gdoutos and Aza, 2014), nano $CaCO_3$ (Kawashima et al., 2014), nano SiO_2 (Gaitero et al., 2008; Quercia et al., 2014; Hou et al., 2015), nano TiO_2 (Krishnan et al., 2013; Li et al., 2014), carbon black (Ding et al., 2013; Rezania et al., 2019) have already attracted the attention of different researchers owing to their extraordinary mechanical properties.

Another kind of carbon-based nanomaterial is the graphene oxide (GO). GO is promising nanoscale carbon-based materials. Attractive advantage of GO is the low cost when compared to other carbon-based nanomaterials, such as CNTs and CNFs, whose large-scale production is considerably more expensive (Lu et al., 2008; Kostarelos and Novoselov, 2014). Graphene oxide (GO) can be used as a nano-reinforcing agent in cementitious materials like CNPs. GO has shown up as a precursor filler nanomaterial offering the potential of cost-effective, large-scale production of graphene-based cementitious materials (Park and Ruoff, 2009). Graphene is a carbon-based nanomaterial because of its unprecedented properties (Geim and Novoselov, 2007), such as high intrinsic mobility of 200,000 cm2 $v^{-1}s^{-1}$ (Bolotin et al., 2008; Morozov et al., 2008), a large specific surface area of 2630 m^2/g, and high Young's modulus of about 1.0 TPa (Lee et al., 2008). Graphene oxide (GO) is one of the surface-functionalized forms of graphene and have extraordinary properties such as high strength, high Young's modulus, and high specific surface area (Zhu et al., 2010). GO in some of the recent investigations has been used as-synthesized or as-received (Lv et al., 2013; Pan et al., 2015; Tong et al., 2016; Devi and Khan, 2020). Striking improvement in strength (Lv et al., 2013; Tong et al., 2016), and permeability properties (Pan et al., 2015; Devi and Khan, 2020) have been presented.

Despite the above-mentioned benefits, large-scale application of graphene-based cementitious composites are facing difficulties such as its uniform dispersion. For GO to show their superior properties in cementitious composites, they must be well dispersed in the mixing water and then in concrete. The superior properties of GO in reinforcing cementitious composites can solely be achieved with well dispersion and distribution in the concrete. The literature review has shown that the GO by use with high shear mixing has been studied very limited in the concrete composites. The aim of this study is to investigate effect of GO on the mechanical and durability properties of concrete. To this end, GOs were synthesized according to the modified Hummer's method and then cementitious concrete composites reinforced with GO with and without FA and SF were produced. Compressive, flexural, and rapid chloride ion permeability test (RCPT) properties of GO-reinforced cementitious concrete composites were tested.

2. Materials and Methodology

2.1. Materials

Ordinary Portland cement, potable water, fine aggregate, coarse aggregate, GO, fly ash (FA), and silica fume (SF) were used in this study. The chemical properties of cement, FA and SF were given in Table 1.

GOs were synthesized according to the modified Hummer's method (Shahriary et al., 2014). In the first stage, 1 g of graphite and 0.5 g of $NaNO_3$ were mixed in a flask. Then, 50 ml of H_2SO_4 (98%) was added under continuous stirring at 5 °C for 1 h. Afterward, 3 g of $KMnO_4$ (1 g every 15 min) was included and special attention was paid to avoid overheating and explosion by keeping the solution temperature under 20 °C. The solution was then diluted by slowly adding 100 ml of distilled water and the solution was treated with 3 ml of 30% H_2O_2 solution and finally, 100 ml of distilled water was added in order to be sure that $KMnO_4$ is completely reacted in the end, the solution was washed with HCl and water and filtrated to obtain dry GO. High shear mixing with 30 min, at 5000 rpm speed was used to disperse the agglomerates and stabilize the GO particles in water solution. High shear mixing with 30 min, at 5000 rpm speed were chosen suggestion of Jiang et al. (2021) and Ozbulut et al. (2018). This uniform dispersed water solution of GO (0.05 wt% and 0.1 wt%) was mixed with cement and aggregates to produce concrete.

Table 1. The Chemical Properties Of The Materials

Chemical composition (%)	Cement	Silica fume	Fly ash
SiO_2	21.04	96.29	36.27
Al_2O_3	4.88	0.24	15.28
Fe_2O_3	2.55	0.05	4.19
CaO	64.26	0.51	29.12
MgO	1.71	0.56	2.04
Na_2O	0.33	0.22	0.62
K_2O	0.41	0.30	1.06
SO_3	3.19	0.53	6.78

2.2. Mixture Proportions

In this study, in order to investigate the effect of different GO ratios on the properties of concrete, control mixtures, and mixtures including 0.05% and 0.1% by weight of GO were designed. The water/cement ratio was kept constant at 0.5. The fly ash and silica fume were replaced by cement at 18.6% and 6.97% by weight of cement, respectively. High shear mixing time was kept constant at 30 min at 5000 rpm speed for all mixtures in order to provide a uniform dispersion of GO and avoid agglomerations. The detailed mix proportions of the mixtures were given in Table 2.

Table 2. Mix Proportions of GO-Based Concrete Mixtures (kg/m^3)

Sample ID	Cement	FA	SF	Water	Coarse Aggregate	Fine Aggregate	GO
Control	430			215	850	804	-
SF	400		30	215	850	804	-
FA	350	80		215	850	804	-
0.05GO	430			215	850	804	0.215
0.05GO.SF	400		30	215	850	804	0.215
0.05GO.FA	350	80		215	850	804	0.215
0.1GO	430			215	850	804	0.430
0.1GO.SF	400		30	215	850	804	0.430
0.1GO.FA	350	80		215	850	804	0.430

2.3. Curing and Testing

Slump, compressive and flexural strengths, and rapid chloride permeability test (RCPT) was applied to designed mixtures. After casting, all the samples were covered with a sheet to inhibit water loss and all samples were removed from the mold after 24 hours. All samples were immersed in potable water until testing day.

For compressive strength test, four cylindric samples with the dimensions of Φ10 × 30 cm cylinders were produced and water cured for 7 and 28 days. Compressive strength test was applied in accordance with ASTM C 39, (2005). For measuring compressive strength, samples were loaded at a loading rate of 0.25 ± 0.05 MPa/s and results were averaged.

For flexural strength testing, three prismatic samples with the dimensions of 10 × 10 × 40 cm were produced and cured for 7 and 28 days. Flexural strength test was applied in accordance with ASTM C 78, (2018). Prismatic specimens were subjected to three-point bending loading between 0.9 and 1.2 MPa/min in to determine the flexural strength, and the maximum flexural load was recorded in order to compute the results.

RCPT of GO-reinforced cementitious concrete composites were explored for 28 days following with ASTM C 1202, (2012). Cylindrical specimens of GO-reinforced cementitious concrete composites with the dimensions Φ10 × 5 cm were prepared. For each mixture, four specimens were tested. During testing, concrete specimens were placed in the experimental cell one end of that was in contact with 3% NaCl solution and the other with 0.30 M NaOH solution. Each specimen's total current flow during the course of 6 hours at a constant voltage of 60.0 ± 0.1 V was noticed and recorded in Coulombs (C) units.

3. Results and Discussion

3.1. Slump Test

Slump test results of the mixtures designed within the scope of the study were presented in Figure 1. The slump values of the control, SF and FA coded mixtures resulted in 115, 110 and 120 mm, respectively. As it is known in the literature, the spherical grain shape of FA significantly improves the workability and slump value of concrete mixes, thanks to the ball bearing effect that provides lubrication between cement particles (Shaikuthali et al., 2019). In the mixture containing SF, it can be stated that the slump values decrease due to the very high specific surface of SF (Imam et al., 2018). Besides, it was observed that 0.05% GO substitution reduced the slump values of the control mix, SF substituted mix, and FA substituted mix by 4.35%, 4.55%, and 8.33%, respectively. 0.1% GO substitution reduced the slump values of the control mix, SF substituted mix and FA substituted mix by 8.70%, 9.09% and 12.5%, respectively. Similar results were reported on the decrease in workability with the addition of GO in the previous studies (Li et al., 2018; Chuah et al., 2018).

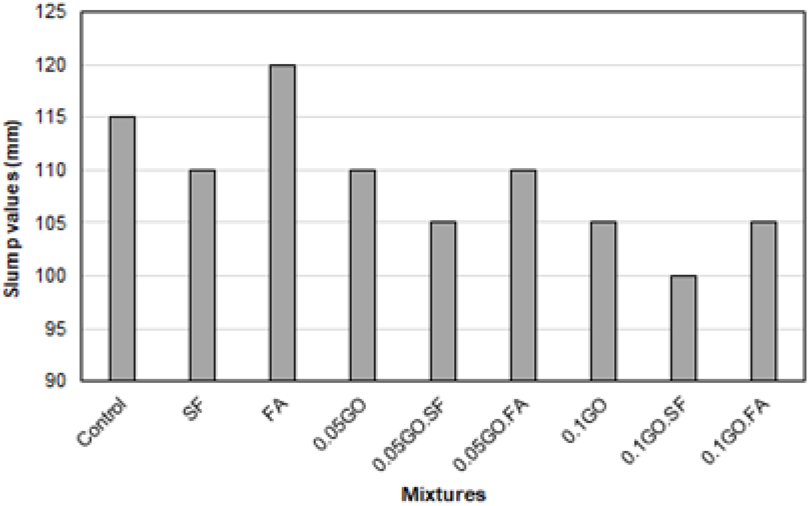

Figure. 1. Slump Values of Different GO-Reinforced Concrete Composites.

3.2. Compressive Strength Test

The 7- and 28-day compressive strength results of the mixtures designed within the scope of the study were presented in Figure 2. Results showed that SF and FA addition caused a decrease in 7-day compressive strength according to the control mixture by 5.29% and 8.37%, respectively. On the other hand, while SF inclusion caused an increase in the 28-day compressive strength result by 1.31%, FA inclusion yielded a decrease of 2.35% at the same age. As it is known, when supplementary cementitious materials such as SF and FA are included in cementitious systems, they generally decrease the strength at an early age, while they contribute to the achievement of better strengths compared to the control mixture with the increase in pozzolanic activities in later ages (Chandra and Hardjito, 2015).

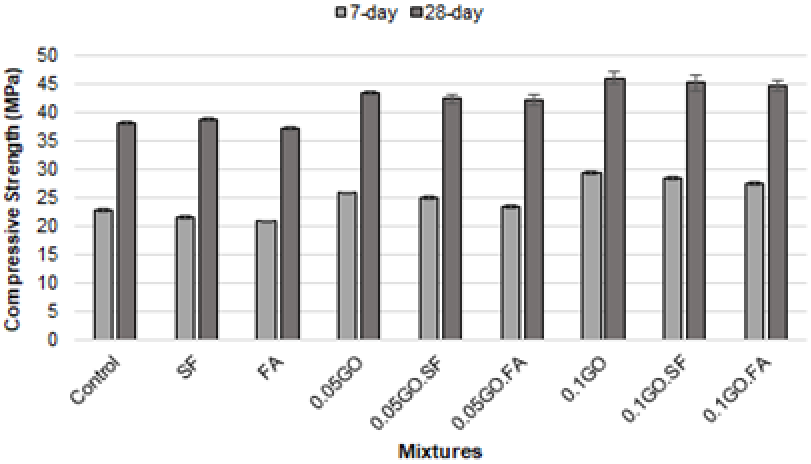

Figure. 2. Compressive Strength Test Results of Different GO-Reinforced Concrete Composites

As shown in Figure 2, it was observed that the GO reinforcement significantly improved the compressive strengths of the mixtures. In this context, the compressive strength of the control mixture increased by 15.85% after 28 days with 0.05% GO inclusion, while an increase by 24.09% was detected at the same age with 0.1% GO inclusion. In the SF substituted mixture, 0.05% and 0.1% GO substitutions increased the compressive strength of the samples by 9.82% and 19.12%, respectively, at the end of 28 days. A similar trend was also seen in FA substituted mixtures and 0.05% and 0.1% GO inclusion resulted in an increase of 13.40% and 19.84% in the compressive strength of the samples, respectively, at the end of 28 days. The rise in the mixes' compressive strengths can be attributed to the high specific surface area of GO, which promotes the nucleation of cement hydrates and generates strong covalent connections at the interface between the cement matrix and GO. Therefore, addition of GO to the mixtures, resulting in improvement of the compressive strength of GO-reinforced concrete composites. Previous studies in the literature stated that a small amount of GO additives increased the compressive strengths significantly (Lv et al., 2013; Gong et al., 2015; Pan et al., 2015; Devi and Khan, 2020). Also, the increase in compressive strength confirmed that GO is well dispersed in concrete composites without agglomeration.

3.3. Flexural Strength Test

The flexural strength results of GO-reinforced concrete composites after the 7- and 28-day water curing were presented in Figure 3. Similar to the compressive strength results, the control mixture showed a higher flexural strength of 3.68 MPa at 7 days than the SF and FA substituted mixtures, while the SF substituted mixture showed the highest flexural strength result among these three mixtures with 5.24 MPa at the end of 28 days.

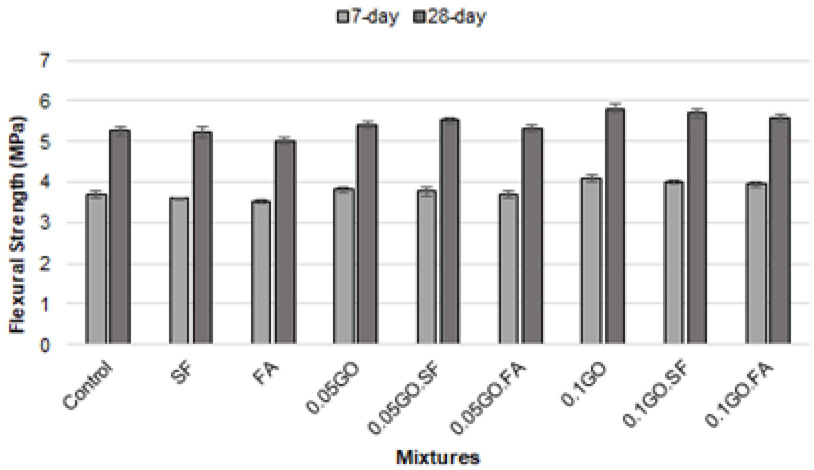

Figure 3. Flexural Strength Test Results of Different GO-Reinforced Concrete Composites

Considering the flexural strengths of GO-reinforced mixtures, the mixture produced with 0.1 GO and 100% Portland cement exhibited the highest flexural strength result with 5.82 MPa at the end of 28 days of curing. The mixtures produced with 0.1 GO and SF and FA substitution showed results close to the Portland cement mixture with flexural strengths of 5.71 MPa and 5.58 MPa, respectively, at the end of 28 days. In general, it was observed that the GO substitution significantly improved the flexural strengths of the mixtures. This is due to the fact that GO significantly improves flexural load transfer by forming strong interface adhesion with hydration products in the cement matrix (Pan et al., 2015).

3.4. Rapid Chloride Permeability Test

The rapid chloride ion permeability test (RCPT) results of GO-reinforced concrete composites was after the 28 days curing were presented in Figure 4. According to the RCPT analysis, the Coulomb value of the control mixture was 3851, while the Coulomb values of the SF and FA substituted mixtures were found to be 3885 and 3786, respectively. As a result of 0.05% GO-reinforcement, Coulomb values decreased by 11.01% for the control mixture, 9.88% for the SF substituted mixture and 7.24% for the FA substituted mixture. The Coulomb values of the mixtures designed with 0.1% GO-reinforcement resulted in 3125, 3201 and 3219 for the control, SF substituted and FA substituted mixtures, respectively. As can be clearly seen from the results, it can be said that the GO-reinforcement has a very positive effect in terms of durability as well as mechanical improvement by significantly reducing the chloride ion permeability of the mixtures. The decrease in Coulomb values as a result of GO-reinforcement was due to the interconnected layers of the GO and the ability to retain chloride ions (Shanmuga et al., 2021). Results were found to be very consistent with the compressive and flexural strength test results.

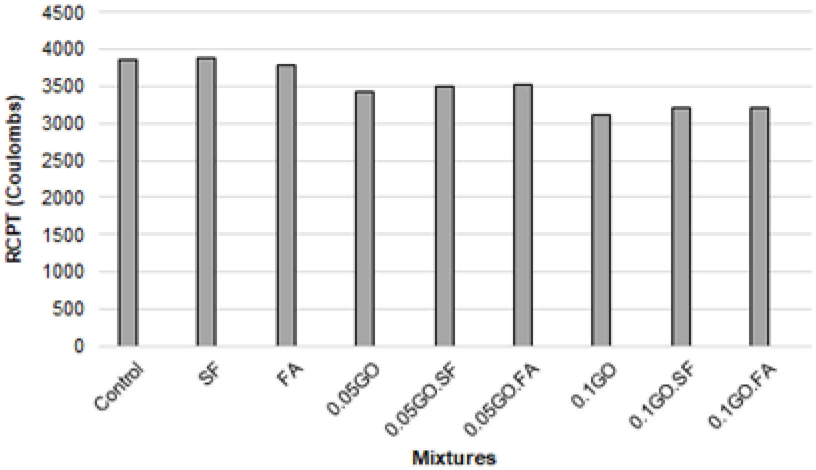

Figure 4. The 28-Day RCPT Results of Different GO-Reinforced Concrete Composites

4. Conclusion

In this study, slump, compressive and flexural strength and rapid chloride permeability test (RCPT) characteristics of 0.05% and 0.1% graphene oxide (GO) reinforced cementitious concretes were investigated. Within the scope of the study, the control mixture incorporates entirely Portland cement, 18.6% fly ash (FA) substituted mixture and 6.97% silica fume (SF) substituted mixture were designed. The main findings of the study can be summarized as follows:

- While the slump value of the control mixture was found to be 115 mm, it decreased by 5 mm for each 0.05% GO addition. In SF substituted mixtures, each 0.05% GO addition to the control mixture caused a 5 mm decrease in slump values. In FA substituted mixtures, the slump value decreased from 120 mm to 110 mm and 105 mm, respectively, after each 0.05% GO addition.
- The addition of 0.05% GO increased the 28-day compressive strengths of the control mixture, SF and FA substituted mixtures by 15.85%, 9.82% and 13.40%, respectively, while the addition of 0.1% GO increased the compressive strengths by 24.09%, 19.12% and 19.84%, respectively. In this context, the highest compressive strength in this study was achieved in the 0.1 GO-reinforced Portland cement concrete with the 46.1 MPa after the 28-day curing.
- The flexural strengths of the mixtures designed within the scope of the study increased by 2.65% for the control mixture, 5.34% for the SF substituted mixture and 5.98% for the FA substituted mixture at the end of 28-day as a result of the addition of 0.05% GO. When the GO addition was 0.1%, the flexural strengths increased by 10.43% for the control mixture, 8.97% for the SF substituted mixture and 11.16% for the FA substituted mixture. The highest flexural strength after the 28-day curing was obtained with 5.82 MPa from the mixture in which the binder phase consisted of 100% Portland cement and 0.1% GO was added.
- According to the RCPT analysis results, the addition of 0.05% GO caused the Coulomb values of the control, SF-substituted and FA-substituted mixtures to decrease by 11.01, 9.88 and 7.24%, respectively. The addition of 0.1% GO decreased the Coulomb values of the control, SF-substituted and FA-substituted mixtures by 18.85%, 17.61% and 14.98%, respectively. GO-reinforcement showed a very positive effect by significantly reducing the chloride ion permeability of the mixtures.

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