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Investigation of the Reduced Graphene Oxide Effect on the Concrete Strength, Impedance and Corrosion Resistance of Rebar Inside

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Abstract

In this work, to investigate the effect of reduced Graphene Oxide nanoparticles on the strength of concrete and the corrosion resistance of rebar inside it, Graphene Oxide nanoparticles were added to the mortar with different weight percentages of cement. The strength of concrete was evaluated by measuring the compressive strength of 7 days and 28 days and comparing with the results of the control sample. To investigate the effect of reduced Graphene Oxide nanoparticles on the corrosion resistance of rebar inside it, polarization and EIS tests were performed in a 3.5% salt solution. The results showed that the addition of reduced Graphene Oxide nanoparticles increases the compressive strength and electrical impedance of the concrete. It was also found that there is an optimal value that the addition of Graphene Oxide nanoparticles more than that value causes a relative decrease in concrete strength and electrical impedance. Finally, by simulating the equivalent circuit, the reason for the decrease in impedance and as a result the corrosion resistance by adding more than the optimal value was investigated.

Keywords: Reduced graphene oxide, Compressive strength, Corrosion, Impedance, EIS.

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Introduction

Cement is a chemical product that has adhesive properties. When cement is mixed with sand and water, it hardens and forms a solid material called concrete. Nowadays, the use of concrete structures is expanding due to raw materials and low cost. Increasing the strength of concrete is an important requirement for the design of light structures. Different materials can affect the strength of concrete. Nowadays, Nano technology is used to improve the quality of concrete. Nano particles improve concrete by filling the pores and preventing the penetration and expansion of water. Also, nanoparticles prevent the growth of calcium hydroxide crystals. So far, the effect of different nanoparticles such as carbon nanotubes [1], zinc Oxide [2-4], Iron Oxide [5,6], titanium dioxide [7,8], alumina [9,10], copper Oxide [11] Zirconium Oxide [12], silica [13-19] and CaCO3 [20] on the strength of concrete has been investigated. Among the nanoparticles, nanoparticles based on carbon and Nano silica are very popular [21]. But one of the compounds that can have a significant effect in this direction is Graphene Oxide The use of Graphene Oxide nanoparticles is more suitable than other carbon-based nanoparticles. Because its production is much cheaper than other carbon nanomaterials such as carbon nanotubes and carbon nanofibers. Graphene Oxide is comprised of a single layer Graphene sheet, covalently bonded to oxygen functional groups on the basal planes and edges of the sheet. On the basal planes, there are both hydroxyl and epoxy groups; the edges can include carboxyl, carbonyl, phenol, lactone, and quinone groups. This feature of Graphene Oxide in the composition of cement mortar can be considered. Functional groups, especially hydroxyl and carboxylic could weakly generate negative charges in the solution due to deprotonation, giving a hydrophilic nature and easily adsorbs free radicles of water from the cement composite prepared. Also, these nanoparticles disperse well in water compared to other nanoparticle [22, 23]. The researchers have reported that Graphene Oxide sheets with oxygenated functions enhance the reaction of cement with water and accelerates the hydration activity [21]. So, many studies have been conducted on the use of Graphene Oxide [24-32] and reduced Graphene Oxide [33-36] in concrete to increase the performance of building materials. It has also been determined that in concrete improved with Graphene oxide, the amount of $Ca(OH)_2$ is higher than in concrete improved with reduced Graphene oxide [33].

These studies showed that although Graphene oxide and reduced Graphene oxide increase the solubility in cement due to having functional groups such as hydroxyl, epoxide, carboxyl and carbonyl, and serve as a nucleation agent for C-S-H crystals, but them large amounts (due to the absorption and retention of water) reduce the strength of cement. Although the effect of Graphene oxide and reduced Graphene oxide Nano particles on the strength of cement has been well investigated, but its effect on the corrosion resistance of rebar inside it has not been well investigated. One of the uses of concrete, in addition to being used in urban buildings, is marine structures. In the sea environment, many structures such as bridge foundations, breakwaters, barrages, underwater tunnels, etc. are made of concrete. The concrete used in these cases must be high-strength concrete, otherwise the structure will be destroyed after some time. Corrosion of rebar inside concrete is one of the main causes of destruction of concrete structures in the sea environment. Therefore, the use of concrete that protects the corrosion of rebar inside it is a basic need. Considering that when the material loses electrons it is corroded, in order to prevent material corrosion, the tendency and speed of electron loss must be reduced, which means an increase in electrical impedance. Considering that electrical impedance is an imaginary quantity and depends on elements such as capacitance, electrical resistance, etc., to increase the electrical impedance of mortar, its electrical resistance must be increased and its capacitance decreased [37]. To increase the electrical resistance, the use of oxide nanoparticles can be useful. Also, the amount of material added to concrete has an effect on its electrical impedance. Due to the fact that a large number of functional groups have been removed in the Reduced Graphene Oxide nanoparticles, also these nanoparticles in large quantities can have less aggregation properties than Graphene Oxide nanoparticles, Therefore, in this work, the effect of Reduced Graphene Oxide nanoparticles with different amounts added to concrete and its effect on the concrete strength, concrete impedance and corrosion resistance of rebar inside it has been investigated.

Expermental

Each of the concrete materials play an important role in creating its characteristics. Cement causes adhesion between aggregates, and aggregates are responsible for filling, also, bearing weight, and water is used for hydration and smoothness of the mixture. In this work, Portland II cement was used with the compositions given in Table 1.

Table1. Chemical composition of Portland cement II (wt%, determined using X-ray fluorescence).

SiO_2	**MgO**	**Na_2O**	**K_2O**	**SO_3**	**CaO**	**Fe_2O_3**	**Al_2O_3**	**LOSS**
21.9	1.5	0.3	0.5	1.5	65.00	4.20	4.00	1.1

Aggregates and water were added to cement according to the C109 standard to obtain a control sample. Table 2 shows the amounts of materials used to obtain the control sample. To prepare concrete with nanoparticles, Reduced Graphene Oxide nanoparticles with the specifications given in Tables 3 and 4 were purchased.

Table 2. Quantity of materials used in kg/m^3.

Materials	**Quantity of materials (kg/m^3)**
Coarse Aggregate	1058
Fine Aggregate	625
cement	432
Water	206

Table3. Specifications of Reduced Graphene Oxide nanoparticles used in this work.

Sample	powder
Purity (%)	98
Number of layers	6-10
Thickness average (z dimension) (nm)	3-4
Lateral dimension (µm)	>12
Length average (X and Y dimensions) (µm)	5-10
Surface Area (m^2/gr)	>210
Electrical conductivity: Conductor(S/M)	560

Table 4. Chemical composition of Reduced Graphene Oxide used in this work.

C	**O**	**S**	**H**	**N**
87	10	<1	2	<1

Table 4 shows that the functional groups were largely removed and only 10% of oxygen is present in the structure of these nanoparticles. FTIR spectrum and SEM images of reduced Graphene oxide nanoparticles are shown in Figures 1 and 2, respectively.

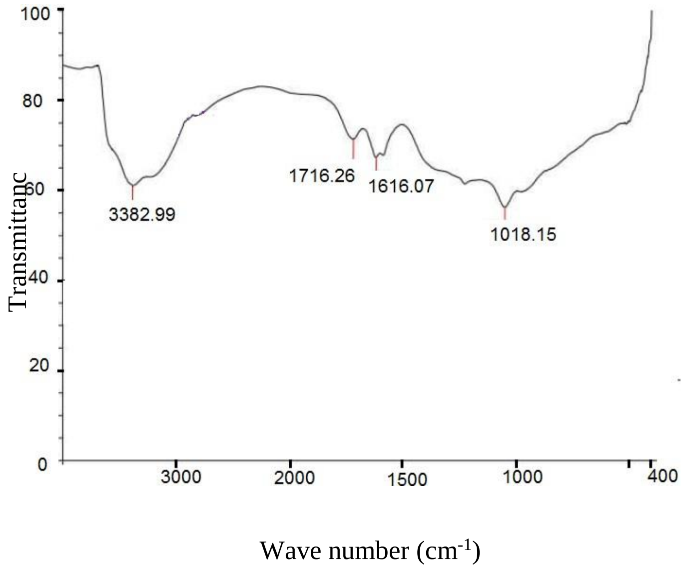


Figure 1. FTIR spectrum of reduced Graphene oxide.

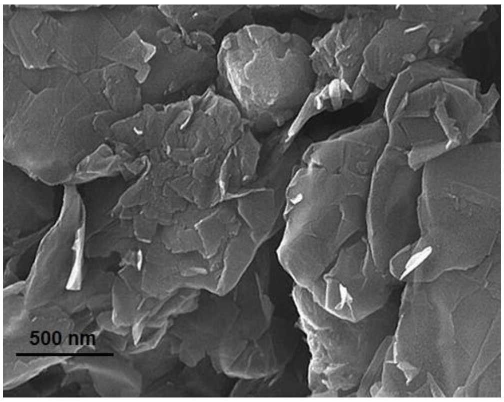


Figure 2. SEM image of reduced Graphene oxide nanoparticles.

The FTIR spectrum also shows that most of the functional groups have been removed in the reduction process. To prepare concrete with nanoparticles, these nanoparticles were added to water with weight percentages of 0.03%, 0.06% and 0.1% of cement weight, and were added to the composition of Table 2. In the preparation of concrete with Graphene oxide, the amount of Graphene oxide added was reduced from the amount of cement. Concrete prepared without nanoparticles and concrete prepared with the nanoparticles were placed in molds of 10 cm×10 cm×10 cm. To ensure the test results, seven of each sample were prepared. Three of them were used to measure 7-day compressive strength and averaging, three of them were used to measure 28-day compressive strength and averaging, and the last sample was used for measurements of corrosion tests. Inside the concrete of the sample intended for measuring corrosion tests, a cylindrical rebar with a diameter of 1 cm and a length of 10 cm, with the specifications given in Table 5, was placed in such a way that 5 cm of it is placed in the concrete and another 5 cm be out of concrete. After smoothing the surface, the samples were placed in the humidity chamber for 24 hours, and finally, the 7-day and 28-day compressive strength of the prepared samples was measured according to the standard. To measure the corrosion resistance of rebar inside the concrete, after 28 days of exposure of the sample with rebar in lime water, polarization and EIS tests were performed in 3.5% salt using potentiodynamic method with a potentiostat coupled to PC (Ivium, De Zaale 11, 5612 AJ Eindhoven, Netherlands). The sample was placed in the solution so that only 5 cm of the rebar was outside for connection. Figure 3 shows how to make connections for measuring corrosion tests. Polarization measurement was performed starting from -8 to 8 V with a rate of 100 mV/s and EIS test was performed in the frequency range of 100,000 Hz to 0.001 Hz and with a voltage range of 0.01 V. Before starting the measurement, the samples were placed in the solution for half an hour to stabilize the ocp potential.

Figure 3. Connections for measuring corrosion test.

Results and discussion

Strength results

Table 6 shows the results of 7-day and 28-day compressive strength of different samples. From the results, it is clear that by adding Graphene oxide nanoparticles with 0.03%, the 7-day and 28-day compressive strengths are improved.

Table 6. Compressive strength of different samples.

Compressive strength -28day (Kg/cm^2)	**Compressive strength -7day (Kg/cm^2)**	**Added Nano particle (%)**
410	295	0
429	313	0.03
434	338	0.06
406	292	0.1

It is also clear from the results that by increasing the amount of nanoparticles added up to 0.06%, the 7-day and 28-day compressive strengths increase more. The reason is the filling of concrete pores by Nano particles and improving the hydration reaction. In addition, nanoparticles added to concrete act as nuclei and strengthen the reaction of water and cement. The results show that adding more amounts decreases the strength of cement. The reason for the reduction of concrete strength by adding more amounts of Graphene oxide is attributed to the cumulative effects of Graphene oxide. Figures 4(a) and 4(b) show the change of compressive strength of 7 days and 28 days, respectively, by adding different percentages of nanoparticles. From the results, it is clear that, there is an optimal amount for adding reduced Graphene oxide nanoparticles to concrete, and adding more than this value causes the strength to decrease.

Electrical Impedance and corrosion analysis

Figure 5 shows the polarization test results of different samples. From the results, it is clear that the sample with 0.03% of nanoparticles has more corrosion potential and less corrosion current than the control sample.

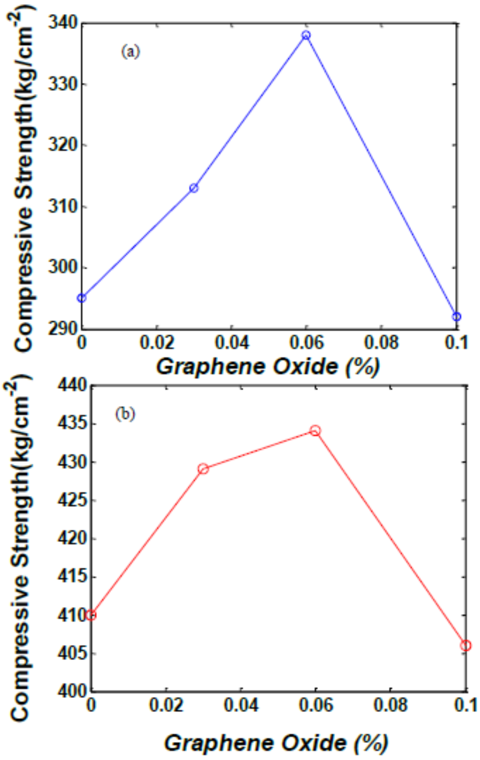


Figure 4. Change in compressive strength a) 7 days and b) 28 days, by adding different amounts of reduced Graphene oxide nanoparticles.

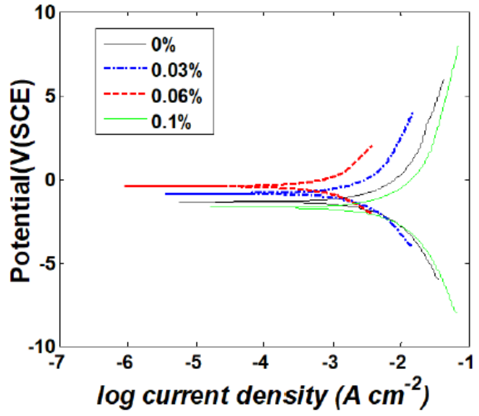


Figure 5. Polarization test of different samples.

Considering that the corrosion potential and corrosion current express the tendency and speed of corrosion, respectively, it can be concluded that by adding this amount of nanoparticles to concrete, the tendency and speed of corrosion will decrease. It is clear from the figure that the addition of nanoparticles with 0.06% of cement weight increases the corrosion resistance. Also, the results show that the addition of nanoparticles to the amount of 0.1%, causes a decrease in corrosion resistance, which is due to the tendency of Graphene oxide sheets to absorb and retain water. The results of corrosion current and corrosion potential of samples are given in Table 7, Figures 6 and 7 respectively.

Table 7. Polarization test results of different samples.

Added nano particle (%)	0	0.03	0.06	0.1
$V corrosion\ (V)$	-1.3	-0.8	-0.4	-1.6
$I corrosion\ (A)$	20×10^{-4}	9×10^{-4}	2×10^{-4}	30×10^{-4}

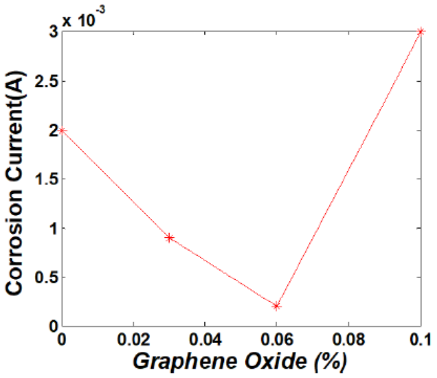


Figure 6. Corrosion current of different samples.

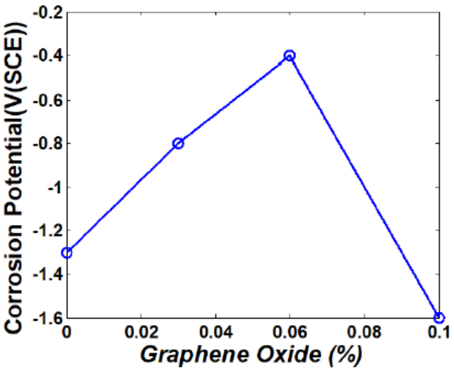


Figure 7. Corrosion potential of different samples.

It is clear from the figures that there is an optimum value for the amount of reduced Graphene oxide nanoparticles where the rate and tendency of electron loss decreases. This shows that at the optimal value, the corrosion current is minimal. The results of the table show that adding the optimal amount of reduced Graphene oxide, which is much less compared to the amount of cement (0.06 percent by weight of cement), reduces the corrosion current by 10 times. In order to investigate the reason for the decrease in corrosion current in the optimal amount of reduced graphene oxide added to concrete, the EIS test was performed and the impedance of different samples was investigated. The results of this analysis that shown in Figure 8, show that the electrical impedance of concrete with 0.03% of nanoparticles is higher than the impedance of the control sample. Also, the figure shows that the impedance of concrete is maximum for the sample with 0.06 % of nanoparticles.

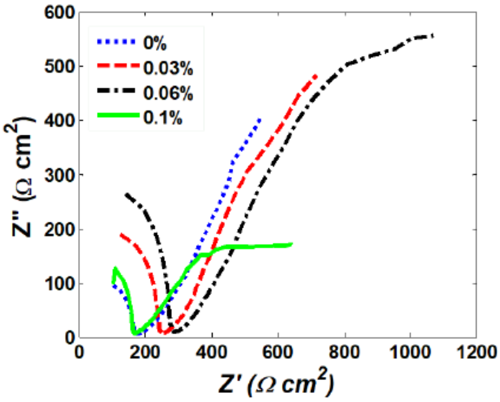


Figure 8. EIS test of different samples.

The high electrical impedance of this sample shows that the electron loss rate of this sample is slower than other samples. The results show that the sample with 0.1 % of nanoparticles has the lowest impedance and the lowest corrosion resistance. As mentioned above, the reason is attributed to the aggregation of Graphene sheets and the increase in capacitance. EIS Analyser software was used to further investigation of electrical impedance and also simulation of different samples with equivalent circuit. Figure 9 shows the equivalent circuit of different examples.

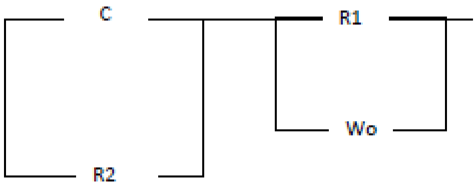
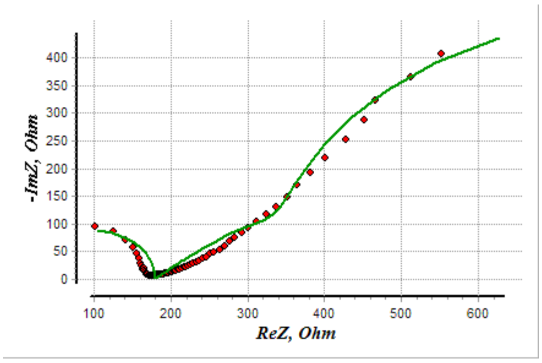


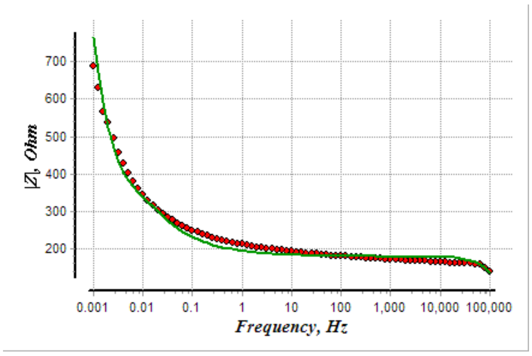
Figure 9. Equivalent circuit of different samples in salt solution.

In this circuit, C is the capacitance due to the presence of concrete between the rebar and the salt solution, R1 and R2 are electrical resistance at low frequencies and electrical resistance at high frequencies respectively, Wo represents the open Warburg impedance which accounts for the finite diffusion behaviour. The shape of the Wo in the Nyquist plot looks like a 45° line at higher frequencies, and transitions into a semi-circle shape at low frequencies. On the other words, at high frequencies, wo is almost exactly that of the Warburg impedance. The capacitor in the circuit is a set of series capacitors due to the penetration of the solution into the concrete. Fitting the experimental and simulation results to obtain the quantities of equivalent circuit elements for samples with different weight percentages of 0%, 0.03%, 0.06% and 0.1% of Graphene oxide are shown in Figures 10 to 13, respectively. The quantities obtained from this simulation are given in Table 8.

(a)



(b)



(c)

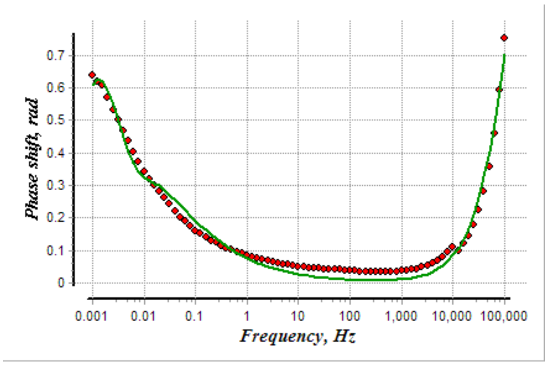


Figure 10. Fitting experimental data (dotted line) and simulation (solid line) of concrete sample with 0% Graphene oxide nanoparticles. A: Nyquist diagram, B: Bod diagram, C.

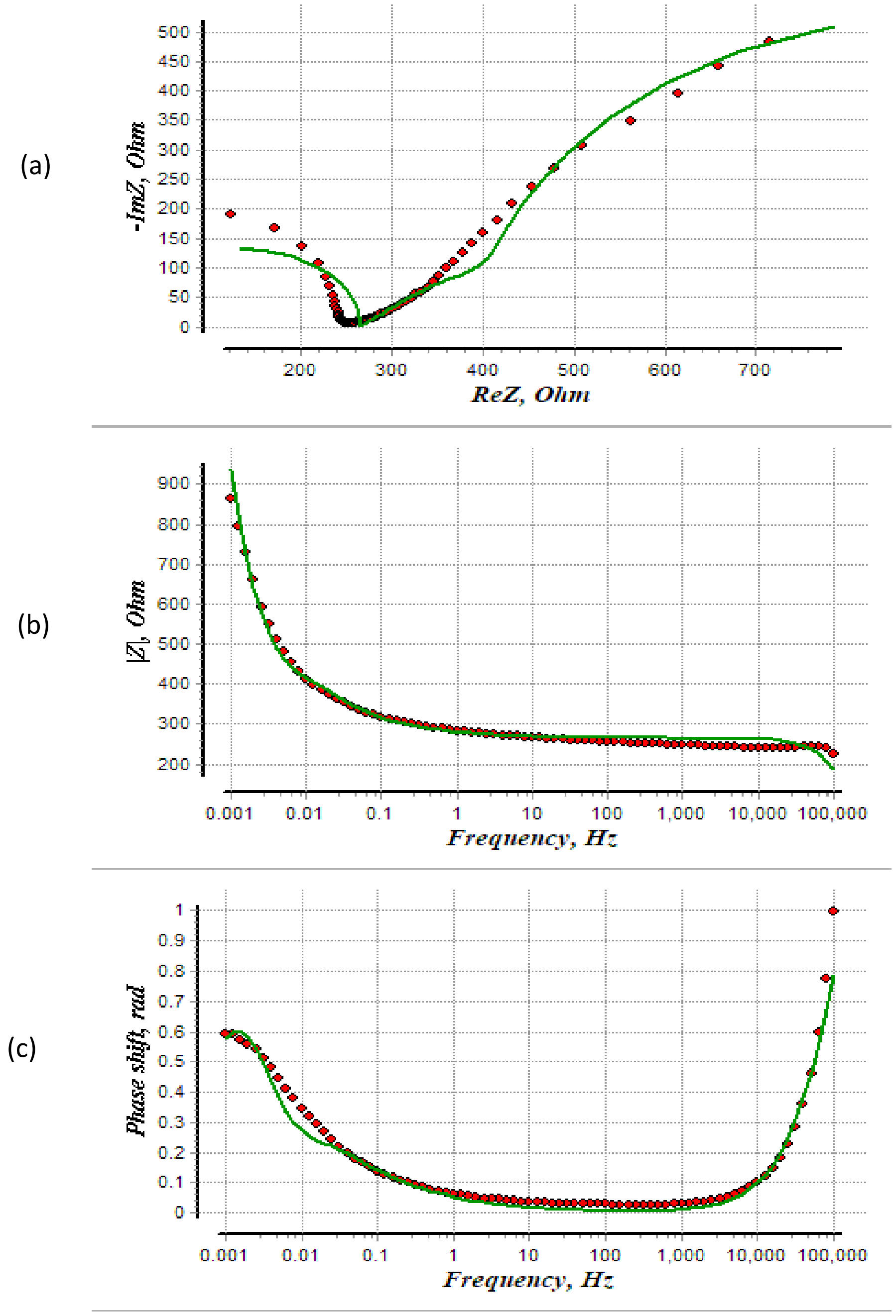


Figure 11. Fitting experimental data (dotted line) and simulation (solid line) of concrete sample with 0.03% Graphene oxide nanoparticles. A: Nyquist diagram, B: Bod diagram, C: Phase diagram.

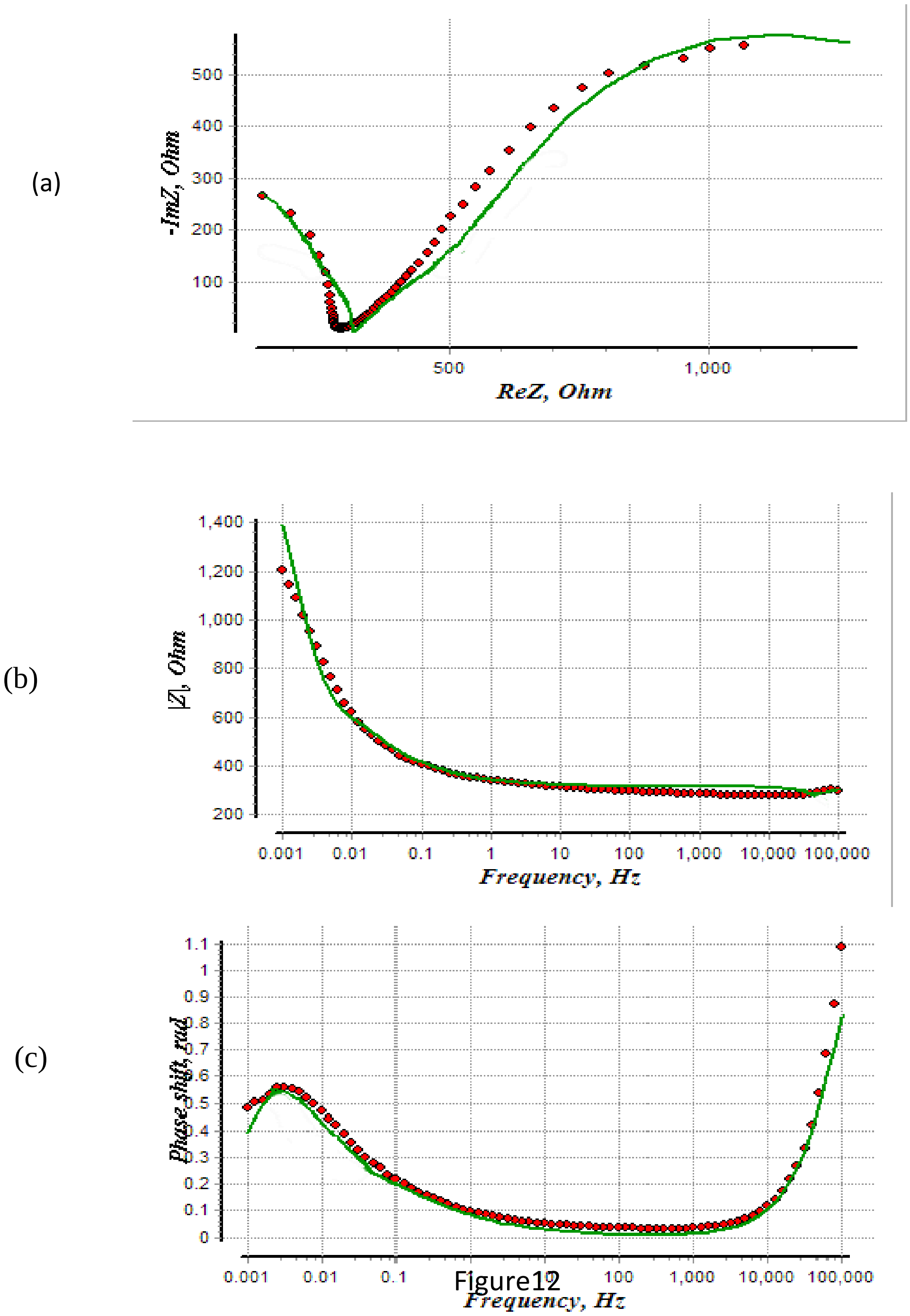


Figure12. Fitting experimental data (dotted line) and simulation (solid line) of concrete sample with 0.06% Graphene oxide nanoparticles. A: Nyquist diagram, B: Bod diagram, C: Phase diagram.

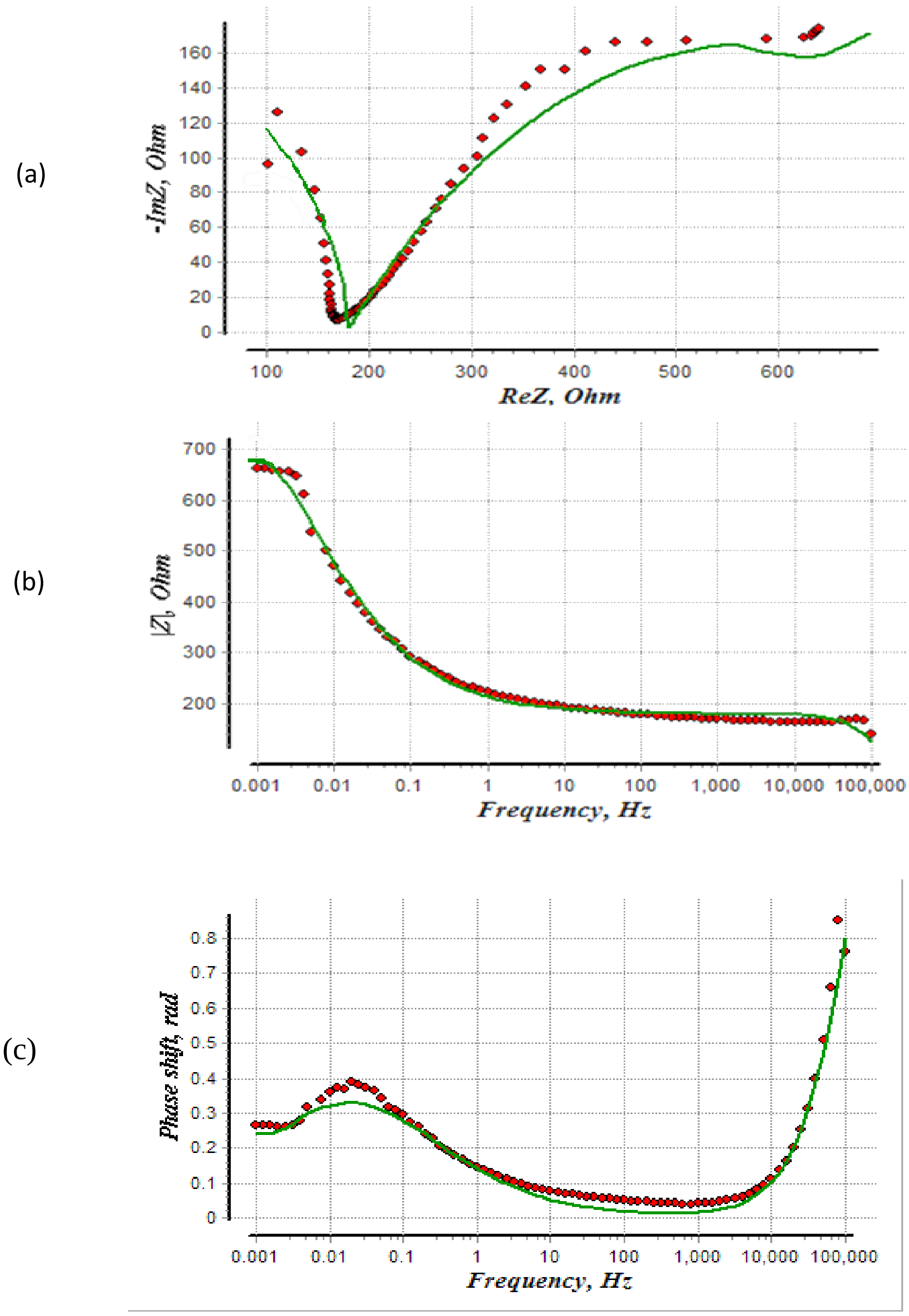


Figure 13. Fitting experimental data (dotted line) and simulation (solid line) of concrete sample with 0.1% Graphene oxide nanoparticles. A: Nyquist diagram, B: Bod diagram, C: Phase diagram.

Table 8. Quantities obtained from the equivalent circuit of different samples.

Nano particle (%)	٪0	٪0.03	٪0.06	٪0.1
C) F)	7.45×10^{-9}	6.07×10^{-9}	5.45×10^{-9}	1.02×10^{-8}
R1 (Ω)	1148	1211	3500	618

R2 (Ω)	179	265	290	178
Wo_R ($\Omega\ s^{-1/2}$)	41	45	90	35
Wo_c	10	12	50	8

In this table, Wo_R is Warberg's coefficient and Wo_c is equal to the following relation:

$$Woc = d/D^{0.5} \qquad (1)$$

Where, d is Nernst diffusion layer thickness and D is the diffusion coefficient. From the results of the table, it is clear that for the optimal amount of reduced graphene oxide added, the Warburg coefficient, and Warburg impedance is maximum. The results show that by adding Graphene oxide nanoparticles up to 0.06%, the capacitance decreases and the electrical resistance at high and low frequencies increases, and as a result, the impedance of concrete increases. The reason for the decrease in the capacitance is the increase in the number of series capacitors due to the uniform dispersion of nanoparticles in the concrete. The increase in the impedance of concrete indicates the increase in the corrosion resistance of the rebar inside it.

It is also found from the table that the amount of reduced Graphene oxide added more than the optimal amount causes an increase in capacitance, a decrease in electrical resistance, and as a result, a decrease in concrete impedance. Adding more than the optimal amount due to the cumulative property and non-uniform dispersion causes an increase in capacitance and a decrease in impedance. Also, due to the conductivity properties of reduced Graphene oxide nanoparticles (table 3), adding more than the optimal amount will cause a decrease in electrical resistance and therefore a decrease in electrical impedance. In other words, the reason for increasing the corrosion resistance of the rebar inside the concrete in the optimal amount of graphene oxide is the increase in the concrete impedance due to the decrease in the capacitance and the increase in the electrical resistance in all frequencies and the increase in the Open Warburg impedance.

Conclusion

In this work, nanoparticles were used to improve the properties of concrete. Because uniform dispersion is an important factor in determining the strength of concrete, and because graphene oxide nanoparticles have a high ability to disperse in water due to having oxygen functional groups, and because these nanoparticles have high reactivity and the absorption and retention of water reduce the performance of concrete, reduced graphene oxide nanoparticles with different weight percentages of cement weight were considered to improve concrete. The effect of different amounts of reduced graphene oxide on the strength of concrete and the corrosion resistance of the rebar inside it was investigated. In these investigations, it was found that although the addition of reduced graphene oxide nanoparticles increases the 7-day and 28-day compressive strengths, there is an optimal value that adding more than that optimal value weakens the strength of concrete. The cause was attributed to the cumulative effects of reduced graphene oxide nanoparticles. In the investigation of the corrosion resistance of the rebar in the concrete by measuring the polarization test, it was found that the addition of this Nano particle increases the corrosion resistance of the rebar in the concrete and adding it in an optimal amount reduces the corrosion current to a large extent. The EIS test measurement showed that the reason for the increase in the corrosion resistance of the rebar inside the concrete and the decrease in the corrosion current, is the increase in the impedance of the concrete. By simulating the equivalent circuit, the cause of the increase in impedance in the optimal amount of reduced graphene oxide was investigated and attributed to the decrease in capacitance, increase in electrical resistance, and increase in Warburg impedance.

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