Research Article

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Effect of graphene oxide on chloride penetration resistance of recycled concrete

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Abstract: Graphene oxide (GO) is a nanomaterial with ultra-high strength, good hydrophilicity, and dispersibility. To study the effect of GO on chloride penetration resistance of recycled concrete, the mechanism of action is investigated. The electric flux method is used to test the chloride penetration in recycled concrete specimens with 0, 0.03%, 0.06%, and 0.09% of GO. The volume change, microscopic pore distribution, and micro-structure morphology are characterized using laser rangefinder, and techniques such as X-ray tomography and scanning electron microscopy. The results show that the "coagulation nodule" effect of GO provides a growth basis for cement hydration, which results in a more uniform distribution of the hydrate microcrystals. It fills the micro-cracks of the recycled concrete, reduces the most probable aperture, increases the number of harmless small apertures, and enhances the volume stability of recycled concrete, thereby improving the chloride penetration resistance, which is important for improving the durability of concrete.

Keywords: Graphene oxide; chloride penetration resistance; recycled concrete; electric flux method

1 Introduction

The main methods by which the chloride ions penetrate the concrete include diffusion, capillary action, permeation, and electrochemical migration [1], which are affected by physical and chemical bonding, as well as adsorption between the concrete materials [2]. Covered with old mortar on the surface, the recycled aggregate has a complex and loose interface structure [3–5], and many micro-cracks are formed on the inside and the surface during the concrete crushing production process. Upon the erosion of the external environment, the micro-cracks and pores readily form a network distribution, which becomes a fast channel for the intrusion of the aggressive medium into the concrete [6, 7]. This reduces the chloride penetration resistance of recycled concrete, affecting its compactness and durability, rendering it inferior to the natural aggregate concrete [8–11]. This also hinders the use of recycled concrete in coastal, frozen, saline-alkali soil environments [12].

Nowadays, nanomaterials are widely used in medicine, biology, energy, food and new composite materials [13–16]. At the same time, nano materials can also be seen in concrete, agriculture, microelectronics, electricity, chemistry, environmental monitoring, information industry and other fields [17–19].

Graphene oxide (GO) is a new type of nanomaterial with ultra-high strength, good hydrophilicity, and dispersibility [20–26]. Many studies have described the incorporation of GO into cement-based materials to study the influence of this material. Babak [27] et al. found that an appropriate dosage of GO could improve the tensile strength of cement-based composites. Shenghua [28–30] et al. showed that the mechanical properties of cement mortar could be significantly improved, and the microstructure of cement hydration products could be effectively adjusted, thereby improving the strength and toughness of the cement-based materials. Devasena [31] et al. reported that the compressive strength, splitting tensile strength, and flexural strength of concrete were effectively improved with the addition of GO. Its analogues or products are also
widely used, such as biology, medicine, materials, chemistry, physics, energy, environment, etc. [32–34].

Prior studies have focused on the effects of GO on the mechanical properties of recycled concrete, but less amount of research has been conducted on the durability of its chloride penetration resistance. Permeability determines the infiltration rate of molecules and ions. It is correlated with water retention and waterproofing properties of concrete, and directly affects the frost resistance and erosion resistance of the concrete [35]. The overall model proposed by Metha [36] indicates that the penetration rate of aggressive media and the volume stability of concrete are the two main factors affecting the durability of concrete. Therefore, based on the effects of different dosages of GO on the chloride penetration resistance of recycled concrete, the effect of GO on the volume shrinkage of recycled concrete was also studied. X-ray tomography was employed for the detection of internal pore size distribution and the effect of GO on the pore structure of recycled concrete was observed by scanning electron microscopy (SEM) to explore the mechanism of action of GO in improving the chloride penetration resistance of recycled concrete.

2 Materials and methods

2.1 Test materials

P.O42.5 ordinary Portland cement produced by Shenyang Jidong Cement Co., Ltd. (Shenyang, China) was used. Medium sand with a particle size of 0.5–0.25 mm and a fineness modulus of 3.02 was used. The recycled coarse aggregate was a waste component of the structural laboratory of the Shenyang Jianzhu University. After a series of treatments such as manual crushing and screening, the recycled coarse aggregate concrete of good grade with particle sizes of 5–25 mm was obtained. The physical properties are shown in Table 1. Deionized water was used, and powder polycarboxylic water reducer with a water reduction rate of ~20–40% was used as the admixture. The water reducer content was 0.3% of the cement quantity. The mix proportion of the recycled concrete is shown in Table 2 and the mix ratios in the following tests are in accordance with this mix ratio. Industrial GO slurry was prepared by the modified Hummers method (with a mass fraction of 1%), and the specific parameters are shown in Table 3. All the test mainly determines the influence of GO dosage on the performance of recycled concrete, and the replacement rate of recycled aggregate is 100%. The ratios of GO amount to cement mass are 0, 0.03, 0.06, and 0.09%, and the test block group numbers are RC0, RC03, RC06, and RC09, respectively.

2.2 Chloride penetration resistance test

In the chloride penetration resistance test, ASTM C1202 DC direct current method [37] was employed. Each group of fresh concrete was made into three specimens with a size of $\Phi 100 \times 50$ mm (Table 2 lists the mixture ratio), and the standard curing was done for 28 d. Then, for vacuum saturation, the specimen was placed in a vacuum water saturation instrument, the absolute pressure of the vacuum vessel was reduced to ~1–5 kPa in 5 min, and the vacuum was maintained for 3 h. Distilled water was injected, and the specimen was immersed for 1 h before returning to the normal pressure. After soaking for 18 h, the specimen was retrieved and dried. The specimen was installed in the test tank as shown in Figure 1, and NaCl solution with a mass concentration of 3.0% and NaOH solution with a mass concentration of 0.3 mol/L were injected into the positive and negative electrodes, respectively, of the test tank on both sides of the specimen. A DC voltage of 60 V was applied, the current recording interval was set to 15 min, and energized for 6 h. An automatic data collection test device was employed, which could automatically calculate the electric flux without manual recording. Finally, the chloride penetration resistance of the concrete was determined based on the electric flux values.

| Water absorption (%) | Apparent density (kg/m³) | Crushing index (%) |
|----------------------|--------------------------|---------------------|
| 2.3                  | 2537.8                   | 16.3               |

Figure 1: The electric flux test device
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Table 2: Mix proportion of recycled concrete

| Water-cement ratio (w/c) | Sand content (%) | Water (kg/m³) | Cement (kg/m³) | Sand (kg/m³) | Recycled aggregate (kg/m³) |
|------------------------|-----------------|--------------|---------------|-------------|---------------------------|
| 0.42                   | 35.99           | 180          | 432           | 698.6       | 1242                      |

Table 3: Basic parameters of graphene oxide

| Thickness /nm | Single slice diameter /µm | Strippable rate /% | Oxygen content /% | Carbon content /% | Sulfur content /% |
|---------------|---------------------------|-------------------|-----------------|------------------|-----------------|
| 1             | 0.2~10.0                  | >95               | 51.6            | 42.7             | <2.1            |

2.3 Volume shrinkage test of recycled concrete

The volume change of the recycled concrete was measured using a German SICKD-79183 laser displacement sensor (Figure 2). The maximum output power of the instrument was 1 mW, pulse duration was 6.4 ms, and wavelength was 655 nm. The working principle is shown in Figure 3. The semiconductor laser is focused by the lens onto the measured object, and the reflected light is collected by the lens and projected onto the charge-coupled device array. The signal processor calculates the spot position on the array through the triangle function and then obtains the distance from the object. Loukili A [38] et al. studied the relationship between the self-shrinkage of concrete, hydration process, and internal relative humidity. The hydration of the concrete was very fast in the beginning, but shrank after four days, and mainly concentrated in the early stage. The shrinkage was 45% of the total volume on day 1, reached 95% on day 10, and stopped thereafter. Therefore, the observation period for volume shrinkage in the experiment was selected to be 14 d. The test was carried out under constant temperature and humidity conditions with a temperature of 20±2°C and a relative humidity of 60±5%. The non-contact shrinkage test was carried out with a mold. During the complete test, the position and direction of the specimen placed on the deformation tester remained unchanged. The specimen was a square test block with a size of 100 mm × 100 mm × 100 mm, and the test mixture ratio is shown in Table 2.

2.4 Pore size detection test

The cylindrical specimens prepared in this test had the dimensions of Ø50 mm × 55 mm, and the GO dosages were 0 and 0.06%, corresponding to the test group numbers of RC0 and RC06. The TomoScope HV Compact 225 X-ray tomography coordinate measuring instrument (Werth Messtechnik GmbH, Giessen, Germany) was used to detect the pore distribution inside the recycled concrete. The maximum X-ray tube voltage was 225 KV, maximum power was 320 W, detection accuracy was 4.5 µm + L/75 µm, maximum imaging pixelation was 1024 × 1024, and highest spatial resolution was 0.01 mm. Through the identification and analysis of the formed image, the pore diameter, volume, and percentage of the total volume of recycled concrete was measured.
2.5 SEM observation

After the maintenance of the shrinkage specimens for 28 d, small samples (including the interface area between new and old mortar) were obtained from RC0, RC03, RC06, and RC09. The surface of the sample was dried in a drying oven at 50°C for 24 h. After spraying gold on the surface, the micro-morphology and pore characteristics of the sample were observed by SEM (S-4800, Hitachi, Ltd., Tokyo, Japan). The acceleration voltage of the test equipment was 0.5–30 kV, and the amplification factor was 30–8000000 times.

3 Results and discussion

3.1 Analysis of results

3.1.1 Change regulation between the content of GO and chloride penetration resistance

The total flux of each specimen can be calculated using the following simplified formula:

\[ Q = 900(I_0 + 2I_{30} + 2I_{60} + \ldots + 2I_{300} + 2I_{330} + I_{360}) \]  
(1)

where \( Q \) is the electric flux passed (C), \( I_0 \) is the initial current (A), accurate to 0.001 A, and \( I_t \) is the current (A) at time \( t \), accurate to 0.001 A.

Considering the average value of the electric flux through the three specimens of the same group as the electric flux value of the specimen, the six-hour test results of the recycled concrete are shown in Table 4.

Table 4: Electric flux of recycled concrete in 6h

| Number   | Electric flux (C) | Number   | Electric flux (C) |
|----------|------------------|----------|------------------|
| RC0-1    | 2022.32          | RC06-1   | 1641.81          |
| RC0-2    | 1691.65          | RC06-2   | 1564.99          |
| RC0-3    | 1729.04          | RC06-3   | 1390.87          |
| RC03-1   | 1625.54          | RC09-1   | 1577.84          |
| RC03-2   | 1587.35          | RC09-2   | 1749.23          |
| RC03-3   | 1680.23          | RC09-3   | 1355.17          |

The chloride permeability coefficient is calculated according to the empirical formula for conversion between chloride ion flux and chloride ion diffusion coefficient proposed by Professor Feng Naiqian of Tsinghua University [39]:

\[ D = 2.57765 + 0.00429Q \]  
(2)

In the formula:

\( D \) – chloride ion diffusion coefficient in concrete, \((10^{-9} \text{cm}^2/\text{s})\)

\( Q \) – 6h total electric flux of concrete, (C)

With the incorporation of GO, the chloride ion permeability coefficient of each group of recycled concrete decreases in varying degrees. When the dosage is 0.03%, the chloride ion permeability coefficient decreases from 9.99150×10^{-9} cm^2/s to 9.57477×10^{-9} cm^2/s, i.e., a reduction of 4.17%; when the dosage is 0.06% and 0.09%, the permeability coefficient decreases by 8.40% and 7.19%, respectively. Thus, GO effectively improves the chloride penetration resistance of recycled concrete. Up to a specific limit of GO incorporation, this improvement is observed with an increase in the incorporation amount, but after this limit is reached, the strengthening effect is reduced.

3.1.2 Change regulation between the content of GO and shrinkage

Based on the changes in the spot position and test block distance, the change in the shrinkage of recycled concrete with different GO dosages can be calculated (Figure 4). In the early stage of aging, the shrinkage of the specimens in each group increases rapidly. After 1 d, the shrinkage values for RC0, RC03, RC06, and RC09 are 48.6%, 47.4%, 45.2%, and 46.3% of the total volume, and the shrinkage distances are 5.68×10^{-4}, 4.41×10^{-4}, 2.39×10^{-4}, and 4.64×10^{-4} mm, respectively. After 10 d, the shrinkage process stabilizes. After 14 d, the shrinkage rates for RC03, RC06, and RC09 are 0.0931%, 0.0529%, and 0.1003% respectively, which decrease by 20.36%, 54.75%, and 14.20%,
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Figure 4: Shrinkage change of recycled concrete with different contents of GO

respectively, compared to 0.1169% for RC0. Therefore, the incorporation of GO reduces the volume shrinkage during the hydration of recycled concrete. This best improvement is observed at an optimal dosage of 0.06%. When the dosage is 0.09%, the numerical value is significantly dispersed, hindering the improvement effect.

Based on the test results, the shrinkage rate of RC0 is more than 90% of the total volume at 3 d, whereas more than 90% of the total shrinkage is observed for RC03, RC06, and RC09 at 7 d, 6 d, and 8 d, respectively, which confirms the prophase shrinkage theory of concrete. Moreover, RC0 volume change rate curve is more advanced, and the early shrinkage is fast. The addition of GO causes a right shift of the shrinkage curve and retards the specimen shrinkage, indicating that GO has a delayed coagulation effect on the hardening of the recycled concrete.

3.1.3 Pore size determination

The non-destructive X-ray scanning of the recycled concrete was performed. After analyzing the gray value of the initial image by VG Studio MAX, and identifying and separating the aperture, the 3D image is shown in Figure 5. The sizes and distribution of the pores can be clearly observed. The scanning results were sorted and the micro-pores were counted (the stomata of more than 1 mm brought in during the mixing process were not counted). The total number of micro-pores of the RC0 and RC06 are 10598 and 10931, respectively. The pore size distribution of the recycled aggregate concrete with different GO contents is statistically analyzed. The pore size distribution percentage of each specimen is shown in Figure 6.

Figure 5: The 3D imaging of recycled concrete software after treatment

Figure 6: Percentage of pore size distribution of recycled concrete with different GO content
The most probable probability pore size (the most probable pore size) is determined and the calculated most probable pore sizes of RC0 and RC06 are 6490.371 µm and 4471.144 µm, respectively. The value of RC06 is approximately 31.14% lower than that of RC0. A comparison of the data for RC06 and RC0 shows that the peak value of RC06 shifts to the left, and the volume of the aperture within 400–500 micron is the largest. The probability of the aperture larger than 600 micron is small, while the peak value of RC0 appears in the 600–700 micron range. This indicates that the content of large aperture decreases while that of small aperture increases after GO is added. As shown in Figure 5, the voids appear independently without any connection. This shows that in the process of chloride ion penetration, the channels less than 100 microns in size exist inside the concrete to connect the holes, and the chloride ion erosion occurs. As the aperture size increases, the surface area increases, and the possibility of the existence of hole connectivity becomes higher. Therefore, higher the number of channels, more easily they are eroded.

### 3.1.4 Effect of GO on the microstructure of recycled concrete

The scanning electron microscopy analysis of the recycled concrete affords the microscopic topography as shown in Figure 7. The microscopic morphology of RC0 specimen is shown in Figure 7(a). The surface crystal structure is disordered and unsystematic with group stacking, and the interface has high porosity and loose structure. Figures 7(b), 7(c), and 7(d) show that the hydrated crystal has a regular shape and more pronounced aggregation effect, which makes the concrete micro-section dense. A comparison of Figures 7(b) and 7(c) shows that with an increase in GO
dosage, the "condensation nucleus" effect of GO provides a growth basis for cement hydration [40]. The optimized dosage of GO affords hydrated products that are clearly shaped and intertwined, thus refining and sealing the pore structure between the regenerated aggregate and cement mortar, and optimizing the aperture distribution such that the internal bonding of the structure is dense. When the GO dosage is 0.09% (Figure 7(d)), a significant difference is observed compared to other specimens and it shows a greater degree of decline compared to RC06. The hydration products aggregate in clusters, but do not form an overall structure and the gap between the aggregated crystals is large. There are two possible reasons for this phenomenon, besides the agglomeration and flocculation of nanomaterials in cement-based materials. First, because of the very high GO concentration and numerous auxiliary points of the hydrated crystal products, the growth of flaky bulk crystals is difficult. Second, GO exhibits strong water retention, which affects the complete hydration of the concrete when the dosage is very high.

3.2 Mechanism analysis

There are three main modes of chloride ion migration into the concrete, namely, diffusion, capillary action, and hydrostatic pressure, which may occur simultaneously. The high alkalinity of the solution in concrete holes maintains the steel bars in passivation state. When the chloride concentration in the vicinity reaches the critical value, the steel bars begin to rust, which leads to the deterioration of the performance of the reinforced concrete. The chloride ion penetration resistance of the recycled concrete is lower than that of ordinary concrete with the same mix ratio. First, inevitable cracks are present in the recycled coarse aggregate in the production process. These microcracks are very small and difficult to repair by the re-encapsulation of ordinary cement mortar. Second, similar to the ordinary concrete, after the completion of pouring, the cement paste with saturated water in recycled concrete loses a significant amount of water that is physically adsorbed in C-S-H when the environment gradually becomes less saturated (compared to the saturated humidity conditions), which leads to the formation of an internal cavity and volume shrinkage deformation of concrete. The excessive volume shrinkage inevitably leads to the cracking of concrete structure [41]. The direct or indirect connection of the old and new cracks and holes form a passage for chloride ion penetration, resulting in the reduction of concrete durability.

When the recycled concrete is mixed with GO nanomaterial exhibiting size and tunnel effects, its oxygen-containing groups can provide the growth basis for hydrated crystals, due to which the cement hydration reaction occurs preferentially on these groups and the microcracks in the recycled coarse aggregate can be repaired to a certain extent. The two-dimensional lamellar structure and large specific surface area of GO results in the formation of uniform hydrated crystals that cross each other. This further results in a dense and microcrystalline structure, which refines and closes the microporous structure of the recycled concrete and reduces the chloride ion penetration in the capillary channels. SEM analysis shows that the differences between the microstructures of the general recycled concrete paste and recycled concrete mixed with GO are not readily observed, but the differences in the pore phases are noticeable, particularly in the interfacial transition zone between the new and old mortar. The common recycled concrete specimens are more likely to have continuous holes. The radial length of the continuous holes is large, which affords a significantly larger most probable aperture for the ordinary recycled concrete compared to that of the recycled concrete mixed with GO in aperture testing. GO changes the pore structure of recycled concrete and fills the micro-cracks, which reduces the volume shrinkage and enhances the volume stability. In the shrinkage test, the maximum shrinkage rate of the ordinary recycled concrete is 0.117%, which exceeds 400–1000 micro-strains of the ordinary concrete. This shows that the volume stability of recycled concrete is lower than that of the ordinary concrete. However, the shrinkage rate of the recycled concrete specimens with GO content of 0.06% at 14 d is stable at 0.053%, that is, 500 micro-strains, which is 54.75% less than that of the ordinary recycled concrete. The improvement in the volume stability prevents the cracking of the concrete structure caused by concrete shrinkage and the formation of the connected pore. Boddy’s diffusion model based on Fick’s second law and Powers’ permeability test show that the size and connectivity of the pore in solid microstructure determine its permeability. By improving the pore structure of the concrete, GO enhances the volume stability of the concrete, reduces the content of pores with large sizes, and increases the chloride penetration resistance of recycled concrete.

4 Conclusions

The following conclusions are obtained.
1. The incorporation of GO improves the chloride penetration resistance of recycled concrete, and this effect increases with an increase in the dosage up to a certain limit. When the dosage is 0.06%, an optimal chloride penetration resistance is obtained. However, when the dosage is very high, the enhancement effect reduces.

2. GO reduces the volume shrinkage of recycled concrete and increases its volume stability. At an optimal dosage of 0.06%, the volume shrinkage is reduced by 54.75% compared to the commonly used recycled concrete.

3. The addition of GO has a slight effect on the porosity of recycled concrete, but the pore structure is refined and sealed, which reduces the content of pores with large sizes and increases the content of pores with small sizes.

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