

## Article

# Mechanical Properties, Impermeability, and Microstructure of Mortar Containing Recycled Fine Aggregates Modified by Graphene Oxide After High-Temperature Exposure

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**Abstract:** Graphene oxide (GO) has been verified to ameliorate the performance attenuation of cement-based materials exposed to high temperatures; however, its efficacy on cement-based materials containing recycled fine aggregates (RFAs) remains ambiguous. Therefore, this study assesses the macro/microperformance variations in mortar containing RFAs with diverse GO dosages (0~0.08%) after high-temperature exposure. The hydration products, pore structure, and microstructure of mortar containing RFAs and GO were analyzed using an X-ray diffractometer, mercury intrusion porosimetry, and scanning electron microscopy, respectively. The study finds that GO can reduce the mass loss rate of mortar exposed to high temperatures and enhance the residual compressive strength and flexural strength. The incorporation of 0.06% GO leads to a 10.4% increase in compressive strength and a 32.3% increase in the flexural strength of mortar containing 50% RFA after high-temperature exposure at 500 °C. Furthermore, the incorporation of GO refined the pore size and improved the densification of the interfacial transition zone (ITZ) of the mortar after high-temperature exposure. Taking into account the mechanical properties, water absorption, and microstructure of the mortar containing RFAs, a GO dosage of 0.04~0.06% could be recommended as the optimum engineering dosage.

**Keywords:** graphene oxide; recycled fine aggregate; high-temperature exposure; pore structure; microstructure



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## 1. Introduction

Due to the exceptional compressive strength, durability, high-temperature resistance, and cost advantage at bargain prices, cement-based materials have become one of the world's most widely used building materials [1–3]. Currently, the annual consumption of cement concrete worldwide has exceeded 11 billion tons, and its demand, especially in concrete buildings, is continuing to rise [4–7]. However, concrete buildings may be at risk of fire during their lifespan. When a building catches fire, the surface temperature of its concrete structures can reach high temperatures of several hundred degrees in a short time. This would not only result in the decomposition of hydration products but also would lead to cracks in the concrete due to the differences in thermal expansion coefficients among its constituted materials, thus decreasing its durability [8]. These changes will probably

weaken the mechanical properties (such as compressive strength) of concrete, potentially resulting in a decrease in the safety of concrete buildings [9–11].

With the rapid development of urbanization in China, large-scale renovation of old houses and new building developments have simultaneously led to two major challenges: an increase in abandoned concrete and a shortage of natural sand and gravel resources [12–14]. Crushing and processing waste concrete to form aggregates for the preparation of recycled aggregate concrete (RAC) is key to addressing this issue and can offer significant economic and environmental benefits [15–18]. Currently, research on recycled aggregate concrete focuses more on the use of recycled coarse aggregates (particle size  $> 4.75$  mm). Existing studies [19–21] indicate that recycled coarse aggregates can replace natural coarse aggregates within certain proportions to meet the requirements for practical engineering applications. However, the recycled fine aggregates (RFAs, particle size  $\leq 4.75$  mm) produced during the preparation of recycled coarse aggregates have not been fully utilized. RFAs possess more inherent flaws in comparison to natural fine aggregates and also have a higher concentration of aged mortar lumps and dust. Zeng et al. [22] revealed that the surface of RFAs is coated with porous and fractured aged mortar, resulting in a higher water absorption rate and lower density than natural fine aggregates. In contrast to the concrete utilizing natural river sand, concrete incorporating RFAs needs a greater amount of water to attain the same workability. Additionally, the incorporation of RFAs increases the number of capillary pores and gel pores within the cement matrix, detrimentally impacting its mechanical characteristics [23]. Salgado F et al. [24] suggest that the mechanical attributes of recycled fine aggregate concrete exhibit a tendency to diminish with an increase in the quantity of RFAs.

In recent years, recycled aggregate concrete has emerged as a research hotspot in the field of building materials, which mainly focuses on its basic performance under ordinary temperatures [25–30]. With the expansion of the application scope of recycled aggregate concrete, its exposure to the risk of fire also greatly increases, prompting a growing awareness of its resistance to high temperatures. RFAs contain a large amount of old mortar, and the difference in thermal expansion coefficient makes them more prone to damage in high-temperature environments. Unlike natural aggregate concrete, recycled aggregate concrete is a complex multi-phase material consisting of various interfacial transition zones (ITZs), so high-temperature environments will also cause significant damage to it [31–35].

An increasing number of nanomaterials have been employed to enhance the performance of cement-based materials [36]. Compared to traditional reinforcing materials, the key advantage of nanomaterials lies in their capability to ameliorate flaws in cement-based composites at the microscale. Graphene oxide (GO) stands out as a cutting-edge nanomaterial, boasting high surface activity, and its distinctive structure and exceptional properties have garnered widespread attention. Yu et al. [37] and Chu et al. [38] observed that the incorporation of graphene oxide not only accelerates cement hydration but also fosters the formation of superior crystalline C-S-H gel, significantly enhancing the mechanical properties of cement paste. Shilar et al. [39] indicated that, compared to the control group, the addition of 0.05 wt. % of GO led to a 38% and 23% increase in the flexural strength of concrete at 7 days and 28 days, respectively. Furthermore, it improved the pore diameter distribution within the cement paste, resulting in a denser structure. Moreover, Sujitha et al. [40] showed that the toughening effect of graphene oxide on cement materials primarily stems from the filling effect and the bridging effect on cracks. Jiang et al. [41] further observed that graphene oxide not only mitigates the decline in mechanical properties of ordinary-strength concrete following exposure to high temperatures but also effectively suppresses the phenomenon of thermal cracking in high-strength concrete. Qureshi et al. [42]

indicated that multi-layer graphene oxide aids in enhancing the high-temperature stability of C-S-H. The degradation of mechanical properties of cement-based materials after exposure to high-temperature environments is closely related to changes in cement hydration products and microstructure. However, few studies report on the relationship between the mechanical properties and microstructure of cement-based materials containing graphene oxide after high-temperature exposure, especially those with recycled fine aggregates.

Therefore, this study examined the impact of varying contents of graphene oxide ranging from 0.02% to 0.08% on the mechanical characteristics and water absorption of mortar incorporating recycled fine aggregates following exposure to temperatures of 100 °C, 300 °C, and 500 °C. To clarify the strengthening mechanism by GO, X-ray diffraction was employed to examine the phase alterations in mortar following exposure to high temperatures. Additionally, the pore structure and micromorphology of the mortar were assessed through mercury intrusion porosimetry and scanning electron microscopy. This study comprehensively evaluated the impact mechanism of graphene oxide on the performance of mortar containing recycled fine aggregates after being exposed to high temperatures, which could help promote the utilization rate of recycled fine aggregates in the construction field.

## 2. Materials and Methodology

### 2.1. Raw Materials

The materials utilized in this study include Type II 42.5 ordinary Portland cement (OPC), recycled fine aggregates (RFAs), natural fine aggregates (NFAs), graphene oxide (GO), and water. The chemical composition of OPC is detailed in Table 1. Quartz sand was used as the NFA, while RFAs were derived from crushed and sieved waste concrete from local house demolitions in Xi'an City. As illustrated in Figure 1, RFAs primarily comprise old mortar and stone materials. Table 2 provides an overview of the physical characteristics of NFAs and RFAs. The particle size distribution of raw materials is presented in Figure 2. GO used in this study was industrial grade, having a thickness of approximately 1.5 nm and an average diameter of 750 nm. Its morphology was observed using TEM (Spectra Ultra S/TEM, Thermo Fisher Scientific, Waltham, MA, USA) (Figure 3), revealing that GO was flaky and susceptible to curling.

**Table 1.** Chemical composition of the OPC (wt%).

|     | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | CaO   | Fe <sub>2</sub> O <sub>3</sub> | MgO  | SO <sub>3</sub> | Na <sub>2</sub> O | K <sub>2</sub> O | LOI  |
|-----|------------------|--------------------------------|-------|--------------------------------|------|-----------------|-------------------|------------------|------|
| OPC | 21.50            | 5.86                           | 59.82 | 2.78                           | 2.18 | 2.01            | 0.20              | 0.67             | 3.85 |

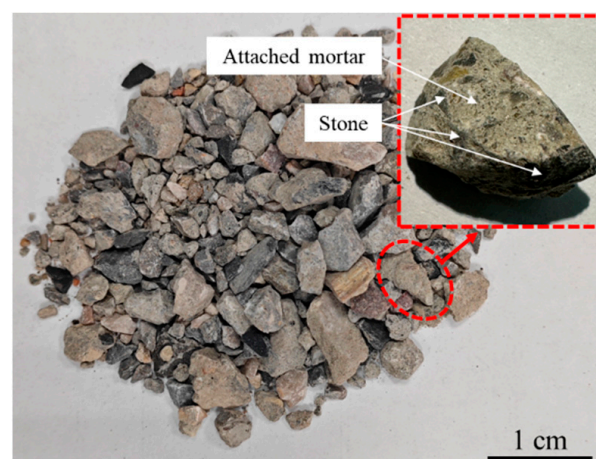
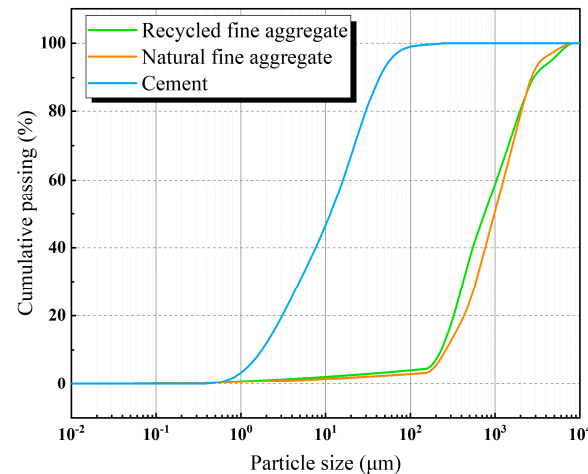
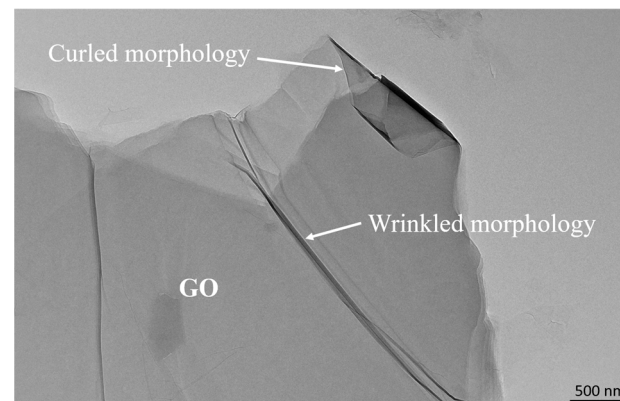


**Figure 1.** Appearance of regenerated fine aggregates.

**Table 2.** Physical properties of the NFAs and RFAs.

| Type | Apparent Density/kg·m <sup>3</sup> | Water Absorption/% | Fineness Modulus |
|------|------------------------------------|--------------------|------------------|
| NFA  | 2559                               | 1.10               | 2.62             |
| RFA  | 2433                               | 7.60               | 2.98             |

**Figure 2.** Particle size distribution of raw materials.**Figure 3.** Micromorphology of GO.

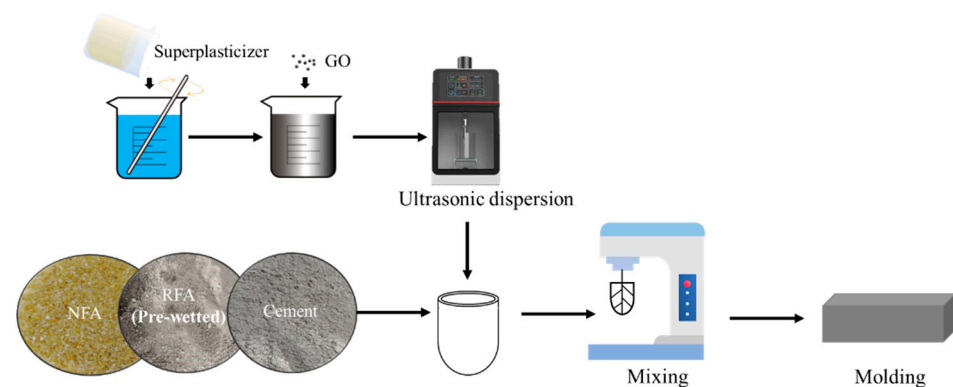
## 2.2. Material Design and Preparation Method

This experiment prepared recycled mortar in accordance with the Chinese standard GB/T17671-2021 [43]. Different replacement rates (30 wt % and 50 wt %, relative to the mass of NFAs of RFAs) were used to replace NFAs in the preparation of recycled mortar. The addition of GO was 0%, 0.02%, 0.04%, 0.06%, and 0.08% relative to the mass of OPC. A control group of mortar using only natural river sand (NM) was also included. The mix proportions are summarized in Table 3. Due to the moisture absorption of RFAs, they were pre-soaked in water for 1 day before mixing and then left to dry naturally until the surface reached a saturated dry state. The water-to-binder ratio for all groups was set at 0.5. Considering the hygroscopicity of RFAs during the mixing process, it is necessary to pre-wet RFAs with tap water during this process. Subsequently, RFAs were dried naturally until their surfaces reached a saturated dry state. Specimens were named RMx-y, where x and y represented the replacement rates of RFAs and GO, respectively. For example, RM50-2 indicated a specimen containing 50% RFAs and 0.02% GO.

**Table 3.** Mix proportion in this study (kg/m<sup>3</sup>).

| Type   | OPC | RFAs | NFAs | Water | GO   |
|--------|-----|------|------|-------|------|
| NM     | 450 | 0.0  | 1350 | 225   | 0.0  |
| RM30-0 | 450 | 405  | 945  | 225   | 0.0  |
| RM50-0 | 450 | 675  | 675  | 225   | 0.0  |
| RM50-2 | 450 | 675  | 675  | 225   | 0.09 |
| RM50-4 | 450 | 675  | 675  | 225   | 0.18 |
| RM50-6 | 450 | 675  | 675  | 225   | 0.27 |
| RM50-8 | 450 | 675  | 675  | 225   | 0.36 |

The procedure for preparing mortar specimens is illustrated in Figure 4. To achieve the effective dispersion of GO, the measured amount of GO was placed in a beaker filled with mixing water and dispersed using an ultrasonic disperser (LD-CP150, Leander, Jinan, China) operating at a frequency of 20 Hz for 15 min. Subsequently, the GO solution was mixed with cement and aggregates for 5 min, before being poured into molds with dimensions of 40 × 40 × 160 mm. After demolding, the specimens were cured in a controlled environment chamber at a temperature of 20 ± 1 °C and a humidity level exceeding 95% for 28 days.

**Figure 4.** Flow chart of specimen preparation.

### 2.3. Methodologies

#### 2.3.1. High-Temperature Heating Test

All the specimens were initially placed in a vacuum oven (RSD-100E, Royalstar, Jinan, China) at 50 °C for drying. Upon reaching a constant weight, the specimens were transferred to a high-temperature furnace (CSL-26-17, Test, Cangzhou, China). The designated temperatures were 20 °C, 100 °C, 300 °C, and 500 °C, with a heating rate of 5 °C/min. Once the designated temperature was reached, the specimens were maintained at a constant temperature for 2 h to ensure a uniform temperature distribution. Following completion of the heating process, the specimens were left to cool to ambient temperature within the furnace before being extracted. The visual changes and weight loss of the specimens at various temperatures were analyzed. In accordance with Section 2.2, the specimens subjected to different temperatures were labeled as T-RMx-y, where T denoted the temperature of exposure for the mortar.

#### 2.3.2. Mechanical Test

According to the Chinese standard GB/T17671-2021 [43], samples with a size of 160 mm × 40 mm × 40 mm were tested using a microcomputer-controlled electro-hydraulic servo universal testing machine (YAW-300D, Taylor, Cangzhou, China) with

loading rates of 0.06 kN/s and 2.4 kN/s until failure, and there were three parallel specimens of each test.

### 2.3.3. Water Absorption Test

The rate of water absorption can indicate the impermeability of the mortar. The specimens were dried to a constant weight in an oven at 50 °C, and then they underwent a water absorption test. The specimens were submerged in a water tank, and at intervals of 0.1 h, 0.5 h, 1 h, 2 h, 4 h, 8 h, 12 h, and 24 h, the specimens were taken out, the surface moisture was wiped off with a damp towel, then the specimens were weighed. The average value of the three specimens was taken as the final test result. The calculation formula is as follows:

$$W = \frac{m_t - m_0}{m_0} \times 100\%$$

where  $W$  is the water absorption of the specimen (%);  $m_0$  is the mass of the specimen after drying (g); and  $m_t$  is the mass (g) of the specimen after soaking for  $t$  hours.

### 2.3.4. X-Ray Diffraction Analysis (XRD)

The mortar specimen after the high-temperature exposure was crushed, and then approximately 10 g of the specimen from the interior was taken and placed in a vacuum oven at 50 °C for drying for 4 h. After the drying process, the specimen was ground and sieved through a 45 µm sieve. The mineral phase composition of the cement hydration products was analyzed using an X-ray diffractometer (D8 ADVANCE, Bruker AXS, Karlsruhe, Germany), using Cu K $\alpha$  radiation with a voltage of 40 kV and a current of 30 mA. The specimens were scanned gradually from 10° to 70° (2 $\theta$ ) at a rate of 5°/min (step size: 0.004°).

### 2.3.5. Pore Structure Analysis (MIP)

The mortar after high-temperature exposure was cut into cubic specimens with a size of 10 mm × 10 mm × 10 mm. Before testing, the specimens were dried in a vacuum oven at 50 °C for 4 h. The pore structure analysis of the bulk specimens was conducted using an automatic mercury porosimeter (AutoPoreIv9510, Micromeritics, Atlanta, GA, USA), with a contact angle of 140° and surface tension of 106.7 psi·µm.

### 2.3.6. Microstructure Analysis (SEM)

The specimen preparation method was identical to that used for MIP testing. Following drying, the observation surface was polished with sandpaper until smooth and subsequently sputter-coated with gold. The microstructure of the specimens was examined using a scanning electron microscope (JSM-7000F, JEOL, Tokyo, Japan).

## 3. Results and Discussion

### 3.1. Appearance Changes and Mass Loss

Figure 5 presents morphological changes in mortar after high-temperature exposure. With the temperature rise, the color of the mortar shifted gradually from gray to a grayish-white hue. Notably, at 300 °C, a pronounced color transformation took place, but no surface cracks manifested on the specimen. Subsequently, at 500 °C, evident microcracks emerged on the surface. The specimen produced a crisp sound when the specimen was struck.

Figure 6 reveals the mass loss rate of mortar after high-temperature exposure. With the rise in temperature, the specimens experienced a continuous increase in mass loss. At 100 °C, the weight reduction in all specimens was predominantly below 2%, primarily attributed to the evaporation of free water in the mortar, along with the decomposition of some C-S-H and Aft phases. Upon reaching 300 °C, the principal reason for the mass

decrease was the dehydration of C-S-H [44,45]. Upon further elevation to 500 °C, the mass loss was predominantly due to the decomposition of  $\text{Ca}(\text{OH})_2$ , with RM50-0 experiencing a weight loss of 18.0%. Following the addition of GO, the mass loss range of RM50 decreased; RM50-4 showed the lowest mass loss (15.5%), while RM50-8 was 17.2%. The mass loss of RM50 slightly increased with the increase in GO content. The results indicated that the inclusion of RFAs enhanced the thermal weight loss of the mortar, whereas the introduction of GO could successfully mitigate this effect. This was attributed to the fact that the incorporation of GO resulted in the generation of more extensively polymerized hydration products within the cement matrix, which exhibit reduced susceptibility to decomposition at elevated temperatures [46–49]. RFAs were composed of abundant cement fragments and mortar, leading to RM30 and RM50 containing a higher proportion of hydration binders. As a result, their weight loss following exposure to high temperatures was more pronounced.

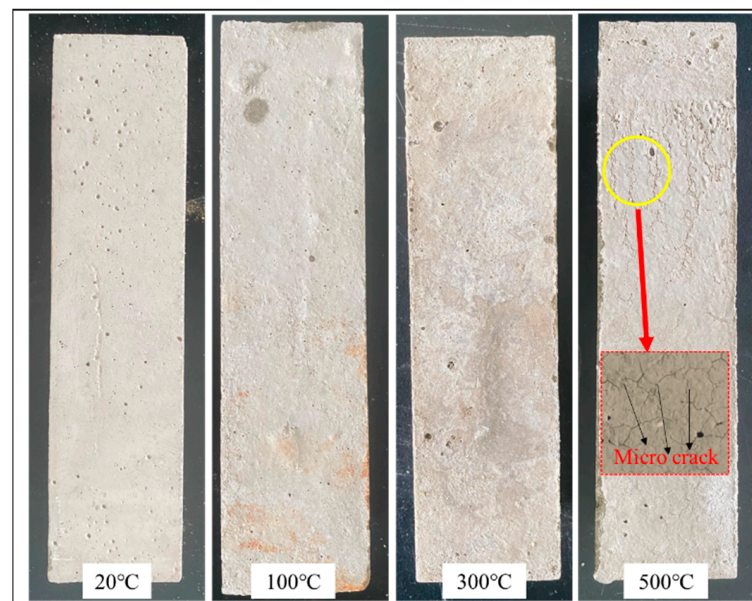


**Figure 5.** Morphological changes in mortar after high-temperature exposure.

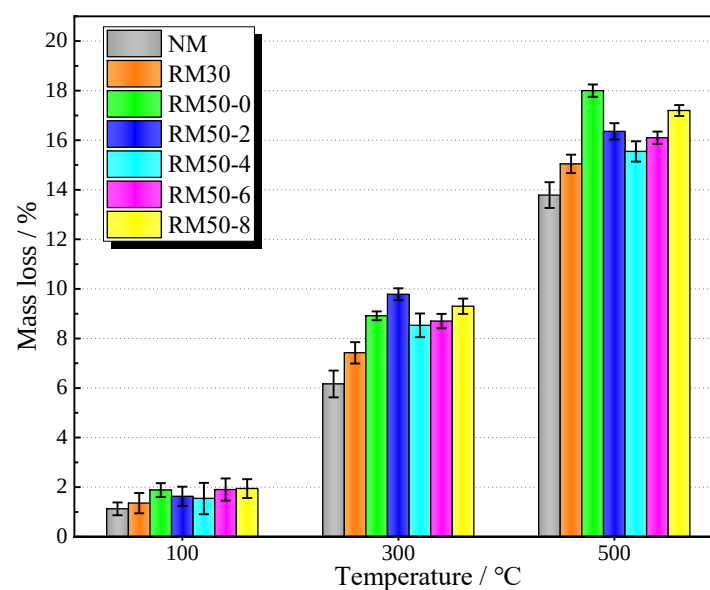


**Figure 6.** Mass loss rate of mortar after high-temperature exposure.

### 3.2. Compressive Strength and Residual Strength Ratio

Figure 7 displays the compressive strength of mortar after high-temperature exposure. The compressive strength of the mortar consistently decreases as the RFA content increases. Upon the addition of GO, the compressive strength of RM50 initially rose before declining. At 100 °C, the compressive strength of NM reaches 54.3 MPa. This could be attributed to the near-complete evaporation of free water in the mortar matrix, resulting in the volume contraction of the cement paste and a reduction in the spacing and proximity between hydration products, thereby minimizing defects such as cracks in the cement matrix. The compressive strengths of RM50-0, RM50-2, RM50-4, RM50-6, and RM50-8 are 40.2 MPa, 43.9 MPa, 45.5 MPa, 44.4 MPa, and 41.4 MPa, respectively. The incorporation of GO mitigated the extent of strength reduction in the mortar. As the temperature continues to rise, the compressive strength of all specimens decreases. At 500 °C, the compressive strengths of NM and RM50-0 are 31.2 MPa and 21.8 MPa, respectively. The compressive strength of RM50-0 is 30.1% lower than that of NM, while RM50-4 has a compressive strength of 28.5 MPa, representing a 30.7% increase over RM50-0. Further increasing the quantity of GO cannot significantly enhance the compressive strength. This is attributed to the high porosity of the aged mortar on the RFA surface and the formation of multiple interface transition zones in the cement matrix resulting from the introduction of RFAs. The thermal mismatch in deformation, resulting from the distinct thermal expansion coefficients of the aggregate and cement paste, caused the cracks between interfaces to widen, leading to a rapid decline in the compressive strength of the mortar. The filling effect of GO aided in reducing matrix porosity, while its nucleation effect could stimulate cement hydration, thereby enhancing the mechanical properties of the mortar [50,51]. However, high dosages of nanomaterials tend to aggregate and could impact the fluidity of the matrix, thereby weakening the comprehensive properties of the mortar.

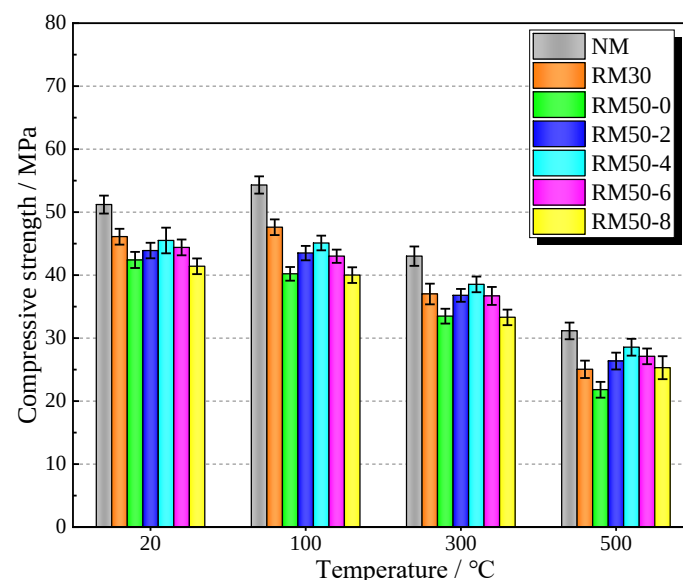


**Figure 7.** Compressive strength of mortar after high-temperature exposure.

Figure 8 displays the residual ratio of the compressive strength of mortar after high-temperature exposure. At all three temperatures, the residual compressive strength ratio of NM was the highest, while that of RM50-0 was the lowest. The incorporation of GO elevated the residual compressive strength ratio of the mortar. At 500 °C, the residual compressive strength ratio of RM50-0 was merely 51.4%, whereas the residual compressive strength ratios of RM50-2~8 could all be sustained above 60%. Remarkably, among all the mortars with added GO, the residual compressive strength ratio of RM50-4 was the highest. The

findings suggested that the inclusion of GO decelerated the strength degradation of mortar after high-temperature exposure. This was attributed to the fact that the introduction of GO increases the volume fraction of high-density C-S-H, thereby enhancing the thermal resistance of C-S-H [52,53]. Moreover, owing to the morphology of GO, it could function akin to fibers in the matrix, inhibiting microcracking, which further contributed to the enhancement of the residual strength of the mortar.

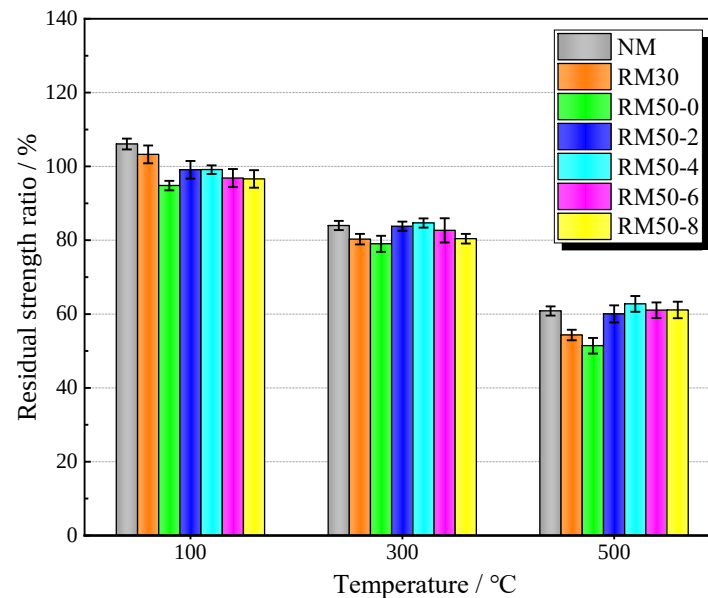


**Figure 8.** Residual ratio of compressive strength of mortar.

### 3.3. Flexural Strength and the Ratio of Flexural to Compressive Strengths

Figure 9 reflects the flexural strength of mortars after high-temperature exposure, as well as their ratios of flexural to compressive strengths. The flexural strength of the mortar decreased when RFAs partially replaced natural river sand. With the increase in GO content, it initially increased and then gradually decreased. At 500 °C, the flexural strengths of RM50-0, RM50-2, RM50-4, RM50-6, and RM50-8 were 3.1 MPa, 3.7 MPa, 4.0 MPa, 4.1 MPa, and 3.6 MPa, respectively. RM50-6 is 32.3% higher than RM50-0. Overall, the flexural strength exhibited the same trend as the compressive strength. From the results shown in Figure 9b, it could be observed that RFAs reduced the flexural-to-compressive strength ratio, while the addition of GO significantly improved this ratio. The flexural-to-compressive strength ratio of RM50-6 was the highest (0.1476) among all samples, indicating that GO could enhance the toughness of the mortar after high-temperature exposure. Flexural strength was closely related to the degree of cement hydration and the bond strength between aggregates and cement. As described in Section 3.2, due to the nucleation site effect, GO could regulate the C-S-H gel in the cement matrix. As the hydration process progressed, CH crystals were dispersed in the C-S-H gel, forming a structure centered around GO, making the ITZ between RFAs and the cement matrix more compact [54]. Simultaneously, due to its large specific surface area, GO exhibited a significant fiber bridging effect, reducing the formation of cracks in the matrix after high-temperature exposure, which was one of the reasons for the increase in flexural strength and the flexural-to-compressive strength ratio. In summary, GO could significantly enhance the flexural properties of the mortar, but higher doses did not necessarily result in better outcomes.

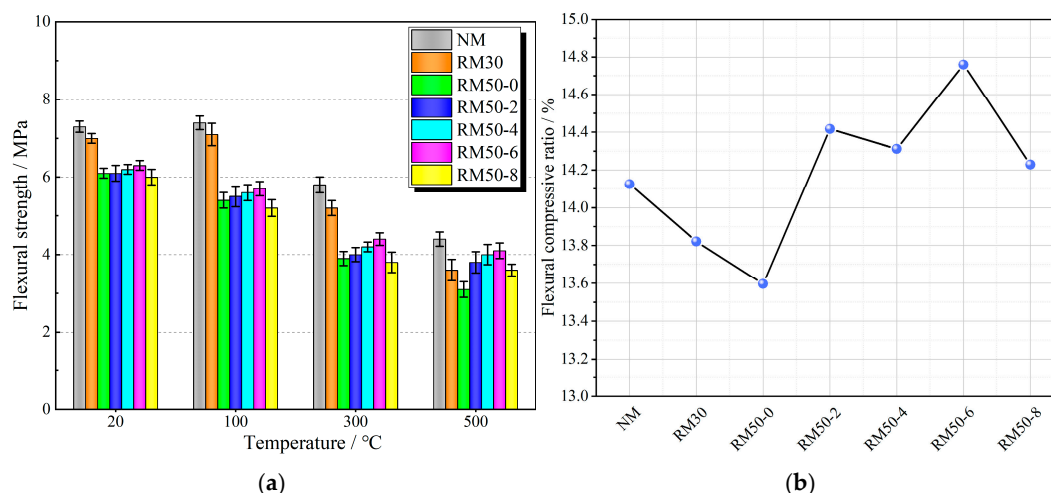


**Figure 9.** (a) Flexural strength and (b) flexural-to-compressive strength ratio after exposure at 500 °C.

### 3.4. Water Absorption Ratio

Figure 10 presents the water absorption rate of mortar after high-temperature exposure. It was evident that all mortars exhibited rapid water absorption within the initial 2 h, followed by a gradual rise in absorption rate. At 300 °C, RM50-0 displayed the highest water absorption rate. The incorporation of GO notably diminishes both the rate and amount of water absorption in the samples; after 24 h of absorption, the water absorption amount of 300 °C-RM50-4 was reduced by 8.0% in comparison to 300 °C-RM50-0. Upon elevating the exposure temperature to 500 °C, the water absorption rate further increases due to the decomposition of C-S-H, which enhances the porosity of capillaries in the cement matrix [55–57]. Interestingly, the 24 h water absorption amount of 500 °C-RM50-8 was marginally higher than that of 500 °C-RM50-0. This could be attributed to the filling effect of GO, which enhances the pore structure of the matrix and its nucleation effect that stimulates cement hydration, resulting in the formation of highly polymerized C-S-H and pore filling [58], thereby reducing the water absorption rate and absorption rate. However, the excessive addition of GO, owing to its high specific surface area and robust molecular interaction, tended to induce agglomeration. It introduces more voids within the cement matrix during the formation process, consequently increasing the water absorption rate of the mortar, resulting in an increase in the water absorption rate of the mortar.

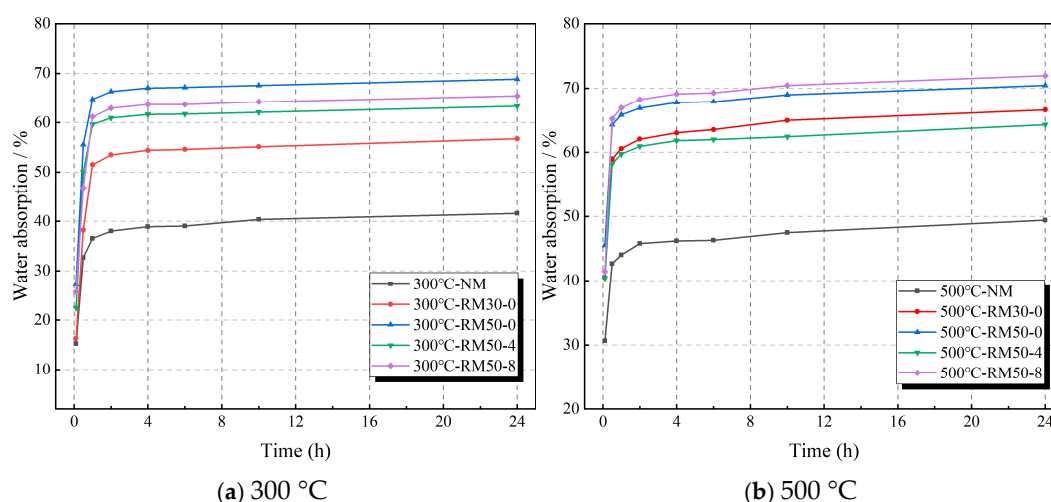


**Figure 10.** Water absorption of mortar exposed at different temperatures.

### 3.5. XRD

Figure 11 shows the XRD of mortar after high-temperature exposure. The addition of GO to the cement did not result in the formation of any new products. The primary mineral phases include CH, C-S-H, unhydrated  $C_2S$ , and  $C_3S$ . In the temperature range from 20 °C to 500 °C, the peak intensities of  $C_2S$  and  $C_3S$  exhibited a gradual decrease with rising temperature. Moreover, the decline in peak intensity was more pronounced when GO was incorporated at the same temperature, possibly due to the enhanced hydration of unhydrated cement particles under elevated temperatures. Furthermore, the addition of GO at the same exposure temperature led to an increase in the peak intensity of C-S-H, with the peak intensity showing a positive correlation with the quantity of GO added [59,60]. This enhancement in peak intensity could be attributed to the promotional impact of GO on cement hydration.

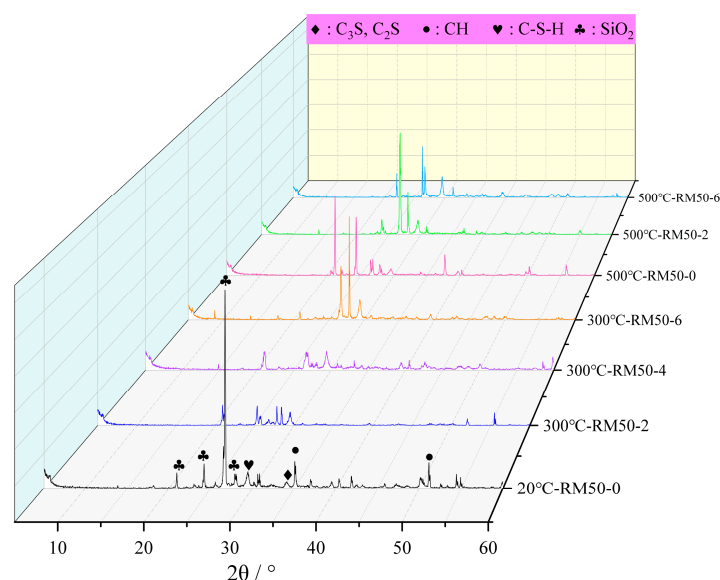


**Figure 11.** XRD of mortar after high-temperature exposure.

### 3.6. MIP

The pore structure and pore size parameters of the mortar are illustrated in Figure 12 and Table 4. According to the study conducted by Guo et al. [61], pores were categorized based on their size as follows: macropores (>1000 nm), capillary pores (100~1000 nm), and gel micropores (10~100 nm) [61]. Bai et al. [62] classified the impact of pore size on concrete performance as follows: benign pores (<20 nm), slightly detrimental pores (20~50 nm), detrimental pores (50~200 nm), and highly detrimental pores (>200 nm). As depicted in Figure 12a, as the heat exposure temperature rose, the peak pore size of 500 °C-RM50-0 shifted towards larger pores compared to 300 °C-RM50-0. Upon the addition of GO, the peak pore size of 500 °C-RM50-6 shifted to the left, indicating finer pores below 50 nm. Furthermore, the combination of Figure 12b and Table 4 revealed that an increase in heat exposure temperature led to a rise in the content of detrimental and highly detrimental pores in RM50-0. Conversely, the introduction of GO resulted in a decrease in the porosity of RM50-6 from 1.55% to 1.35%, accompanied by a notable reduction in macropores and capillary pores and an increase in benign pores. In conclusion, the appropriate incorporation of GO effectively refined the pore size of the mortar after high-temperature exposure, and decreased its porosity, thereby enhancing the mechanical properties and permeability resistance. Similar results were found in the study of Chen et al. [63].

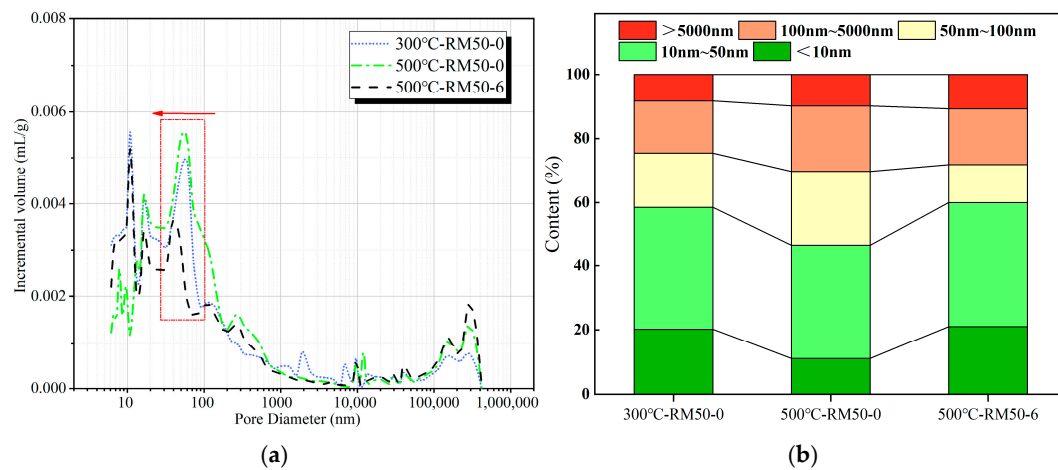


**Figure 12.** Pore structure of mortar: (a) different pore size distribution; (b) different pore size content.

**Table 4.** Pore diameter parameters of mortar.

| Type          | Median Pore Diameter (nm) | Average Pore Size (nm) | Porosity (%) |
|---------------|---------------------------|------------------------|--------------|
| 300 °C-RM50-0 | 30.6                      | 19.2                   | 0.97         |
| 500 °C-RM50-0 | 28.5                      | 18.9                   | 1.55         |
| 500 °C-RM50-6 | 28.7                      | 16.2                   | 1.35         |

### 3.7. SEM

Figure 13 displayed the SEM images of mortar. The interface transition zone (ITZ) is the thin layer of bonding between RFAs and cement paste. The ITZ is usually considered a weak link in cement-based materials, which may lead to the generation and growth of microcracks or become a channel for water infiltration. A denser ITZ correlates with improved mechanical properties in cement-based materials [64]. At a temperature of 300 °C, a few microcracks were observed in the cement paste, and the ITZ bonding was relatively compact. Upon reaching 500 °C, numerous cracks emerged in the cement paste, causing the hydration products to become loose and unevenly distributed. Furthermore, cracks and pores were evident in the ITZ. The incorporation of GO substantially enhanced the extent of cement hydration, resulting in the formation of more high-density C-S-H gel and other hydration products that filled the pores and cracks in the ITZ. Consequently, the hydration products were uniformly dispersed, leading to a decrease in cracks within the 500 °C-RM50-0 cement matrix. This suggested that the addition of GO could improve the density of the ITZ and decrease the quantity and width of cracks in the cement matrix, validating the previous assertion that the mechanical properties and resistance to permeability of the mortar were enhanced after the introduction of GO.

### 3.8. Mechanism Analysis

The level of mechanical property degradation in mortar containing RFAs after high-temperature exposure exceeds that of conventional mortar. The addition of GO could enhance the mechanical properties of mortar containing RFAs after high-temperature exposure. The uniformly dispersed GO facilitates cement hydration, aiding in the development and linkage of hydration products, resulting in the formation of heat-resistant C-S-H. Furthermore, as a nanomaterial, GO can fill voids in the mortar, reducing porosity and enhancing sample density. With its distinctive morphology, the 'bridging effect' of GO effectively restrained temperature-induced cracking in the mortar during high-temperature exposure. Consequently, the strength of RFA-containing mortar after exposure to high tem-

peratures was significantly improved by the inclusion of GO. The strengthening mechanism is outlined in Figure 14.

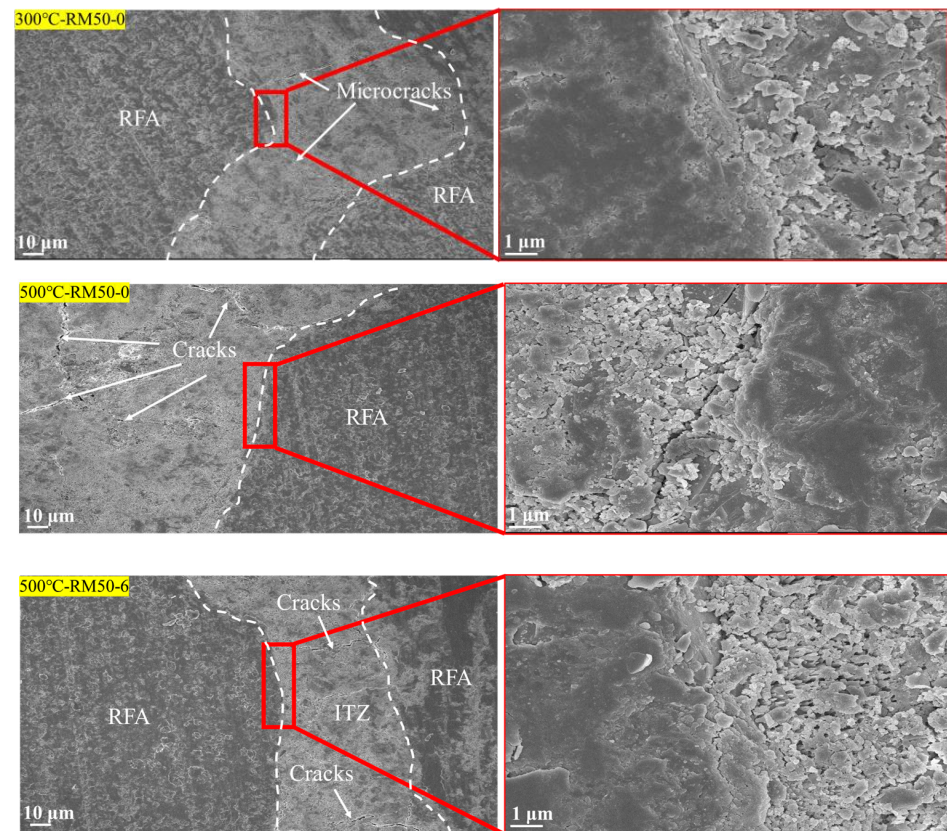


Figure 13. SEM images of mortar.

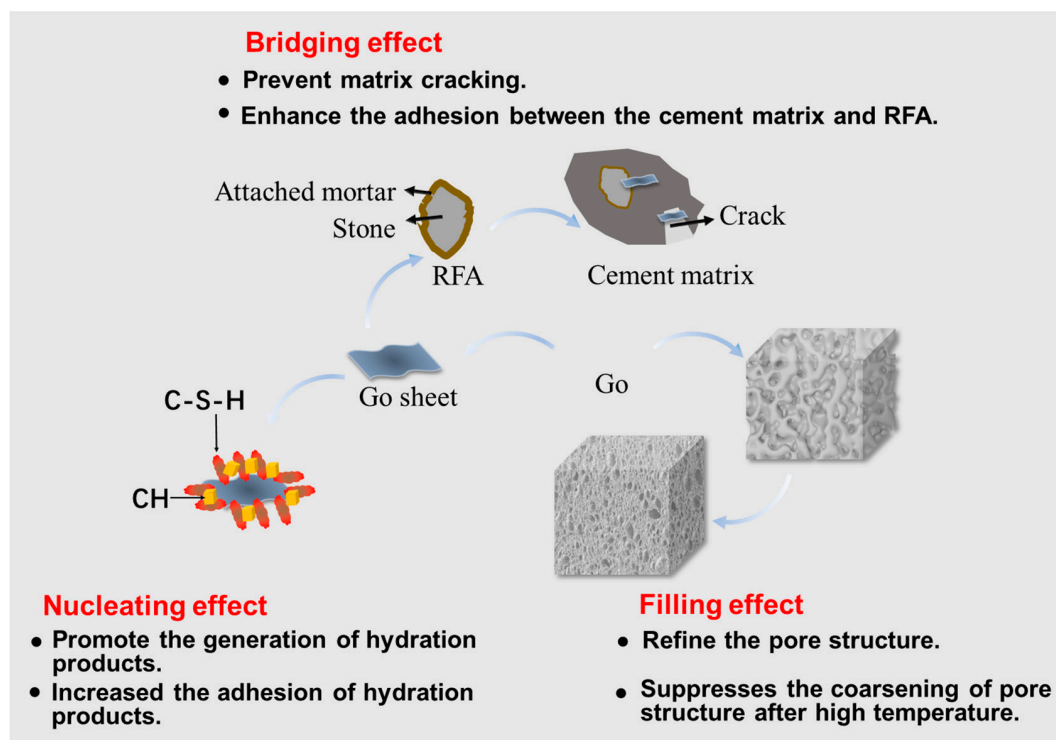


Figure 14. Strengthening mechanism of GO on mortar containing RFAs after high-temperature exposure.

## 4. Conclusions

In this work, the performance of mortar containing RFAs reinforced by GO after exposure to temperatures of 20 °C, 100 °C, 300 °C, and 500 °C was studied. The mass loss, mechanical properties, permeability resistance, hydration products, and pore structure of the mortar after high-temperature exposure were assessed. Furthermore, scanning electron microscopy was utilized to observe the ITZ and cement matrix of the mortar. The key findings were as follows:

1. After exposure to high temperatures, microcracks developed on the surface of the mortar. The thermal weight loss of the mortar significantly increased with the addition of RFAs. The incorporation of GO can mitigate the thermal mass loss of RM50.
2. The compressive strength and flexural strength of the mortar decreased progressively with the rise in RFA content. The compressive strength of 500 °C-RM50-4 increased by 30.7% compared to 500 °C-RM50-0. The residual compressive strength ratio of 500 °C-RM50-2~8 could all be maintained above 60%. The inclusion of GO can enhance the flexural-to-compressive strength ratio of the mortar after high-temperature exposure and improve the toughness of the mortar.
3. GO promoted the formation of C-S-H with high-thermal stability, thereby increasing the diffraction peak of C-S-H in mortar after high-temperature exposure. The porosity of 500 °C-RM50-6 decreased from 1.55% to 1.35%, and the pore size of the mortar was effectively refined.
4. The addition of GO resulted in the uniform distribution of hydration products and dense filling of the ITZ with hydration products, thereby reducing both the quantity and width of cracks in the cement matrix.

This study provides a simple method to enhance the high-temperature resistance of cement-based materials containing RFAs. However, this study focused on the changes in mechanical properties, and the durability performance of cement-based materials requires further research.

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