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Performance of concrete containing pristine graphene-treated recycled concrete aggregates

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ABSTRACT

Upcycling recycled coarse aggregates (RCAs) in concrete is a promising way to decrease the environmental effect of construction and demolition waste and improve concrete sustainability. Pretreatment of RCAs helps to enhance the quality of the resulting concrete and results in an increased level of confidence for material suppliers to systematically use RCAs to replace virgin aggregates for concrete production. This study demonstrates the utilization of pristine graphene for pretreating RCAs with the aim of effectively alleviating the loss of mechanical and durability performance of concretes when compared to virgin aggregate concretes. The RCAs were presoaked in water solution containing graphene concentration ranging from 0 to 0.5 %. Then, 50 % of natural coarse aggregates were replaced with the RCAs for concrete production. Different properties of concrete including slump, axial compression, splitting tension, water absorption and drying shrinkage were measured. In addition, microanalysis of the aggregates was performed by x-ray diffraction analysis (XRD) and scanning electron microscopy (SEM). Based on results, treating the RCAs with 0.2 wt% pristine graphene results in increased workability (13 %), compressive strength (21 %) and tensile strength (12 %) and decreased water absorption (22 %) and drying shrinkage (20 %) of the RCA concrete. Increasing the concentration of the graphene beyond 0.2 wt % is found to decrease the workability and strength properties and increase the water absorption and drying shrinkage. This is attributed to the agglomeration of the graphene at high concentrations, leading to non-uniform filling effects and ineffective microcrack and void filling. It is also found that 0.2 wt% is the optimum pristine graphene concentration, which can lead to an RCA concrete with similar mechanical and durability properties to the conventional concrete.

1. Introduction

There is an urgent need for addressing environmental impacts of solid wastes due to their contribution to climate change (Pierrehumbert, 2019). One of the most important sources of solid wastes in Australia and elsewhere globally is construction and demolition (C&D) waste. About 2.59 billion tons of C&D waste will be generated in 2030 globally, which is expected to rise to 3.4 billion tons by 2050 (Kaza et al., 2018). Meanwhile, the production of concrete requires the extraction of a high amount of natural resources (32–50 billion tons annually), which is causing serious ecological problems (Bendixen et al., 2019). With an annual global consumption of 25 billion tons (Rashid et al., 2020), the conventional concrete as the most extensively used material in construction is responsible for the utilization of 30 % of non-renewable natural resources, 8 % of atmospheric greenhouse gas emissions including CO_2 and 50 % of landfill waste (Brebbia and Sendra, 2017). Recycling of concrete aggregates obtained from C&D waste towards concrete production is a vital strategy to tackle the environmental impact of both C&D waste and concrete production (Xing et al., 2023). However, recycled coarse aggregates (RCAs) are still disposed of in landfills and not widely used to produce concrete because of their inherent poor properties compared to virgin aggregates. The use of RCAs as partial replacement of virgin aggregates comes with both plant investment and additional risk which concrete suppliers tend to avoid (Sofi et al., 2012; Wijayasundara et al., 2016). Improving the quality of the RCAs by an advanced and cost-effective treatment is an important step to overcome the drawbacks of their use in construction industry.

Several studies have been done on using RCAs in concrete (e.g., (Ali

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et al., 2021; Gholampour and Ozbakkaloglu, 2018; Kurda et al., 2019; Ouyang et al., 2023; Ozbakkaloglu et al., 2018; Tang et al., 2020; Wu et al., 2020)). It is generally agreed that properties of RCA-based concrete such as workability, mechanical and durability are weaker than those of conventional concrete. This is related to the larger porosity of RCAs when compared to gravels because of the porous residual old mortar matrix of the RCAs (Wang et al., 2021). The old mortar matrix also creates a weak interface between the RCA and the paste of the new concrete (Zhan et al., 2020). As a result, different chemical and mechanical methods have been proposed for improving RCA properties (Ouyang et al., 2023). For example, Munir et al. (2020) studied axial compressive behavior of confined concretes containing accelerated carbonation-treated RCAs, and found that the treated RCA concretes had a higher peak stress and modulus of elasticity and lower peak and ultimate strains compared to untreated RCA concrete at a given confinement level. Munir et al. (2021) used acetic acid immersed and mechanically rubbed RCAs in concrete, and found that the ultimate and peak strains of the RCA concrete under axial compression were higher than those of conventional concrete. Kazmi et al.(2021) studied the effect of different RCA treatment techniques, including accelerated carbonation, soaking in lime with accelerated carbonation, soaking in acetic acid, soaking in acetic acid with rubbing, and soaking in acetic acid with accelerated carbonation, on the thermal performance of RCA concrete. They reported that the concrete with RCA treated through soaking in lime with accelerated carbonation technique developed a higher compressive strength and thermal conductivity and a lower water absorption than other treated RCA concretes. Kazmi et al. (2023) and Munir et al. (2022) developed models to predict elastic modulus and compressive strength of treated RCA concretes, respectively. Based on the literature review, cleaning of the old mortar matrix of RCAs by heat, acid presoaking, ultrasonic or mechanical treatment is energy intensive and costly with a high carbon footprint (Wang et al., 2020). In other methods such as carbonation of RCAs (Lu et al., 2019; Munir et al., 2020; Zhan et al., 2020), and coating of RCAs with sulfoaluminate cement and mineral additives (Zhang et al., 2018; Zhang et al., 2021), there have been concerns related to the application of the methods at industrial scale since they become resource intensive or increase the risk of alkali-silica reaction, and the improvements are not enough compared to the conventional concrete (Wang et al., 2020).

The latest attempts for enhancing the RCA properties have been using nanotechnology through presoaking or spraying RCAs with nanomaterial solutions. Owing to the small size and high reactivity of the nanomaterials, they act as a pore-filling effect in RCAs and take part in enhancing the reaction of cement hydration of interfacial transition zone (ITZ) of fresh cementitious paste. Rapid development of nanotechnology has been accompanied by the optimization of the treatment processes, the reduction of the cost of nanomaterials and nano/micro-scale characterization (Zhan et al., 2022). Although relatively new, they have found their way into the concrete industry. For example, Zhang et al. (2015) used nanosilica and cement slurry to presoak RCAs, and found that the treatment improved the new ITZ and did not have any significant effects on the old ITZ. Shaikh et al. (2018) reported that soaking RCAs in nanosilica had a better pore refinement effect (about 45 %) than the direct mixing of the nanosilica in the RCA concrete. Li et al. (2021b) studied efficiency of various treatment procedures of RCAs, including carbonation and nanosilica spraying, and reported that using nanosilica spraying method led to a larger improvement of mechanical performance of the concrete than carbonation method. Chen and Jiao (2022) presoaked RCAs with nanosilica at different concentrations and reported that 2 wt% was the optimum concentration of the nanosilica to increase the compressive strength of the concrete by 20 %. Feng et al. (2023) used nano limestone for presoaking RCAs and found that it improved the ITZ of the RCAs based on the cementation and filling of the ITZ. However, existing studies have shown that for natural gravel replacements of 50 % and more where RCAs are treated with the nanomaterials, the RCA-based concrete still exhibits inferior mechanical and durability properties in comparison to conventional concrete, or alternatively, a larger dosage of nanomaterials is needed to achieve similar properties to the conventional concrete. This behavior was because of the low specific surface area of the used nanomaterials.

As a 2D carbon material, graphene has several derivatives, e.g., pristine graphene, graphene oxide and reduced graphene oxide (Gholampour and Ozbakkaloglu, 2022). These different types of graphene were explored as additives in construction materials, showing significant improvements (30–60 %) on their mechanical properties (e.g., (Gholampour et al., 2017; Ho et al., 2021; Li et al., 2022; Liu et al., 2022)) using very minor dosages ($<$ 0.1 %). Excellent performance of graphene-enhanced concrete can be explained by the strong reaction together with existing functional groups and large surface area of graphene than other nanomaterials (Yao et al., 2022). The production of pristine graphene involves directly separating graphite, resulting in a graphene sample with minimal functional groups, fewer defects, enhanced crystallinity, and superior mechanical properties in comparison to graphene oxide and reduced graphene oxide. However, they are less dispersible in water than graphene oxide and reduced graphene oxide (Qureshi and Panesar, 2020). The dispersibility issue of the pristine graphene can be resolved by using superplasticizer and ultrasonication (Wang et al., 2016). Therefore, pristine graphene can be a promising material to improve the properties of RCAs.

This paper presents the outcomes of the study looking at improving properties of RCAs by using pristine graphene. This study sought to establish the optimum concentration of pristine graphene to mitigate the loss of mechanical and durability characteristics of RCA-based concrete compared to conventional concrete. Different tests on RCAs, including specific gravity and water absorption, were conducted. RCA-based concrete properties including workability, compressive and splitting tensile strengths, drying shrinkage and water absorption were also assessed. X-ray diffraction analysis (XRD) and scanning electron microscopy (SEM) were also undertaken for evaluating microstructural influences of the addition of graphene on concrete. A summary of the experimental plan is presented initially, which is followed by the results and discussion.

2. Research significance

Development of concretes containing RCA at virgin aggregate replacement levels of more than 30 % with similar mechanical and durability properties to conventional concrete has significant benefits and meets principles of circular economy. Using RCA can lead to a significant reduction in greenhouse gas emissions, with a potential decrease of 65 %. Additionally, it has the capacity to reduce non-renewable energy consumption by 58 % and reduce C&D waste by 75 % (EPA, 2021).

This study presents the optimum concentration of pristine graphene that significantly improves RCA properties which then translates into improved new concrete properties. It should be noted that 1–2 MPa improvement in strength properties can be significant particularly when considering early age properties of concrete (Sofi et al., 2019). The developed concrete will provide major environmental, economic and health benefits by: i) adding value to RCA as material for use towards concrete production, ii) reducing the level of waste intended for landfills which has an ongoing environmental and economic costs, iii) helping conserve the ecology by reducing the extraction of natural aggregates, which are needed at large quantity, and iv) reducing concrete life-cycle costs, owing to improved RCA durability, extended service life and reduced repair and maintenance costs. The proposed approach will contribute significantly toward resource efficiency in the construction industry and the advancement of the next-generation construction materials that have a low environmental footprint.

3. Experimental plan

3.1. Materials

Ordinary Portland cement as the binder was supplied from Adbri Ltd in South Australia, Australia. Natural river sand (NS) and coarse aggregate (NCA) were supplied from Fleurieu Peninsula, Australia. The RCA was supplied from ResourceCo in South Australia, Australia. Table 1 and Fig. 1 present the physical characteristics and sieving analysis of aggregates, respectively. To reduce the effect of different maximum particle sizes of NCA and RCA and water absorption of RCAs on the test results, all coarse aggregates were utilized in a saturated surface dry (SSD) condition. Trial mixes with varying proportions of RCA, adjustments to the mix design, and thorough testing of fresh and hardened concrete properties were also conducted to reduce the maximum particle size effect of the aggregates.

Pristine graphene was produced in the Sustainable Construction Lab at Flinders University by liquid-phase high-shear exfoliation (Zhang et al., 2018) of graphene powder supplied from First Graphene Ltd. The graphene had a tap density below 0.1 g/cm^3, a specific surface area ranging from 180 to 280 m^2/g and a particle size (D50) below 10 μm. Superplasticizer MasterGlenium SKY 8700 was used to improve the dispersion of the pristine graphene in water (Ho et al., 2020). Fig. 2 shows the SEM and XRD spectra of the pristine graphene. Based on the SEM image, the pristine graphene had wrinkled structure with irregular shapes. According to the XRD spectra, the primary peak of the graphene occurred at 26.64° with d-spacing of 0.33 nm between its layers, contributing to few layers graphene.

3.2. Treatment of RCAs by pristine graphene

The RCAs were reinforced with pristine graphene to reduce their permeability and strengthen their old mortar-aggregate ITZ. Following the presoaking techniques as in references (Li et al., 2021b; Zhang et al., 2015), initially the RCAs underwent thorough drying in a laboratory oven at 105 °C for 24 h, followed by natural cooling at room temperature. Thereafter, the RCAs were soaked in the graphene solutions for 24 h, where different concentrations of 0.1, 0.2, 0.3 and 0.5 wt% (by aggregate) were considered for the graphene solutions based on trial studies. The RCA-modified materials were then subjected to drying in an oven set at 105 °C for a duration of 12 h. Fig. 3 shows the RCAs soaked in the graphene solutions. Fig. 4 shows the typical images of NCA, untreated RCA and treated RCA, and Fig. 5 shows the typical SEM image of the surface of a pristine graphene-treated RCA.

The specific gravity and water absorption of the untreated and treated RCAs were assessed in accordance with AS-1141.6.1 (2000). Table 2 presents the specific gravity and water absorption values of the RCAs before and after treatments. It can be observed that the specific gravity of the RCAs increased with a higher graphene dosage. In addition, an increased graphene dosage of up to 0.3 % led to a 27 % reduction of the RCAs' water absorption. However, further increase of the graphene concentration to 0.5 % increased the RCAs' water absorption by 3 %, which might be explained by a non-uniformity

Table 1
Properties of aggregates.

Aggregate type	Maximum size (mm)	Specific gravity (in SSD)	Water absorption (%)	Fineness modulus
Natural sand	2.13	2.55	0.65	2.56
Natural coarse aggregate	13.2	2.97	1.58	6.95
Recycled coarse aggregate	9.60	2.31	6.60	6.81

SSD: Saturated surface dry.

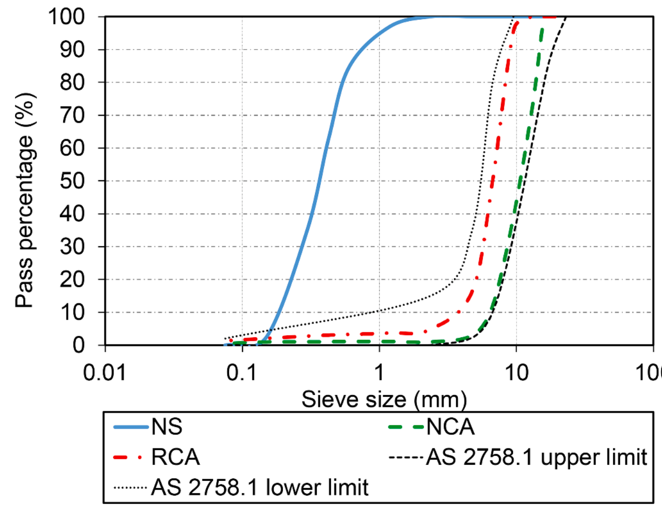
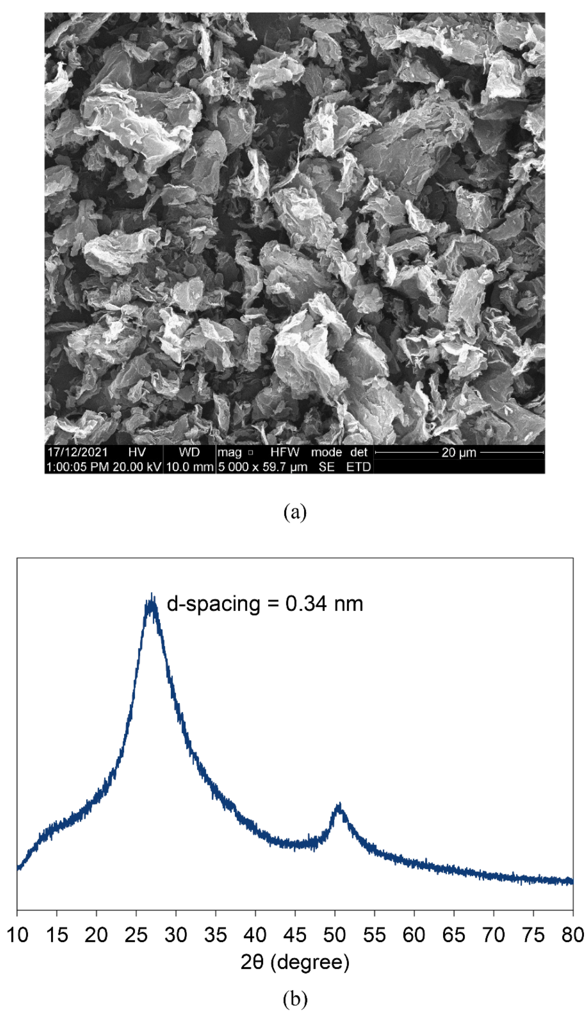


Fig. 1. Sieving analysis of aggregates (NS: natural sand, NCA: natural coarse aggregate, RCA: recycled coarse aggregate) with AS-2758.1 (2014) limits for coarse aggregates.



(a)

(b)

Fig. 2. (a) SEM and (b) XRD spectra of pristine graphene.

distribution of the nanomaterial on RCA pores due to their agglomeration (Wang et al., 2019). Based on Tables 1 and 2, all the treated RCAs experienced a higher water absorption than the NCA. The decreased absorption and increased gravity of the RCAs resulting from the

Fig. 3. Recycled coarse aggregates treated in pristine graphene solution.

incorporation of graphene can be due to the filling of RCA pores and reaction between graphene and the calcium hydroxide present in the mortar (Singh et al., 2018). Water absorption of the RCAs decreased by 3–23 % when they were treated with 3–30 wt% nanomaterials such as nanosilica (Li et al., 2021b; Zeng et al., 2020; Zhang et al., 2015).

3.3. Preparation of concrete mixes

Table 3 presents the mix proportions of different concrete mixtures. Six concrete batches were prepared, including a conventional concrete (CC) with no RCA and five RCA-based concrete mixes. The mix proportions were designed based on trial mixes and previous research (Ozbakkaloglu et al., 2018) to develop a target compressive strength of 60 MPa for the CC mix at 28 days of curing. In the concrete mixes containing RCA, 50 % of NCA content was replaced with RCA by

Table 2
Specific gravity and water absorption of pristine graphene-treated recycled coarse aggregates.

Pristine graphene dosage (%)	Specific gravity (in SSD)	Water absorption (%)
0	2.31	6.60
0.1	2.34	5.96
0.2	2.35	5.25
0.3	2.36	4.81
0.5	2.43	4.94

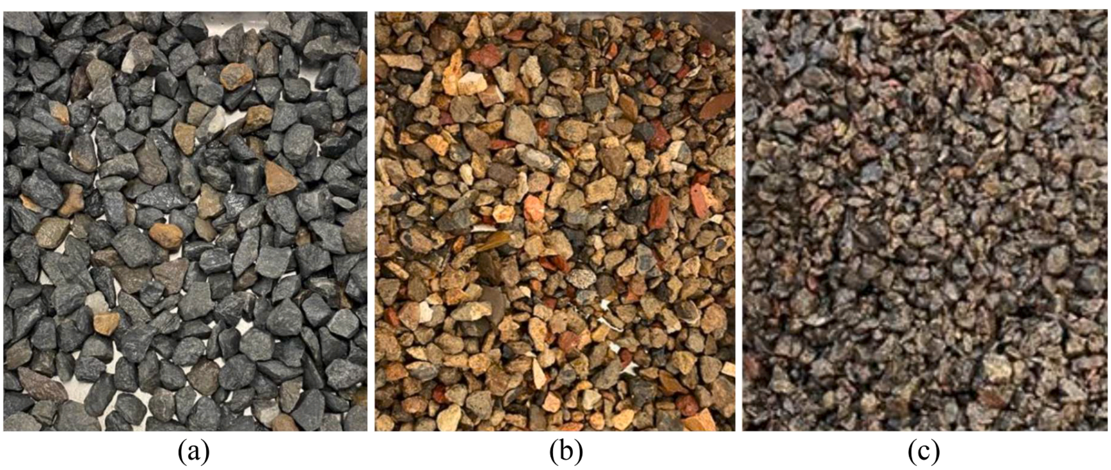


Fig. 4. (a) Natural coarse aggregate, (b) untreated RCA and (c) treated RCA.

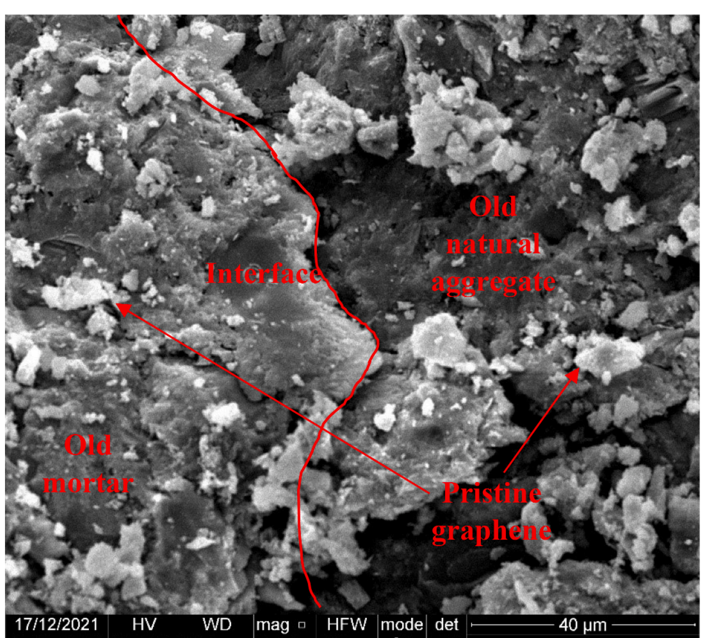


Fig. 5. Typical SEM image of treated recycled coarse aggregate by pristine graphene.

Table 3
Mix proportions of different concrete mixes.

Mix	Graphene (%)	Cement (kg/m^3)	NS (kg/m^3)	NCA (kg/m^3)	RCA (kg/m^3)	Effective water (kg/m^3)	SP (kg/m^3)	w_{eff}/c
CC	0	504	680	1104	–	170	6	0.35
R50P0	0	504	680	552	429	170	6	0.35
R50P0.1	0.1	504	680	552	435	170	6	0.35
R50P0.2	0.2	504	680	552	437	170	6	0.35
R50P0.3	0.3	504	680	552	439	170	6	0.35
R50P0.5	0.5	504	680	552	452	170	6	0.35

NS: natural river sand.
NCA: natural coarse aggregate.
RCA: recycled coarse aggregate.
SP: superplasticizer (70 % water by weight).

volume. The untreated and treated RCAs with 0, 0.1, 0.2, 0.3 and 0.5 % pristine graphene were used in the RCA concrete mixes for investigating the effect of RCA treatment by pristine graphene at different dosages on the performance of the concrete mixes.

In all the concrete mixes, the utilized effective water-to-cement ratio (w_{eff}/c) was consistent and set at 0.35. For the preparation of the mixes, sand, coarse aggregate and cement (dry ingredients) were blended for 3 min. Effective water amount was then gradually added, and mixing continued for about 5 min. The mixes were finally casted in the relevant molds. Following a 24 h curing period in the laboratory environment, all specimens were removed and then cured in a water tank at 23 ± 2 °C temperature till the day of testing.

Labelling of the mixes illustrated in Table 3 is defined as following: CC is the conventional concrete without any RCA. R and P letters stand for RCA and pristine graphene, respectively. The number after R represents the volume % of RCA. The number after P indicates the weight % of pristine graphene in RCA. For instance, R50P0.2 is a concrete mix with 50 % RCA treated by 0.2 % pristine graphene.

3.4. Concrete testing

Slump, axial compression, splitting tension, water absorption and drying shrinkage tests were done on each mix for evaluating fresh and hardened characteristics of the concrete mixes. The slump of the freshly prepared mixes was assessed promptly after mixing, in accordance with AS-1012.3.1 (2014). Axial compression (based on AS-1019.9 (2014)) and splitting tension tests (based on AS-1012.10 (2000)) were performed at 7 and 28 days of curing on cylindrical specimens measuring 150 × 300 mm. Water absorption test was done on cylindrical specimens measuring 150 × 50 mm at 28 days in accordance with AS-1012.21 (2014). Drying shrinkage test was performed on prism shape specimens measuring 40 × 40 × 160 mm at 7, 14, 21 and 28 days, as per AS-1012.13 (2015). Three nominally similar specimens were used for testing each mix and property. Fig. 6 shows photos of different tests.

The SEM and XRD tests were done in the Flinders Microscopy and Microanalysis Laboratory using an FEI Inspect F50 SEM (Flinders-University, 2023) and Bragg-Brentano geometry x-ray diffractometer for microstructural analysis of the mixes after curing period of 28 days, respectively.

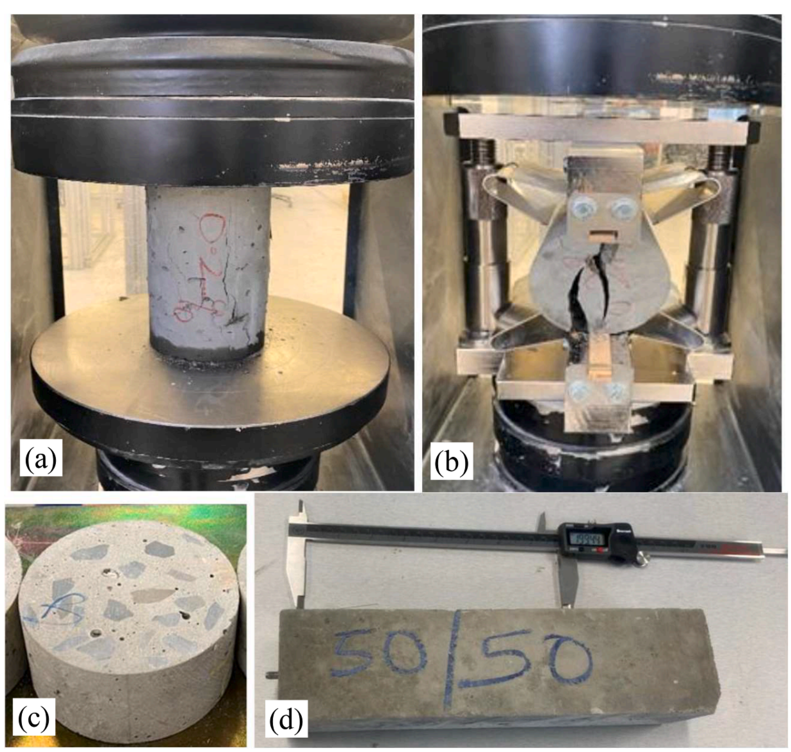


Fig. 6. (a) Axial compression, (b) splitting tension, (c) water absorption and (d) drying shrinkage tests.

4. Results and discussion

4.1. Slump of fresh concretes

Fig. 7 illustrates slump values of different mixes. As shown in the figure, 50 % replacement of NCA with untreated RCA led to about 7 % decrease in the workability of the mixes. This observation agrees with previous studies (e.g., (Majhi et al., 2018; Poon et al., 2007)). Although all of the coarse aggregates were used in SSD condition, this observation might be attributed to the rougher surface texture and more angularity of RCAs than those of NCAs, creating more friction within the RCA concrete mix (Silva et al., 2021).

From Fig. 7 it can be observed that treating RCAs with pristine graphene of up to 0.2 % dosage led to about 13 % increase in the workability of the mixes. However, further graphene dosage caused a reduction in the slump values. The increase of slump for 0.2 % graphene dosage can be ascribed to the reduced water absorption of the RCAs, which increases the available free water in the mix (Junak and Sicakova, 2017). Although the water absorption of the RCAs generally declined with higher graphene dosages (based on Table 2), the reduced slump of the mixes with further graphene dosage can be attributed to the absorption of a portion of the water by the increased graphene layers and an increase in the surface roughness of RCAs, which could offset the effect of reduced water absorption of RCAs on workability. This observation aligns with previous studies on presoaking RCAs with nanosilica (Chen and Jiao, 2022; Zhang et al., 2015, 2016) and sodium silicate solutions (Junak and Sicakova, 2017). Based on the figure, the mix with 0.2 % graphene treated RCA exhibited about 5 % higher workability than the conventional concrete, which was not accessible previously by other nanomaterials such as nanosilica (Chen and Jiao, 2022; Li et al., 2021a).

4.2. Compressive strength

Fig. 8 presents the compressive strength results of the mixes at 7 and 28 days. As can be observed, replacing 50 % NCA with untreated RCA caused about 18 % decrease in the compressive strength. This is expected because the bond between the untreated RCA particles and the new cement mortar is weaker compared to that for NCA. It is commonly accepted that the presence of the porous residual mortar on the surface of untreated RCAs and the two layers of ITZ can impede proper bonding with the aggregate, leading to a reduced strength (Mistri et al., 2021).

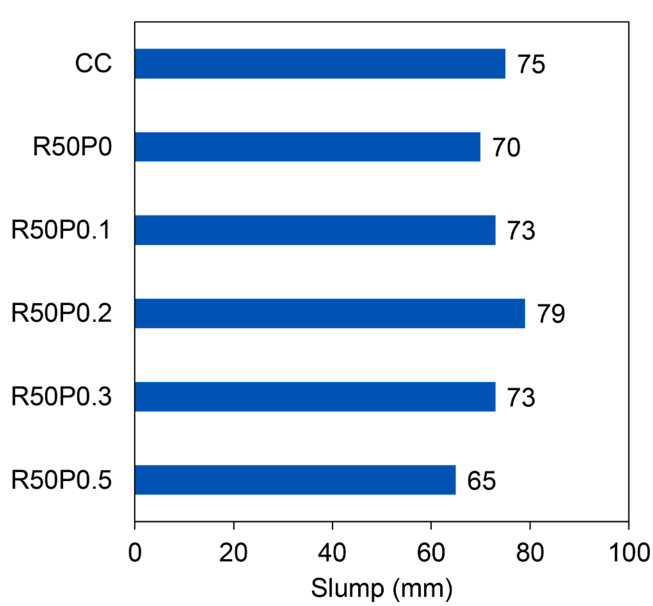


Fig. 7. Slump values of various fresh concrete mixes.

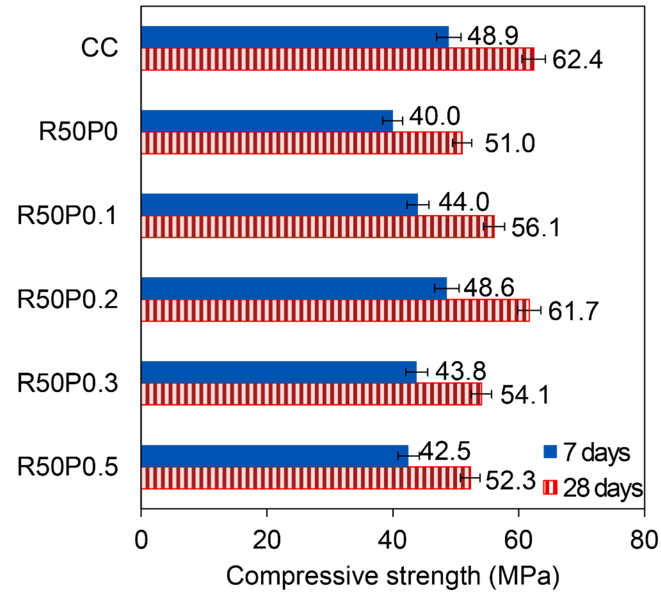


Fig. 8. Compressive strength values of various hardened concrete mixes.

However, as can be observed in Fig. 8, treating RCAs with pristine graphene led to improved strength of the mixes. RCA treatment with 0.1, 0.2, 0.3 and 0.5 % pristine graphene caused about 9, 21, 6 and 2 % increase of the compressive strength values, respectively. Based on the results, treating RCAs with up to 0.2 % pristine graphene caused an increased strength. This is because nanomaterials could fill the microcracks and voids present in the RCA particles and improve the ITZ between the RCAs and new mortar (Chen and Jiao, 2022). This filling effect reduces the porosity within the existing mortar layer, enhances the adhesion to the RCAs and results in an improved overall strength. However, treating RCAs with graphene dosages beyond 0.2 % resulted in a declined strength. At higher dosages, the pristine graphene layers agglomerate on the surface of RCAs and form clusters, which hinder their dispersion and proper distribution within the RCA matrix. The agglomerations lead to non-uniform filling effects, preventing effective microcrack and void filling. Therefore, excessive filling can impede RCA-to-mortar bonding and reduce the interlocking effect, negatively affecting the concrete strength. In addition, water will be required to achieve adequate workability, which can adversely affect the strength development properties. The dilution effect can offset the positive contributions of pristine graphene and result in a reduced strength (Zhan et al., 2023). Li et al. (Li et al., 2021a) assessed 28 days compressive strength of pre-sprayed RCA with nanosilica and reported that 4 wt% was the optimum nanosilica content for enhancing the concrete strength (by about 9 %). Li et al. (2021b) compared compressive strengths of RCA treated concrete by carbonation, nanosilica spraying and a combination of carbonation and pre-spraying methods. They found that pre-spraying RCAs with 3 wt% nanosilica led to the largest improvement of the concrete strength (10 %). Chen and Jiao (Chen and Jiao, 2022) pre-soaked RCAs with 0–2.5 wt% of nanosilica, and found 2 % as the optimum concentration of nanosilica to increase the concrete strength at 28 days (about 20 %). Lyu et al. (2022) assessed the presoaking influence of RCAs with 0–5 wt% nanosilica on the concrete compressive strength and found that highest 28-day strength improvement (about 12 %) was achieved when the RCAs were treated with 5 % nanosilica. However, in accordance with the findings of the present study, pre-soaking RCAs with only 0.2 wt% led to approximately 21 % increase in the 28-day compressive strength.

When comparing compressive strengths of conventional concrete and RCA concretes, it is observed that all the treated RCA concretes exhibited a lower strength, except for the RCA concrete with 0.2 wt% pristine graphene treatment, which showed similar compressive strength to the conventional concrete. This observation indicates that, at an optimum dosage, the pristine graphene could effectively fill in the RCA voids and microcracks and enhance the concrete strength.

4.3. Splitting tensile strength

The 7 and 28 days splitting tensile strengths of various concrete mixes are presented in Fig. 9. According to the figure, a same trend can be observed between the splitting tensile strength and compressive strength of the mixes. 50 % replacement of NCA with untreated RCA led to about 12 % decrease in the tensile strength. Furthermore, all mixes with treated RCA exhibited a higher tensile strength compared to the mix with untreated RCA. Treatment of RCAs by 0.1, 0.2, 0.3 and 0.5 % pristine graphene led to about 6, 12, 5 and 2 % increase in the tensile strength of the mixes, respectively. Based on this observation, for a given pristine graphene dosage, the increases in the splitting tensile strength by RCA treatments were smaller than those in the compressive strength. This indicates that the tensile strength of the mixes was less sensitive to the attached old mortar of the RCAs compared to the compressive strength.

Treating RCAs with up to 0.2 % pristine graphene caused an increased tensile strength, but further dosage of graphene led to a decreased tensile strength. As was discussed before, the increased strength of the mixes can be due to the pore filling effect of uniformly distributed pristine graphene on strengthening the ITZ of RCAs, and the decreased strength of the mixes can be due to the agglomeration and non-uniform filling effect of the pristine graphene, preventing effective microcrack and pore filling. Zhang et al. (2015) studied the splitting tensile strength of concretes with 50 % presoaked RCA in 30 wt% nanosilica, and reported that the strength of the treated RCA mix was about 7 % lower compared to that of NCA concrete. However, in this study, all the treated RCA concretes experienced a small enhancement in the tensile strength in comparison to the control concrete (RCA mix without graphene). Amongst these, the RCA-based concrete with 0.2 wt % pristine graphene treatment exhibited a similar tensile strength to the conventional concrete.

4.4. Water absorption

Fig. 10 illustrates the results of the water absorption test on various concrete mixes at 28 days. 50 % replacement of NCA with untreated RCA

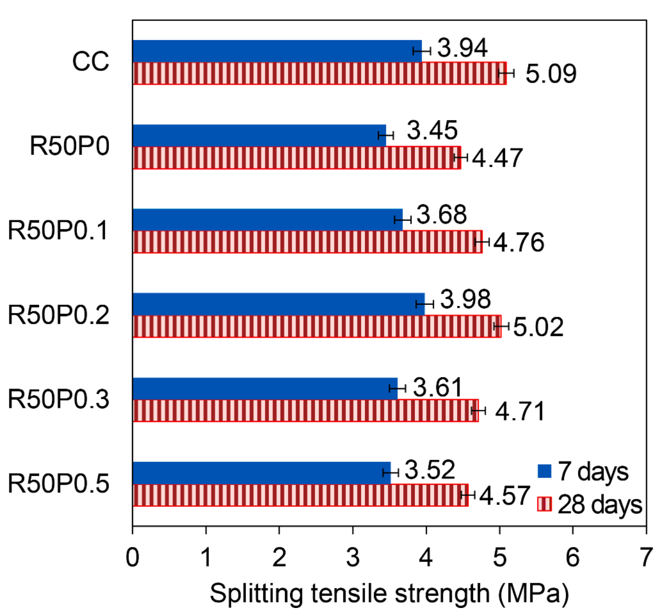


Fig. 9. Splitting tensile strength values of various hardened concrete mixes.

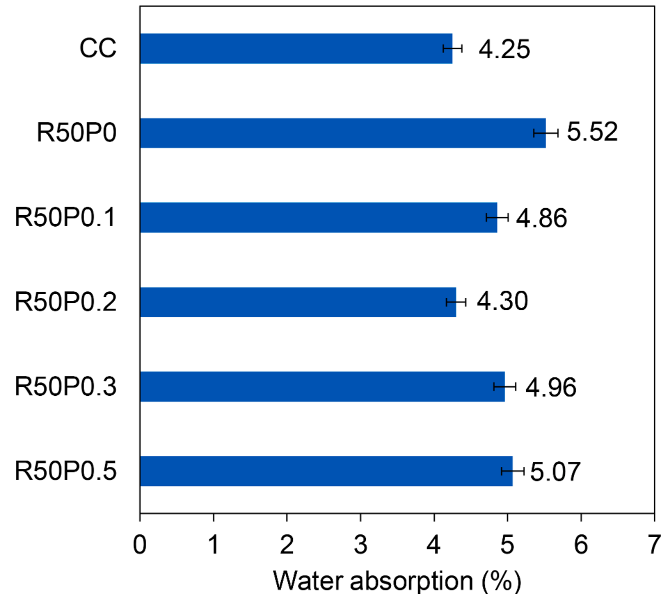


Fig. 10. Water absorption values of various hardened concrete mixes.

led to about 30 % increase in the water absorption. This behavior agrees with literature (e.g., (Li, 2020; Silva et al., 2021)) and is attributed to the porous attached old mortar of the RCA, which facilitates the penetration of water more readily into the concrete matrix (Li, 2020). However, based on the figure, all mixes with treated RCA exhibited a lower water absorption than that containing untreated RCA. Mixes with 0.1, 0.2, 0.3 and 0.5 % pristine graphene-treated RCAs had approximately 12, 22, 10 and 8 % lower absorption in comparison to the untreated RCA mix, respectively. The treatment of RCAs with up to 0.2 % pristine graphene caused a decreased absorption. The observation can be attributed to the filling pores of the concrete by pristine graphene, reducing its permeability and limiting the ingress of water (Chen and Jiao, 2022). However, it can be observed in Fig. 10 that further pristine graphene dosage beyond 0.2 % resulted in an increased water absorption of the concrete mixes. This observation can be attributed to the excessive graphene dosage, which can cause agglomeration of the graphene, where the particles cluster together.

Singh et al. (2018) reported that presoaking RCAs with 3 wt% nanosilica caused a 6 % decrease in the water absorption of RCA concrete. However, treated RCA concrete still exhibited a 19 % higher water absorption than NCA concrete. Li et al. (2021a) investigated the rate of water absorption of mixes with presoaked nanosilica and found that rates of initial and secondary absorptions of treated RCA concretes (at optimum dosage of 3 wt%) were about 36 and 38 % lower than the untreated RCA concrete, respectively. However, they were still higher than those of the NCA concrete. By comparison between the water absorption of the conventional concrete and the RCA concretes of this study, all the treated RCA mixes exhibited a larger absorption (14–19 %), with the exception of the mix incorporating 0.2 wt% pristine graphene treated RCA, which showed similar absorption to the conventional concrete.

4.5. Drying shrinkage

Fig. 11 shows results of the drying shrinkage test on various concrete mixes at 7, 14, 21 and 28 days. At 7, 14, 21 and 28 days of curing, 50 % replacement of NCA with untreated RCA caused about 12, 33, 31 and 28 % increase in the drying shrinkage of the mixes, respectively. This observation agrees with the literature (e.g., (Wang et al., 2020; Zhang et al., 2020)), and can be because of the larger porosity and water absorption of untreated RCAs in comparison to those of NCA, causing

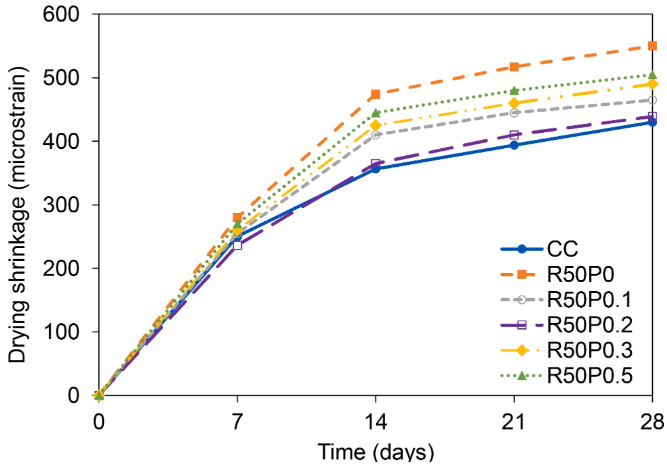


Fig. 11. Drying shrinkage values of various hardened concrete mixes.

increased shrinkage during the drying process of the RCA concrete mixes (Mao et al., 2021).

As can be seen in Fig. 11, treating RCAs by pristine graphene caused the shrinkage reduction. At 28 days, mixes with 0.1, 0.2, 0.3 and 0.5 % pristine graphene-treated RCAs exhibited about 15, 20, 11 and 8 % lower drying shrinkage compared to the untreated RCA mix, respectively. Consequently, treating RCAs with up to 0.2 % pristine graphene led to a decrease of the shrinkage of the mixes. This is because the graphene helps to fill the pores and capillary channels within the RCA matrix, reducing its overall porosity. This, in turn, restricts the water movement and reduces the drying shrinkage of concrete (Zhan et al., 2022). However, using pristine graphene of higher than 0.2 % dosage caused an increased shrinkage of the mixes. This observation can be ascribed to the graphene agglomeration and low effect to fill the RCA pores, which act as pathways for moisture movement during drying. This connectivity of pores allows more water to escape, resulting in a higher drying shrinkage of the concrete mixes (Zhan et al., 2022). In addition, poor dispersion of agglomerated graphene can lead to inadequate bonding between the RCA and the new cementitious matrix. As a result, the effectiveness of reducing shrinkage through pore filling and strengthening of the RCA is compromised, leading to an increased drying shrinkage of the concrete mix (Zhan et al., 2022). The evaluation of drying shrinkage in concrete incorporating treated RCA with nanomaterials has not been done in the literature yet. However, based on the literature, the treatment of RCAs with polymer solutions led to 9–15 % decrease in the shrinkage of the RCA concrete at 28 days (Kou and Poon, 2010; Zhu et al., 2013). In addition, presoaking RCAs in a slurry comprising cement, fly ash and water at a ratio of 1:1:0.15 with 30 % accelerator and 1 % retarder led to a about 9 % decrease in the shrinkage of the RCA concrete (Wang et al., 2020). Comparing the shrinkage of the conventional concrete and RCA concretes of this study, all the treated RCA concretes had a higher 28-day drying shrinkage (about 28, 8, 14 and 17 % at 0, 0.1, 0.3 and 0.5 % pristine graphene, respectively), except for the mix with 0.2 % pristine graphene treated RCA, which had a similar drying shrinkage to the conventional concrete. This observation indicates that, at an optimum dosage of 0.2 %, the pristine graphene could effectively prevent the high drying shrinkage of the RCA concrete mixes.

4.6. Microstructure analysis

4.6.1. Scanning electron microscopy (SEM)

Fig. 12(a) and (b) illustrates typical microstructures of the untreated and 0.2 % pristine graphene treated- RCA in concrete mixes by SEM, respectively. According to Fig. 12(a), the main phase composition of the surface of the untreated RCAs was calcium silicate hydrate (C-S-H) with

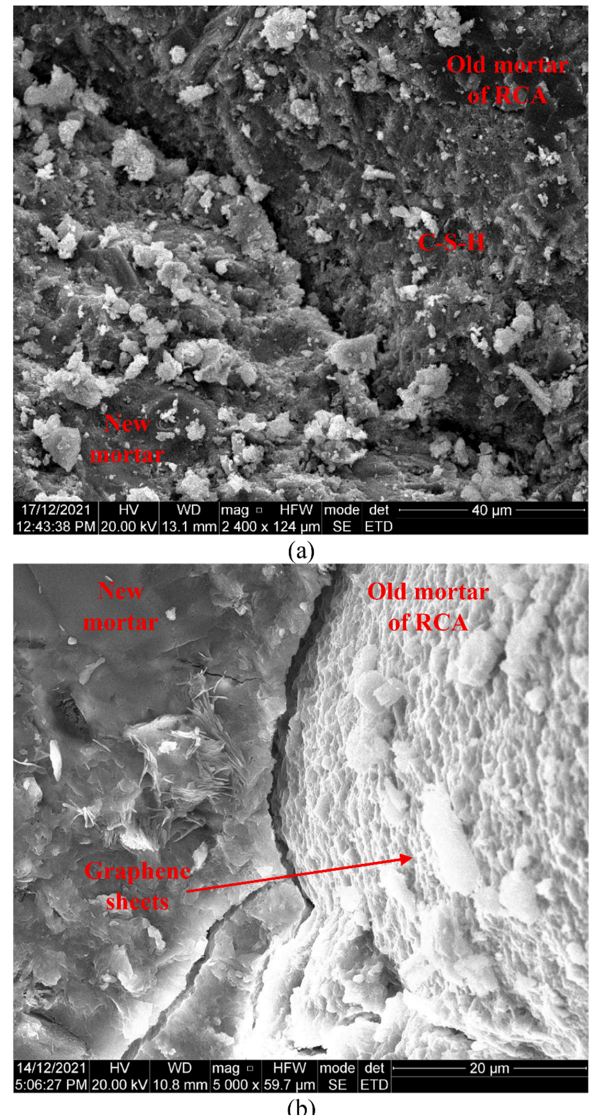


Fig. 12. SEM micrographs of the surface of (a) untreated and (b) 0.2 % pristine graphene treated-RCA in concrete mixes.

a porous structure. However, based on Fig. 12(b), the surface of the treated RCAs was denser and covered by pristine graphene particles. In addition, some pristine graphene particles penetrated the pores of the RCA to react with the old C-S-H owing to the nucleation effect of the pristine graphene materials (Lin and Du, 2020), thereby densifying the microstructure of the RCAs. These effects can explain the reason why the concrete mixes with treated RCAs exhibited lower water absorption and drying shrinkage and higher strength compared to those with untreated RCAs.

Fig. 13 presents the SEM images of various concrete mixes at the interface between new mortar and coarse aggregates. As can be seen in Fig. 13(a) and (b), the interface of the mix with untreated RCA was significantly porous compared to that with NCA. When the RCAs were treated by pristine graphene at up to 0.2 wt% dosage, the interface of the mixes became less porous. However, further increase in the graphene dosage led to a more porous microstructure at the interface.

Fig. 14 shows the low-magnification SEM images of different concrete mixes. Based on Fig. 14(a) and (b), the mix with untreated RCA had more and wider microcracks in its microstructure than the conventional concrete. The existence of more pores and microcracks in the microstructure of the untreated RCA mixes can explain the reason why the concrete mixes with untreated RCAs had higher water absorption and

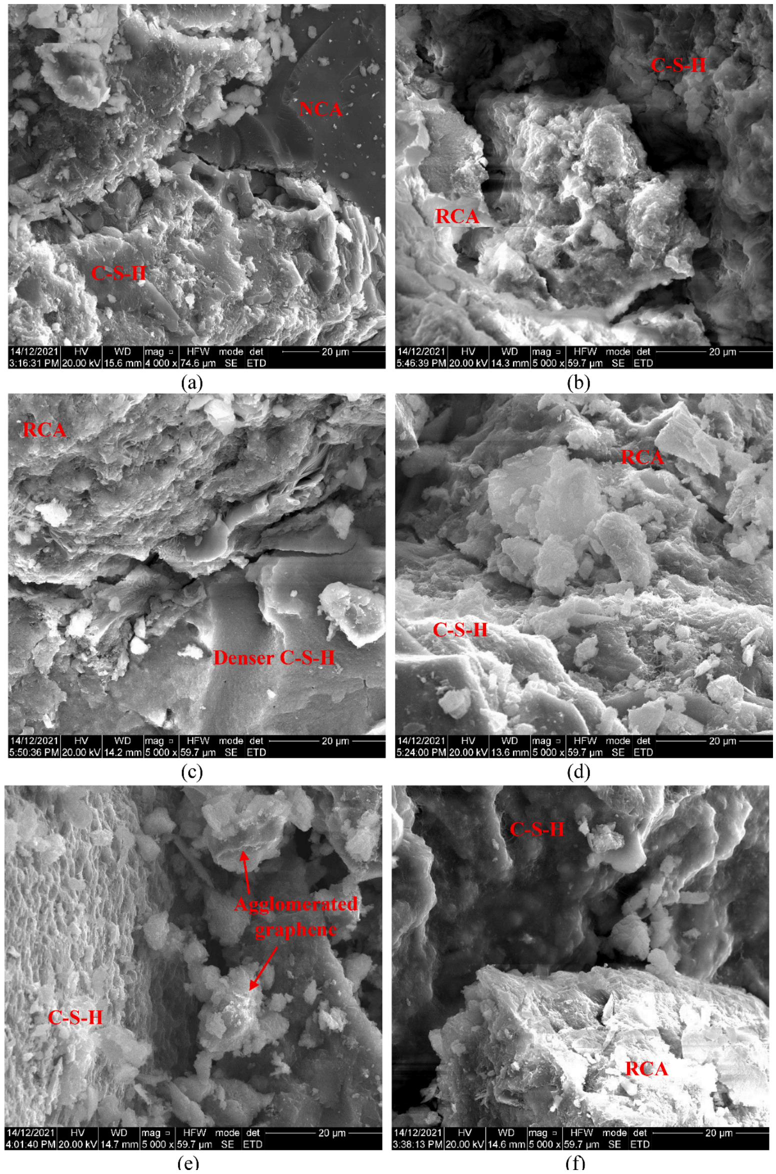


Fig. 13. SEM images of (a) CC, (b) R50P0, (c) R50P0.1, (d) R50P0.2, (e) R50P0.3 and (f) R50P0.5 mixes.

drying shrinkage and lower strength than the conventional concrete. According to Fig. 14(c) and (d), an increase in the pristine graphene dosage by up to 0.2 wt% caused a decrease in the width and number of microcracks in the microstructure of the mixes. However, further increase in the graphene dosage led to an increase in the width and number of the microcracks of the mixes, as shown in Fig. 14(e) and (f). These observations can explain why 0.2 wt% was the optimum pristine graphene dosage of treating RCAs to achieve a concrete mix with similar durability and mechanical performances to the conventional concrete.

4.6.2. X-ray diffraction analysis (XRD)

The XRD spectra of various mixes at the ITZ are displayed in Fig. 15. For conducting more accurate and easier comparisons between the crystalline phases, all the spectra were normalized. According to the figure, the primary peak of the mixes was at the scattering angle (2θ) of 29.5° as an indicative of C-S-H, followed by 24.5° and 59° for tricalcium silicate (C_3S), 34.5° for portlandite (C—H) and 42.5° for dicalcium silicate (C_2S). No new crystalline phases were created by incorporating pristine graphene in the RCAs. Based on the figure, the untreated RCA concrete mix had a lower cumulative intensity of the peaks than the conventional concrete mix. Treatment of RCAs with pristine graphene led to an increase in the peaks' intensity, indicating improved crystallinity at the ITZ of the treated RCA concrete, which caused stronger ITZ in the treated RCA concrete than that in the untreated RCA concrete. It can also be observed that the intensity of the peaks increased when the RCAs were treated by up to 0.2 % pristine graphene. The intensity then generally decreased when the RCAs were treated with further pristine graphene dosage. This observation aligns with the SEM analysis results and can be related to the uniform pore filling and agglomeration behavior of the pristine graphene at low and high dosages, respectively.

4.7. Cost analysis

Regarding graphene cost, it involves a simple soaking process, and the production line is easy to build. Although some commercial graphene products are now between \$80 and \$300 per kg, these prices might be considered as acceptable for the concrete industry considering the low concentration and high benefits offering by the graphene to RCA and concrete. Based on the obtained experimental results, using only 0.2 % pristine graphene suspension (which is about 1 kg pristine graphene in 1 m^3 of treated RCA concrete) significantly improved the properties of the RCA concrete. Table 4 presents the cost analysis of the conventional

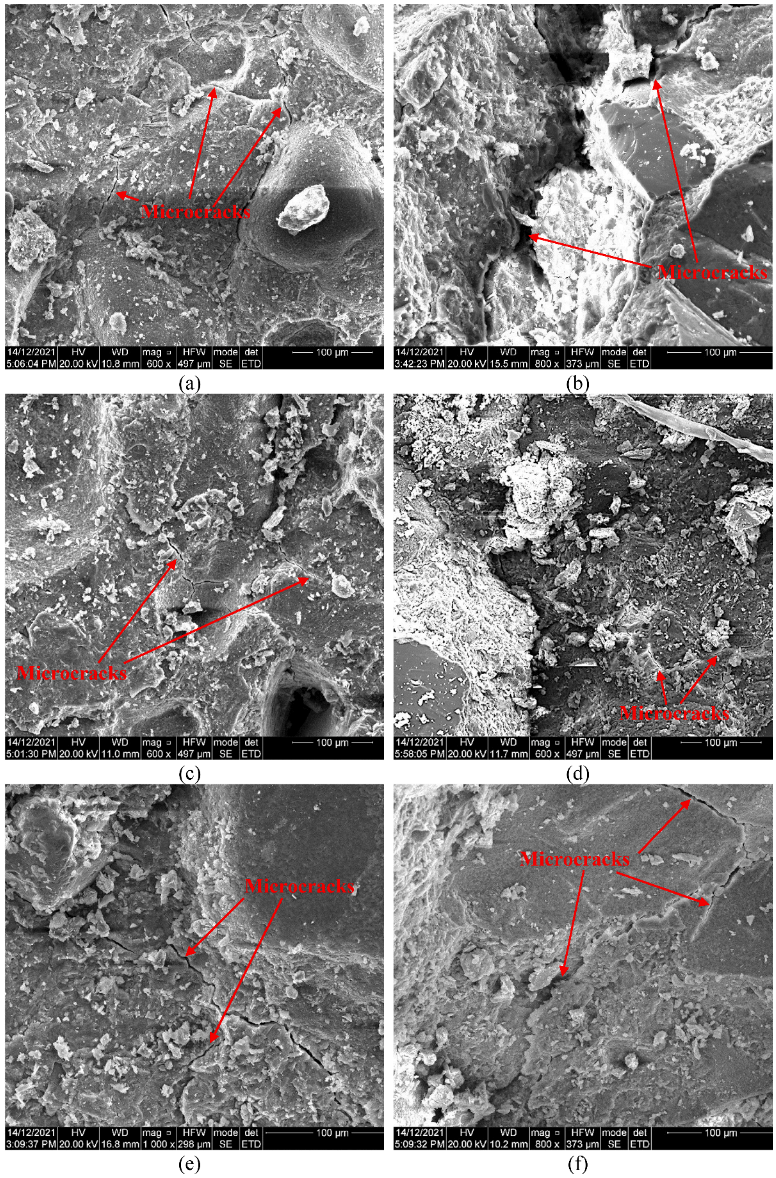


Fig. 14. Cracking pattern of (a) CC, (b) R50P0, (c) R50P0.1, (d) R50P0.2, (e) R50P0.3 and (f) R50P0.5 mixes.

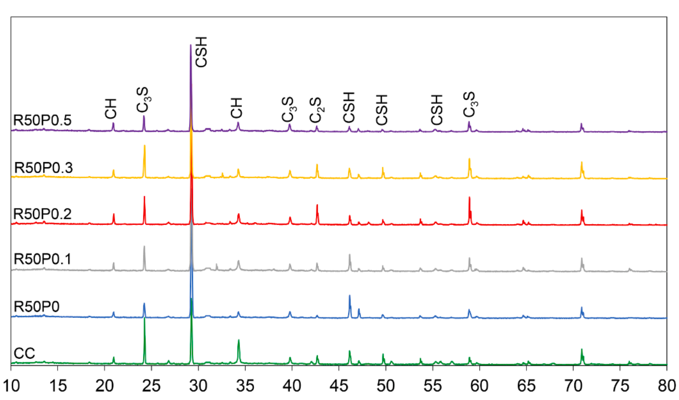


Fig. 15. XRD spectra of different concrete mixes.

concrete mix (CC) and the mix with 50 % RCA reinforced with 0.2 % pristine graphene (R50P0.2). It should be noted that the analysis is based on the average cost of each material in Australia excluding labor and delivery costs: cement ($10 per 20 kg), sand ($30 per ton), NCA ($70 per ton), RCA ($40 per ton), graphene ($190 per kg) and superplasticizer ($11 per liter) for 1 m^3 of concrete. Based on the cost analysis, the cost of the optimized graphene-reinforced RCA concrete is currently higher than that of the conventional concrete. However, based on existing reports (e.g., (Kireev et al., 2021; Manchala et al., 2019)), the price of graphene derivatives is expected to be reduced in the following years to make the graphene application in the concrete industry more

Table 4
Cost analysis of CC and R50P0.2 mixes.

Material	CC	R50P0.2	CC	R50P0.2
	kg per m^3		Average cost ($) per m^3	
Cement	504	504	252	252
NS	680	680	20	20
NCA	1104	552	76	38
RCA	0	437	0	17
Graphene	0	1	0	190
SP	6	6	66	66
Total average cost ($)	–	–	414	583

attractive.

5. Conclusions

This study has assessed the efficiency of presoaking RCAs by pristine graphene on mechanical, durability and microstructural characteristics of concrete containing 50 % RCA as a replacement for NCA. Below conclusions can be made:

1. Specific gravity of RCAs increases with increasing pristine graphene concentration. An increase in the concentration of graphene to 0.3 wt % causes a 27 % decrease in the water absorption of RCAs. However, further increase in the graphene concentration from 0.3 to 0.5 wt% results in a 3 % increase in the water absorption of the RCAs, which can be attributed to the non-uniform distribution and agglomeration of the graphene particles within the RCA pores at high concentrations.
2. Treating RCAs with pristine graphene at up to 0.2 wt% results in about 13 % increase in the workability of the RCA concrete. However, further graphene concentration causes a decreased workability. The concrete with 0.2 wt% pristine graphene treated-RCA exhibits about 5 % higher workability than the conventional concrete.
3. Presoaking RCAs with pristine graphene at up to 0.2 wt% leads to an increase (about 21 and 12 %, respectively) in the compressive strength and splitting tensile strength of RCA concrete. This strengths improvement is attributed to the pore filling effect of the graphene which enhances the bonding and decreases the porosity within the RCA and concrete matrix. Further graphene concentrations cause a decrease in the strengths, which is because of the agglomeration of the graphene at high concentrations, leading to non-uniform filling effects and preventing effective microcrack and void filling. However, treated RCA concretes exhibit lower strengths, except for the RCA concrete with 0.2 wt% pristine graphene treatment, which exhibits similar strengths to the conventional concrete.
4. Concrete incorporating presoaked RCAs in pristine graphene exhibits a lower water absorption (8–22 %) and drying shrinkage (8–20 %) than untreated RCA concrete. However, all the treated RCA concretes exhibit higher water absorption (14–19 %) and drying shrinkage (8–17 %), except for the concrete with 0.2 wt% pristine graphene treated-RCA, which shows similar water absorption and drying shrinkage to the conventional concrete.
5. The SEM analyses revealed that the surface of the treated RCAs was denser and covered by pristine graphene particles than untreated RCAs. It was also noted that augmenting the pristine graphene concentration by up to 0.2 wt% caused a decrease in the width and number of microcracks and porosity of the RCA concrete. However, further increase in the graphene concentration led to an increase in the width and number of the microcracks and porosity of the RCA concrete. The XRD spectra revealed that the intensity of the cement hydration peaks increased when the RCAs were treated with up to 0.2 wt% pristine graphene and then decreased when they were treated with further graphene concentration.

In summary, at an optimum concentration of 0.2 wt%, the pristine graphene could effectively fill the RCA voids and microcracks and improve the fresh and hardened characteristics of concrete mix. These results are promising as they show that a lower concentration of pristine graphene is required compared to other nanomaterials used previously to develop RCA concretes with similar properties to conventional concrete, which can decrease the cost of treated RCA concretes. The developed RCA concrete will be suitable for a wide range of applications where conventional high-strength concretes are currently being used, including bridges and infrastructure, high-rise buildings, marine structures, and repair and rehabilitation. However, further studies are needed to manipulate properties of graphene for different concrete and RCA grades obtained from different resources.

CRediT authorship contribution statement

Aliakbar Gholampour: Formal analysis, Conceptualization, Data curation, Investigation, Methodology, Resources, Project administration, Writing – original draft, Writing – review & editing. **Massoud Sofi:** Formal analysis, Validation, Writing – review & editing. **Houman Alipooramirabad:** Formal analysis, Writing – original draft, Writing – review & editing. **Youhong Tang:** Formal analysis, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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