

Effects of Pre-treatments on Microstructure and Mechanical Properties of Recycled Concrete Aggregates

Shalaka Nirantar^{1,*}, Premanand Naktode²

¹Faculty of Civil Engineering, N. K. Orchid College of Engineering & Technology, Solapur, 413002, India

²Civil Engineering Faculty, Sandip University, Nashik, 422213, India

Received November 12, 2022; Revised February 14, 2023; Accepted March 12, 2023

Cite This Paper in the Following Citation Styles

(a): [1] Shalaka Nirantar, Premanand Naktode, "Effects of Pre-Treatments on Microstructure and Mechanical Properties of Recycled Concrete Aggregates," *Civil Engineering and Architecture*, Vol. 11, No. 3, pp. 1297 - 1305, 2023. DOI: 10.13189/cea.2023.110315.

(b): Shalaka Nirantar, Premanand Naktode (2023). *Effects of Pre-Treatments on Microstructure and Mechanical Properties of Recycled Concrete Aggregates*. *Civil Engineering and Architecture*, 11(3), 1297 - 1305. DOI: 10.13189/cea.2023.110315.

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Abstract Deficiency of natural resources in construction industry is one of the major issues in today's construction era. Another major problem, which not only construction industry, but the entire world is facing, is the management of construction and demolition (C&D) waste. Therefore, it becomes inevitable to recycle the C&D waste and to replace it partially with natural aggregate so as to reduce the strain on natural resources. Recycled concrete aggregates (RCAs) cannot be directly reused as it possesses poor quality in terms of physical and mechanical properties. The objective of this experimental work is to improve the properties of RCAs by using pretreatments such as mechanical abrasion, water soaking, acid soaking, and heating. It is found that using mechanical abrasion, weak acid soaking and moderate heating, the micro structure of the RCA is improved. Water absorption and porosity are improved by 20% and 17.75% respectively. Using pre-treated RCAs, with partial and complete replacement for natural aggregates, concrete cubes were cast and tested for evaluation of compressive, split tensile and flexural strengths. Compressive strength for 40% replacement is improved by 17.41% as compared with the M25 grade concrete made using natural aggregates. Also, replacement by 100% is possible and compressive strengths are comparable. Thus, using the pre-treatments, C&D waste utilization, reduction in consumption of natural aggregates (NA) is possible in medium grade applications leading to sustainable construction.

Keywords Recycled Aggregates, Clung Mortar, C&D Waste, Treatments, Sustainable Construction

1. Introduction

Aggregate is a key component in the building process. The aggregate market is projected to grow by 6.85% annually [1, 2] according to numerous studies.

However, C&D waste is being produced in enormous quantities as a result of the demolition of old buildings to make way for new multi-story buildings.

There was 1181 million tonnes of construction and demolition debris produced worldwide, a decade ago [3]. There are currently around 150 million tonnes of C&D garbage produced annually in India [4]. Disposal and management of construction and demolition debris are becoming increasingly difficult due to a lack of suitable disposal sites.

The construction industry also faces a shortage of natural raw materials in the present day. The gap between the need for and the availability of natural aggregate is enormous. About 60 to 75% of concrete is made up of natural aggregate. As a result, there is a rise in the manufacturing of aggregate made from natural stones, which could potentially disrupt ecological balance [5]. Many countries, including India, are experiencing a severe aggregate scarcity at the present time [6].

Thus, recycling C&D waste is one of the relevant and effective methods to alleviate pressure on waste management and advance the development of sustainable construction practices.

Therefore, use of RCA helps to reduce both the natural resources and costs. But, with RCA, the interfacial zone between fresh and aged cement paste weakens, and it absorbs more water. This is a major drawback of using RCA and offers inferior quality as compared to natural aggregate concrete [7]. Results from microstructural analyses of recycled aggregate (RA) have shown that the presence of clung mortar significantly affects RA's bonding performance [8]. Wasted mortar stuck to the surface of aggregates causes problems such increased porosity, higher crushing value, reduced workability, higher creep, and shrinkage [9]. Researchers advocated for the use of recycled concrete aggregate for non-structural applications such as road-bed material, due to the material's inferior properties.

To achieve the same level of performance as natural aggregate concrete, RA characteristics must be improved. The main source of recycled aggregate concrete's low quality is the mortar that has adhered to the surface of old aggregate. When compared to natural aggregate concrete, clung mortar had a higher water absorption rate, weaker interfacial transition zones, and more micro cracking. [10]

If RA is to be used in the concrete mix, it must be treated first to improve its quality. Clung mortar can be partially removed with mechanical scrubbing and heat treatments. Up to 20% of NA can be replaced by RA by using these methods [11]. But, mechanical and physical properties of the final concrete degrade as the percentage of RCA used in the replacement process increases. Therefore, RCA replacement was limited at 30% [12-14]. Methods such as pre-soaking, surface treatment, water compensation, mechanical, silica fume impregnating, CO₂ injection, and pozzolanic materials can all be used to reduce the need for additional mortar. Pre-soaking RCA in acids such HCL, H₂SO₄, and H₃PO₄ was used to remove adhered mortar and boost RCA quality [15]. The researchers varied the acid dosage from 0.1 mol to 0.8 mol and the temperature from 100°C to 700°C.

Most acid therapies involved use of either HCL or H₂SO₄; however, H₂SO₄ treatments yielded better outcomes [16]. Additional coating with a solution of calcium meta-silicate after soaking in 0.5 mol HCL acid, resulted in changed aggregate surfaces, which in turn enhanced RA's characteristics [17]. The rate of water absorption was drastically decreased by using an impregnation technique based on a polymer containing silicon [18].

Micro-cracks, porosity, and weak interface transition zones are some of the drawbacks that arise from the crushing process of RCA. Scanning electron microscopy (SEM) analysis revealed that the broken concrete surface was covered with a substantial quantity of crumbs ranging from just a few microns to several hundred microns in sizes. Clung mortar adhered was visible as crumbs [19]. Poor quality in terms of mechanical properties and durability [20] results from drawbacks like ITZ and porosity, which adversely affect the microstructure.

Earlier studies reported a maximum 30% substitution of recycled aggregates for natural ones. Removing adhered mortar also required the use of strong acids and higher temperature ranges, which degraded the RA's surface quality and were also difficult to work with. The range of temperatures used in heat treatments was also crucial. Above 100°C [21, 22], clung mortar begins to loosen, but above 500°C, the aggregate surface begins to degrade.

Hence, in this research work, better pre-treatment methods and higher percentage of replacements were aimed. For removal of clung mortar and treating the surface of RA, use of mild acetic acid with different concentrations was determined. Also, moderate range of temperatures between 150°C to 350°C was decided for the heat treatments.

2. Materials

In the experiments, 53-grade Ordinary Portland Cement (OPC) as per the standards of IS 269-2015 and river sand as the fine aggregate, meeting the standards of IS 2386 (Part III)-1963 are used. Table 1 displays the characteristics of river sand. The NA used is in the form of crushed stone mined from local areas. As can be seen in Fig. 2, RA is made from a combination of locally sourced C&D trash and tested/crushed concrete specimen waste (a). First, the raw concrete was broken down by hand, and then it was put through a laboratory abrasion machine before being sieved. The aggregate used for RA is the material that can pass through a 20 mm filter but is still partially held by a 4.75 mm sieve. Particle size distributions measured in accordance with IS 383-1970 (IS 1970) are listed in Table 2 for both the natural and recycled coarse aggregates with a nominal size of 20 mm. Table 3 displays the results of an analysis of the mechanical properties of NA and RA performed in accordance with IS 2386-1963 (IS 1963).

Table 1. Properties of Natural Fine Aggregate River Sand

Test Properties	Results
Specific gravity	2.56
Fineness modulus	2.8
Bulk density (kg/m ³)	1655
Water absorption (%)	2.1

Table 2. Sieve Analysis Results of NA and RCA

Sieve size (mm)	Cumulative residue on sieve (%)	
	NA	RA
20.00	0.00	0.00
16.00	21.16	35.48
12.50	50.31	70.26
10.00	67.93	81.33
4.75	99.73	99.33

Table 3. Properties of NA and RA

Property	NA	RA
Specific gravity	2.56	2.67
Water Absorption (%)	0.5	5.1
Bulk density (kg/m ³)	1508	1570
Crushing Value	17	21.5
Impact Value	19	20
FM	7.1	6.68
Voids (%)	50.00	52.86
Abrasion value	29	45

3. Pre-treatments

3.1. Treatments to improve RA by removing clung mortar

The removal of adhered mortar from the aggregate's surface is one method suggested by the reviewed literature for enhancing RA quality. The primary element that lowers the quality of concrete is an excessive amount of mortar adhering to the surface of the aggregate. There are chemical, thermal, and mechanical ways to get rid of this unwanted mortar buildup. Fig. 1 depicts a variety of potential pre-treatments that can be applied to recycled aggregates. In addition, it is possible to use a combination of these approaches [23]. Some of the researchers employed a range of concentrations from 0.1 to 0.8 mol, for the three different acids, hydrochloric acid (HCl), sulphuric acid (H₂SO₄), and phosphoric acid (H₃PO₄). However, the presence of chloride and sulphate is increased when strong acids like as HCL, H₂SO₄ react with cement mortar adhering to the aggregate surface. The increasing sulphate and chloride levels may reduce the concrete's durability. Traditional heating methods were used by prior studies [24, 25], and temperatures ranged from 200°C to 950°C for varying times.

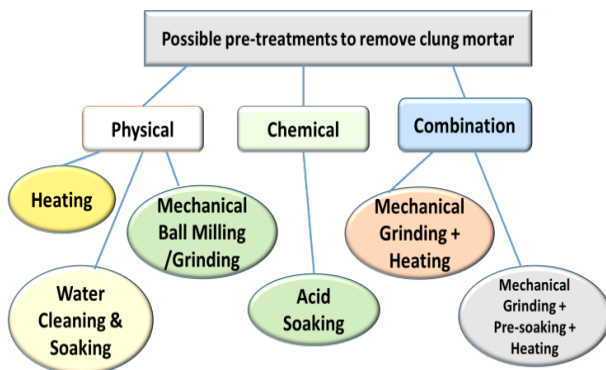


Figure 1. Possible pre-treatments on recycled aggregates

Mechanical methods can also be used to removal of clung mortar. Clung mortar was removed using two

distinct mechanical methods: grinding and an eccentric shaft rotor technique [26, 27].

For the present experimental work, considering the disadvantages and after effects of strong acid treatments and higher temperatures, it was decided to use weak acetic acid and moderate temperature range from 100°C to 300°C along with mechanical treatment.

3.1.1. Mechanical method

In this process, the concrete rubbles are mechanically rubbed by one another to get rid of the cement paste that has adhered to them. To get rid of the mortar stuck, recycled aggregates were tumbled through the Los Angeles abrasion machine, as illustrated in Fig. 2 (f).



Figure 2. RCA and different treatments

On the basis of the abrasion results of both natural coarse aggregate and recycled coarse aggregate, the Los Angeles abrasion machine's revolution speed and charge weight were determined. According to Table 3, RA has an abrasion value of 45%, while natural aggregate only has a value of 29%. As a result, it can be inferred that the contribution of clung mortar in RA to improving the abrasion value is only around 16%, with the remainder attributable to the inherent feature of the coarse aggregate. Experimentation led to the conclusion that rubbing the RA in a Los Angeles abrasion machine for 2-3 minutes using a

typical charge would be sufficient to remove the adhered mortar.

3.1.2. Presoaking in water

The recycled aggregates were cleaned with water and soaked for 24 hours at room temperature. Then, the aggregates were dried with light blower and by natural ventilation for one day, as shown in Fig. 2 (b). The RA obtained after water soaking is abbreviated as RA_w. Some trials for longer soaking period were also conducted.

3.1.3. Presoaking in acid

Since acetic acid (CH₃COOH) is a weaker acid, it is safe to handle and can be used to de-scale the stuck mortar. The aggregate samples were treated in the lab with solutions of varying molarities (0.1 M and 0.5 M). As can be seen in Fig. 2, aggregates were submerged in both solutions for 24 hours at room temperature before being allowed to dry on their own (c). A soaking in acid yields RAs designated RA_{AC0.1} and RA_{AC0.5}. It was also tried with stronger acids and for longer periods of time in the soaking solution.

3.1.4. Water Soaking and Heat Treatment

After being soaked in water and dried, the aggregate samples in Fig. 2 (d) and (e) were baked at 150°C, 300°C, and 500°C for 1 hour to undergo heat treatment (e). The RAs obtained following water soaking and heat treatment are abbreviated as RA_{WH150} and so on.

Thermal strains are produced by heat treatment as a result of the dissimilar thermal expansion of the original aggregate and the adhered mortar. The stuck mortar is weakened and dislodged from the aggregate surface.

Table 4. Effect of pre-treatments on WA

Treatment type	WA (%)	(%)WA change
RA	5.1	--
RA _w	4.78	6.27
RA _{WH150}	4.65	8.82
RA _{WH300}	4.67	8.43
RA _{WH500}	4.72	7.45
RA _{AC0.1}	4.5	11.76
RA _{AC0.1H150}	4.39	13.92
RA _{AC0.1H300}	4.45	12.74
RA _{AC0.1H500}	4.58	10.19
RA _{AC0.5}	4.2	17.64
RA _{AC0.5H150}	4.08	20
RA _{AC0.5H300}	4.11	19.41
RA _{AC0.5H500}	4.47	12.35

3.1.5. Acid Soaking and Heat Treatment

Heat treatment in oven at 150°C, 300°C, and 500°C for 1 hour was also applied to recycled aggregates that had been presoaked in an acidic atmosphere at room temperature for 24 hours before being dried. The RAs that were formed following acid soaking and heat treatment are denoted by

the notation RA_{AC0.1H150}, etc. A reaction between acetic acid and the recycled aggregates and mortar took place throughout the soaking procedure.

Table 5. Porosity percentage of RA with different treatments

Treatment type	Porosity (%)	Change (%)
RA	25.62	--
RA _w	24.01	6.28
RA _{WH150}	22.13	13.62
RA _{WH300}	23.06	9.99
RA _{WH500}	27.68	-8.04
RA _{AC0.1H150}	21.01	17.99
RA _{AC0.1H300}	21.62	15.62
RA _{AC0.1H500}	26.64	-3.98
RA _{AC0.5H150}	20.56	19.75
RA _{AC0.5H300}	21.96	14.28
RA _{AC0.5H500}	28.1	-9.67

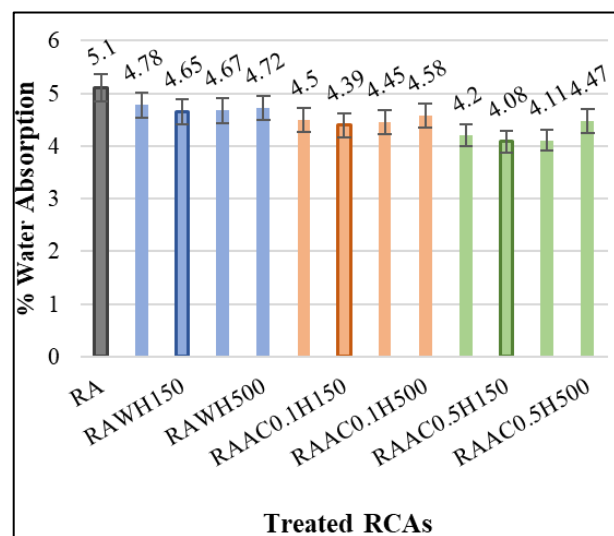


Figure 3. Effect of treatments on water absorption

Calcium carbonate (CaCO₃), calcium hydroxide (CH), and hydrated calcium silicate (C-S-H) all undergo an acidic reaction. As a result of this reaction, the stuck mortar is loosened, and RCA can be detached. Similarly, heating the aggregate surface weakens and loosens any additional mortar adhering to the aggregate.

The effects of these treatments on the properties of the aggregates are studied and properties of the recycled aggregates before and after treatments are compared and discussed as below.

The effects of the above treatments are presented in the Table 4. After water soaking, acid soaking, and heat treatments, it was found that water absorption of treated RAs was significantly enhanced. In Fig. 3, a comparison of the WA modification efficacy of RCA treated with different approaches is presented.

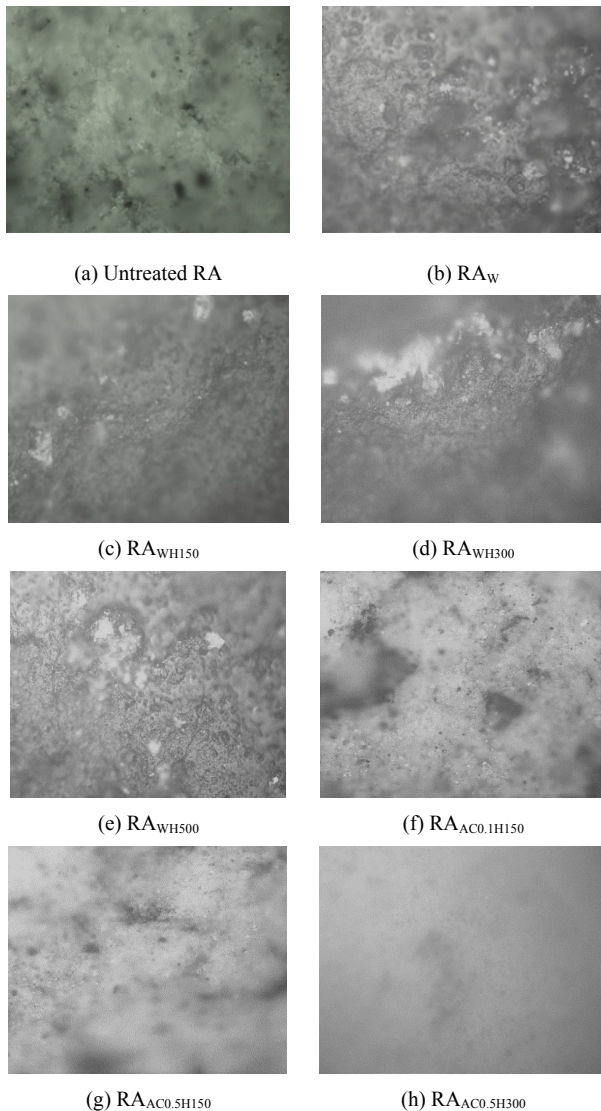


Figure 4. Microscopic images of aggregate surfaces

The data show that RCA samples heated at 150°C after being treated with 0.5 molarity acetic acid showed the greatest improvement, at 20%. The treatments that involved a longer soaking time or a higher acid concentration did not yield any results worth discussing.

3.1.6. Effects of Treatments on Microstructure

In this investigation, the surface microstructure and porosity of both untreated and treated RA were analyzed using a metallurgical inverted microscope with a magnification range of up to 1000X. Images taken with a microscope at a high magnification revealed irregularly shaped holes and particles. Surface texture, or the smoothness or roughness of an aggregate's outer layer, is categorized based on visual inspection and is affected by factors such as the material type, size, source, and crushing process. Microscopic images depicting the treatment's effectiveness are as seen in Fig. 4. RA and NA are radically different in their internal and external shape and surface texture. Fig. 4 (a) depicts the rough and porous surface

texture of untreated aggregate, where the clung mortar is plainly apparent. As a result, unprocessed RCA has a greater capacity to absorb liquids. The outer surface of the water-soaked RA sample shown in Fig. 4 (b) has less clung mortar than the surface. Here, water forms a thin film around the surface. As can be seen in Fig. 4 (c) and (d), when the sample was heated from 150°C to 300°C, the adhering mortar was removed, leaving a clear and smooth surface (d). As can be seen in Fig. 4 (e), the surface micro-cracks appeared after being heated to 500°C. Photographs (f) through (h) in Fig. 4 depict aggregates that have been heated and treated with acid. The mortar that had stuck to the RA surface was more easily removed with the help of an acidic solution of 0.5 mole, resulting in a smoother and more uniform surface. Increasing the temperature from 150°C to 300°C also resulted in a more uniform smooth surface. As the temperature was raised to 500°C and the acid concentration was increased, however, the material began to show signs of cracking and porosity on its outside surface. As can be seen in Figs. 4 taken with a microscope, the surface roughness of the acid-treated sample improves at a moderate temperature of 150°C, with only a trace of clung mortar remaining. Water absorption and porosity test results provided in Tables 4 and 5 are consistent with these findings.

3.1.7. Effects of Treatments on Porosity

The acid concentrations and heat treatment temperatures control the porosity of the aggregates. The Table 5 shows the observed porosity as a result of various treatments. When aggregate was heated to 300°C, the porosity improved for both water soaking and water soaking plus heat treatment, going from 6.28% to 13.62%. Porosity, however, decreased by 8.04% in the sample when heated to 500°C. Similarly, the porosity of materials subjected to acid soaking and heat treatment up to 300°C increased from 14.28% to 19.75%. There has been a significant reduction in permeability. At temperatures up to 500 °C, however, the porosity reduced by 9.67%.

3.2. Mix Proportions and Cube Casting

Since the parent concrete was of the same grade, the appropriate mix proportions for M 25 grade concrete with a target strength of 31.6 N/mm² at 28 days could be determined. The calculated weight ratios for the cement, fine aggregate, and coarse aggregate mix are 1:1.9:3.1. With a water-to-cement ratio of 0.5, the cement content was estimated to be 380 kg/m³. Cubes of 150 mm size as per Indian standard method [IS 10262 (IS 2019)] were cast.

Cubes cast with natural aggregate are used as a benchmark for comparison. Using the same reference, another 03 mix series utilizing treated RCAs and replacing NA by 40%, 60% and 100% [28] were cast. Previous studies have suggested that RCA can successfully replace up to 25–30% of coarse aggregate without requiring alterations to the wc ratio or lowering the concrete's

compressive strength

Table 6. Mix Proportions for Various Concrete Mixtures

Mix Series	W/C	Cement (kg)	Fine Aggregate (kg)	Course Aggregate (kg)	
				NA	RA
NAC	0.5	380	717	1170	0
RAC ₄₀	0.5	380	717	702	468
RAC ₆₀	0.5	380	717	468	702
RAC ₁₀₀	0.5	380	717	0	1170

Therefore, it was decided to cast concrete cubes with RCAs treated in this manner. The mix proportions for all combinations of treatments are as given in Table 6.

3.3. Evaluation of Mechanical Properties of Treated specimens of RCAs

Mechanical performance of treated RCAs is experimentally evaluated in terms of compressive strength. Details of the work carried out are given in the following sections.

Compressive strengths for all test cubes were determined at the age of 3, 7, and 28 days using compression testing machine of capacity 3,000 kN as per the standard testing procedure mentioned in IS 516:2021. Compressive strength for cubes cast with NA was found to be 34.00 N/mm². Compressive strengths obtained for 40%, 60% and 100% replacement categories are shown in Fig. 5, 6, and 7 respectively.

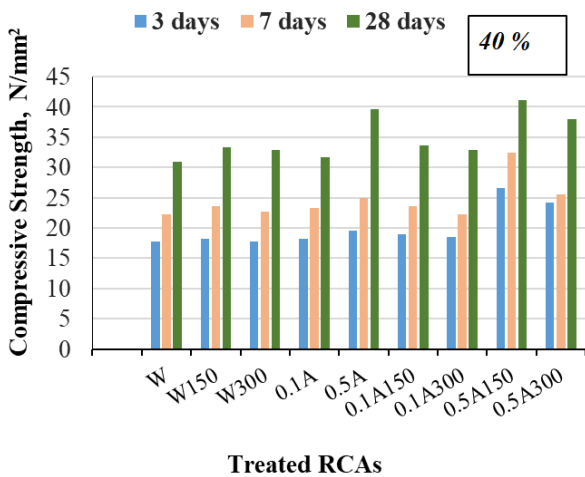


Figure 5. Compressive strengths of treated RCA specimens for 40% replacement

Also, it is observed that the compressive strengths for the specimens having RCAs treated with 0.5 M acetic acid followed by heating at 150°C are the highest among all three replacement categories. More importantly, it is seen that 40% partial replacement, treated with 0.5 M acetic acid and then heated to 150°C, yields the best compressive strength.

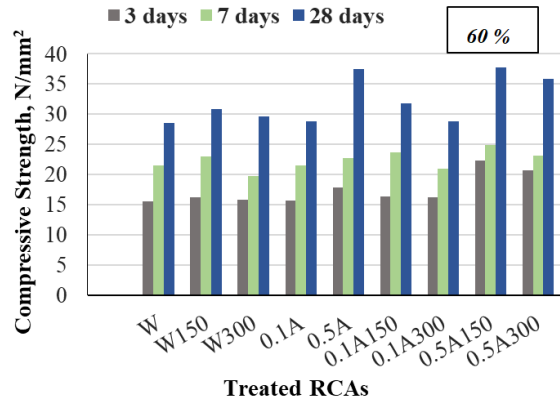


Figure 6. Compressive strengths of treated RCA specimens for 60% replacement

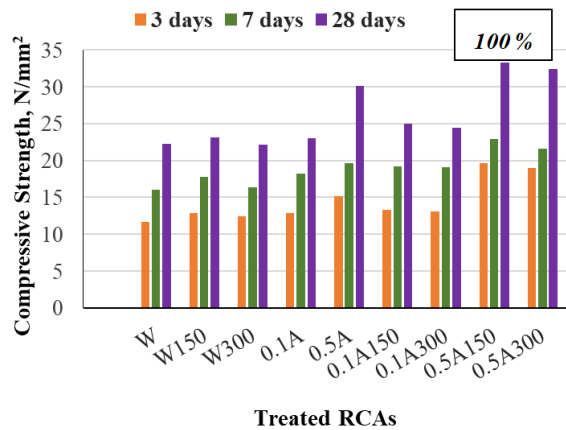


Figure 7. Compressive strengths of treated RCA specimens for 100% replacement

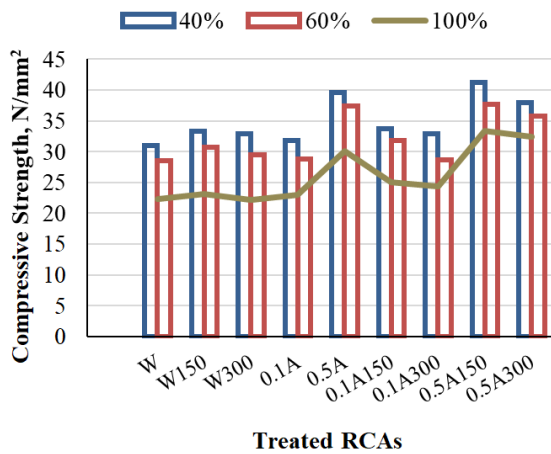


Figure 8. Comparison of compressive strengths of treated RCA specimens for 40, 60, and 100% replacements

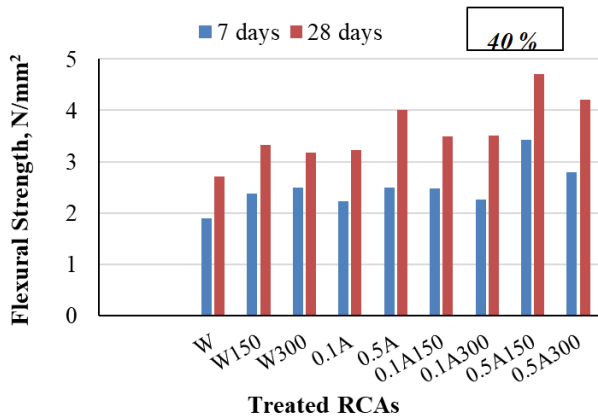


Figure 9. Tensile strengths of treated RCA specimens for 40% replacement

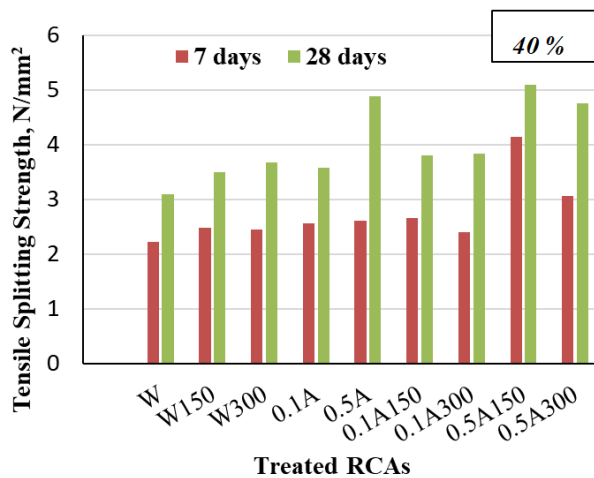


Figure 10. Flexural strengths of treated RCA specimens for 40% replacement

Fig. 8 also provides a visual comparison of the compressive strengths that were achieved across all treatment groups.

Tensile splitting strength of concrete was measured according to IS 516-1959. The tensile splitting strength of concrete was measured for cylindrical specimens with dimensions 150 mm x 300 mm. Flexural strength of concrete was measured in accordance with IS: 516-1959. Experiments used prisms made with size of 100 mm x 100 mm x 500 mm.

Fig. 9 depicts the splitting tensile strength and Fig. 10 shows the splitting flexural strength at 7 and 28 days of cure. The results show that RCA specimens treated with 0.5 M acetic acid and then heated to 150°C have the highest strengths attained for 40% partial replacement.

4. Results and Discussions

Overall micro-structure and interfacial transition zones of recycled aggregates improved due to the combined

pre-treatment of mechanical abrasion, weak acid soaking, and moderate heating. Microscopic examination reveals similar patterns, corroborated by the experimental findings. Mechanical qualities of the concrete blocks were enhanced as a result of the pre-treatments, which enhanced bonding between the recycled aggregates and the new cement paste.

Based on the experimental work carried out, the results obtained are discussed as below:

1. Water soaking treatments followed by moderate heating (150-300°C) improved water absorption as the clung mortar was removed efficiently. Moderate heating develops high thermal stresses in the residual mortar and loosens it from the RA surface. Further heating, resulted in development of cracks on the surface and affects water absorption. With the increase in concentration of acetic acid from 0.1 M to 0.5 M and heating from 150°C to 300°C, removed the clung mortar more effectively and water absorption efficiency improved to a maximum value of 20.00% at 0.5 M and 150°C. Increasing the acid concentration and soaking period further has not much impact on water absorption.
2. Porosity was improved from 6.28% to 13.62% for water soaking and water soaking plus heat treatment, when aggregate sample was heated up to 300°C. Porosity, however, decreased by 8.04% when the sample was heated to 500°C. Heating to higher temperatures causes micro-cracking and surface deterioration of RA. Similarly, for acid soaking and heat treatment up to 300°C, porosity improved remarkably from 14.28% to 19.75%.
3. Mechanical qualities of recycled concrete aggregate were enhanced by pretreatments that strengthened the interfacial zones and bonded the old RA to the new concrete. In comparison to 60% and 100% replacement series, compressive strengths are higher for the 40% replacement series. Maximum compressive strength of specimens (28 days) treated with RCAs for the mix series of 40% replacement is 41.17 N/mm². Thus, it improved by 17.41% as compared with the M25 grade (34.00 N/mm²) concrete made using natural aggregates. Also, for 60% replacement series, the strengths are comparable with that of NA. For 100% replacement, the results of 0.5 M treated (with /without heating) specimens are only comparable.
4. Similarly, splitting tensile and flexural strengths obtained for 40% replacements are also comparable and are ranging from 8-11% of the compressive strengths.

5. Conclusions

Based on the experimental results and above discussions, following conclusions are drawn:

1. The RCA's physical and mechanical qualities can be improved with careful selection for pre-treatments. The combined pre-treatments with mechanical scrubbing, weak acid soaking and moderate heating proved to be most efficient.
2. Using the pre-treatment methods, partial replacements by RCA with 40% and more are possible for medium grade design. However, the scope of the technique has to be explored for higher mix designs.
3. Partial replacement by using such pre-treatments can reduce the consumption of natural resources and be fruitful in effective C&D waste management and lead to sustainable construction applications.

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