

1 **Combined Effect of Multi-Stage Processing and Treatment Methods on the Physical,**  
2 **Chemical and Microstructure Properties of Recycled Concrete Aggregates**  
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## 29 **Abstract**

30 This research aims to examine the effects of multi-stage processing on reducing the old  
31 cement fractions and enhancing the quality of CRA (concrete recycled aggregate). The  
32 investigation involves the use of demolished concrete debris and subsequent treatments in  
33 both single and multi-stage processes. The recycled aggregates (RA) were obtained using a  
34 multi-stage jaw crushing process followed by utilising natural aggregate, untreated RA, RA  
35 treated with hydrochloric acid and sodium silicate immersion (single stage treatment) and  
36 RA treated with mechanical scrubbing and sodium silicate immersion in two separate stages  
37 (multi-stage treatment). The subsequent phase of the experimental inquiry involves assessing  
38 the physical attributes of both treated and untreated RA. This is followed by conducting  
39 microstructural examinations utilising techniques such as scanning electron microscopy  
40 (SEM), energy dispersive X-ray spectroscopy (EDAX), X-ray diffraction (XRD), Fourier  
41 transform infrared spectroscopy (FTIR), and thermogravimetry-differential thermal analysis  
42 (TG-DTA). The findings indicate that employing a two-step process, involving mechanical  
43 abrasion followed by immersion in sodium silicate, yields high-quality CRA. This  
44 conclusion is reinforced by the favourable physical performance observed. The water  
45 absorption values of CRA were lowered by 78% through single-stage treatments such as  
46 immersion in hydrochloric acid. The similar treatment is found to show densest concrete  
47 with Ca/Si ratio reduced to around 81% to that of untreated CRA. Additionally, for single  
48 stage treated CRA samples, microstructural study using FTIR verified the creation of  
49 additional hydration products, whereas for two stages treated CRA specimens, TGA analysis  
50 demonstrated the formation of stable CSH. According to the findings, it is advised to use a  
51 multi-stage process of jaw crushing, then treating it with mechanical abrasion and sodium  
52 silicate. This has the ability to improve the physical, chemical, and microstructural properties  
53 of CRA.

54 **Keywords:** Concrete recycled aggregates, multi cycle processing, hydrochloric acid,  
55 mechanical scrubbing, sodium silicate treatment, microstructure, sustainability.

## 56 **Highlights**

- 57 1. Waste concrete from building demolitions may be used to produce suitable CRA.
- 58 2. The quality of CRA is improved by multi-stage jaw crushing and two stage  
59 treatment approach.
- 60 3. CRA treated with hydrochloric acid illustrated lowest water absorption and Ca/Si  
61 atomic ratio.

62 4. Additional CSH formation is observed for two-stage treated CRA specimens along  
63 with some cracks.

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**LIST OF ABBREVIATIONS**

RAC-Recycled aggregate concrete

RA- Recycled Aggregate

CRA- Concrete recycled aggregate

GGBS- Ground granulated blast furnace slag

SF- Silica fume

SCMs- Supplementary cementitious materials

RHA- Rice husk ash

FA- Fly ash

TSMA- Two stages mixing approach

NS- Nano silica

C&D – Construction and demolition

ITZ- Interfacial transition zone

CDW- Construction and demolition waste

SEM- Scanning electron microscopy

EDAX-Energy dispersive X-ray spectroscopy

XRD- Xray diffraction

FTIR- Fourier transform infrared spectroscopy

TGA- Thermogravimetric analysis

INR- Indian Rupees

## 86 **1. Introduction**

87 The availability of crushed concrete has significantly increased due to the demolition of  
88 outdated buildings and the surplus concrete generated from current construction projects <sup>1</sup>.

89 The primary factors contributing to the accumulation of this debris can be attributed to the  
90 deteriorating state of the buildings and the fact that they have exceeded their lifespan, along  
91 with other solid structures <sup>2</sup>. Furthermore, both developing and developed nations are  
92 generating substantial quantities of construction and demolition waste (CDW) as a result of  
93 rapid urbanization, industrial growth, and increasing populations <sup>3</sup>. China and Russia, the  
94 US, and India account for the majority of the world's CDW production, with rates of 1020  
95 million metric tonnes, 600 million metric tonnes, and 400 million metric tonnes per year,  
96 respectively <sup>4</sup>. Additionally, numerous studies claim that the ongoing buildup of CDW  
97 results in potential risks to safety such as landslides, contamination to groundwater, and other  
98 problems <sup>5</sup>, and expanding landfill prices owing to the land space crunch <sup>6-8</sup>. On the other  
99 hand, governing entities are gravely concerned about the ongoing diminution of natural  
100 aggregates and the lack of accessible terrestrial positions <sup>9</sup>. From this point forward, the use  
101 of recycled aggregates (RA) can both maintain ecological balance and offer a sustainable  
102 solution to the depletion of natural resources. Figure 1 illustrates the C&D waste statistics  
103 across different Indian states as reported in a study <sup>10</sup>

104 It has been reported that a few of countries have already started incorporating RA <sup>11</sup>. It is  
105 found, nonetheless, that the RA has inferior engineering performance <sup>12</sup>. Old cementitious  
106 mortar on RA, that renders it porous and vulnerable to increasing water absorption and rising  
107 strain rates, is the main reason of the substandard performances <sup>11-18</sup>. This is further  
108 explained by the fact that the old interfacial transition zone (ITZ) created by the old mortar  
109 fractions is more vulnerable than the new ITZ created by the new cement paste and  
110 aggregates. The new ITZ is seen to exhibit additional CSH that makes it robust and leads to  
111 superior strength properties in RAC, in contrast to the old ITZ, that is claimed to consist of  
112 many microcracks and ettringite <sup>16,18-20</sup>. Adopting appropriate processing and treatment  
113 techniques is therefore crucial for the sustainable absorption of RA <sup>4</sup>. Crushing, screening,  
114 and contaminant exclusion, if any, are among the processing methods reported by different  
115 authors <sup>12,21,22</sup>. Specifically, a jaw crusher, impact crusher, cone crusher, roll crusher, etc.  
116 can be used to crush RA <sup>23-27</sup>. Jaw and impact crushing are two of the most frequently utilised  
117 crushing mechanisms among these crushers <sup>28</sup>. It should be mentioned that the choice of  
118 crushers becomes a crucial factor in the invention of RA because it significantly influence  
119 the aggregate's shape, size, and corresponding dispersion properties <sup>28</sup>. Additionally, a two-

120 stage crushing method, consisting of jaw crushing and then hammer milling, produces  
121 excellent RA, especially for improved mechanical properties in RAC <sup>29</sup>. An experimental  
122 study <sup>30</sup> examined the impact of multi-cycle jaw crushing (10 crushing cycles) on RA, it was  
123 discovered that recovering supplementary mortar required more number of processing  
124 sequences. The excess cycles, however, required supplementary energy than the standard  
125 crushing procedure produced. In addition, the contamination removal through manual  
126 cleaning brushing is suitable for small scale production of RA whereas in case of large-scale  
127 production, mechanical pre separation is recommended that can scrape off bulky waste such  
128 as wood pieces, plastics or papers <sup>31</sup>. Consequently, it is essential to investigate the ideal  
129 numeral of crushing cycles for procurement of RA.

130 Research has shown that treatments for RA include removing related mortar or its surface  
131 coating, improving the binder, consolidating adherent mortar, and improving the  
132 microstructure between fresh mortar and RA <sup>4</sup>. A detailed review and respective quantified  
133 data on several treatment methods adopted by various experimental studies is presented in  
134 Table 1. Additionally, carbon dioxide curing and nanoparticles help to reinforce remaining  
135 mortar by significantly lowering the water absorption and porosity of the RA <sup>32-34</sup>. Numerous  
136 methods, including grinding of RA <sup>35,36</sup>, heat grinding of RA <sup>37</sup>, and pre-soaking solutions  
137 <sup>38,39</sup>, could be used to eliminate any residual RA mortar. However, integrating the  
138 aforementioned approaches was shown to have several drawbacks, such as poor durability  
139 characteristics <sup>37</sup>, development in chloride and sulphate ions <sup>40</sup>, immense energy costs and  
140 cumulative levels of carbon dioxide ejections <sup>32</sup>, etc. In particular, the chemical treatments  
141 are found to depend on various aggregate parameters such as its composition, source of  
142 origin etc. For instance, aggregates with origin from a higher-grade parent concrete,  
143 presaturated with mineral admixtures and have fewer fractions of residual mortar shows  
144 lesser requirements of chemical treatments <sup>17,41,42</sup>. The use of acid treatments, chemical  
145 immersions through sodium silicate, and mechanical abrasion would offer a practical and  
146 successful eradication method for the leftover cementitious remains in light of the drawbacks  
147 connected with the aforementioned techniques. For instance, it has been found that  
148 immersion in sodium silicate lowers water absorption, limits the penetration of chloride ions,  
149 and creates a denser ITZ at the microstructure level <sup>43</sup>. Henceforth, in order to produce RAC  
150 in a sustainable manner, it is now necessary to investigate the impact of single and two stage  
151 treatment techniques.

152 In the ongoing study, the possibility of CRA processed from demolished concrete waste is  
153 investigated. The demolished concrete debris was first processed in multiple crushing cycles

154 using jaw crusher to attain the necessary CRA, which is then treated by means of single and  
155 two stage treatment approaches. The single stage treatment involved the immersion of CRA  
156 in hydrochloric acid and sodium silicate solution whereas two stage treatment consists of  
157 mechanical abrasion of processed CRA followed by sodium silicate immersion. Five  
158 different aggregate forms have been used in this experimental study. This study investigates  
159 the best approach to treatment in terms of the physical and microstructural performance of  
160 treated CRA samples, compared to control and untreated samples. Along with analysing  
161 treatment approaches' shortcomings, this manuscript makes recommendations for improving  
162 the sustainability measures of treatment strategies that have been implemented.

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TABLE 1 Assessment of several RA treatment approaches

Treatment technique	Variables and Impacts			References
	Chloride penetration in RAC	Water captivation In RAC	Corrosion resilience	
Integrating SCMs (GGBS+SF)	Decreases chloride penetration up to 53%	Decreases water absorption up to 8%	Minimal effect	44,52,53
Integrating ground rice husk ash (GRHA)	Offer resilience against chloride penetration	--	Better resilience against corrosion	44,52,54
Integrating GGBS	Decreases chloride penetration up to 67%	Decreases water absorption	Comparative corrosion resilience to NAC	44,52,55
Integrating FA	Decreases chloride penetration	Decreases water permeability	Corrosion density comparable to NAC at later age	44,52,56
Mechanical abrasion	Decreases chloride penetration	Decreases water absorption	--	44,48,52
Cooperative treatment of sodium silicate+ SF	Decreases chloride penetration up to 80%	--	--	43,44,52
Cooperative treatment of carbonation+ NS spray	Decreases chloride penetration by 24%	Decreases water absorption significantly	--	44,52,57
Cement+ NS slurry	Decreases chloride penetration by 15%	Decreases water absorption	--	44,52,58
Nano surface treatment	Lessens chloride ingress	--	--	44,52,58



## 168 2. Experimental Program

### 169 2.1 Materials Used

#### 170 2.1.1. Preparation of CRA

171 The demolished waste concrete cubes from the NITK structural and materials laboratory in  
172 Surathkal, India, were the source for the procurement of CRA. The discarded materials from  
173 the demolished concrete were cleaned first, then manually hammered to reduce size. In  
174 figure 2, a thorough multi cycle processing methodology is summarized <sup>44</sup>. It should be  
175 emphasized that repeated crushing cycles were used to reduce the quantity of attached  
176 cementitious remnants and simultaneously provide coarse CRA. After that, the crushed  
177 material was again sieved through an appropriate size fraction to produce coarse RA  
178 fractions that were relevant to the treatment methodology's sustainability metrics.

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### 181 2.2 Treatment procedures

#### 182 2.2.1 Hydrochloric Acid (HCL) immersion

183 The single stage treatments comprise of CRA immersions in hydrochloric acid (HCL\_CRA)  
184 and sodium silicate solution (SS\_CRA). The procedure uses HCL as an acidic solvent in a  
185 decomposition process to eliminate remnant mortar fractions from the original CRA. The  
186 percentage of the HCL solution for the treatment of CRA is kept at 15 %. The concentration  
187 is optimized to ensure that the impurities are effectively removed without causing significant  
188 damage to the CRA. The adopted acidic solution was diluted and the CRA were immersed  
189 in the prepared acidic environment at 20 °C for 48 hours <sup>45</sup>. Then aggregates were removed  
190 from the acidic solution. Afterward, the aggregates were cleaned with clear water to  
191 eradicate the acidic solvents along with remnant adhered cement paste followed by oven-  
192 drying at 100<sup>0</sup>-110<sup>0</sup> C for 24 hours.

#### 193 2.2.2 Sodium silicate (SS) immersion

194 The CRA were engrossed in 20% Na<sub>2</sub>SiO<sub>3</sub> solution (waterglass) then assorted and kept for  
195 an hour. Following their removal from the solution, the aggregates were allowed to air dry  
196 for a full day. When sodium silicate and old cementitious mortar are applied on RA, a  
197 chemical reaction develops. This reaction produces CSH, which is useful for fortifying the  
198 mortar remnants and obstructing the capillary pores in concrete, as illustrated in equation 1.



#### 200 2.2.3 Mechanical Scrubbing (MS) and Sodium silicate (SS) immersion

201 A two-stage treatment procedure for CRA was incorporated as indicated in figure 3<sup>44</sup>. Ten  
202 kg of processed RA are placed inside the Los Angeles testing equipment, which is then  
203 charged and rotated at 33 revolutions per minute (rpm) for 17 minutes. With absence of M.S.  
204 balls, rotation was allowed to take place. The impact resulted in the loosening of detached  
205 mortar that was sticking to the aggregate surface. When the spinning period ended, the  
206 aggregates were sieved, and the 12.5 mm retained aggregates were gathered. The selected  
207 mechanical treatment ensures that the treated CRA has homogeneous characteristics and  
208 removes any remaining cementitious mortar. The second stage of treatment comprise  
209 immersion of CRA in sodium silicate solution that initiates with manual water cleaning of  
210 CRA followed by engrossed in 20% Na<sub>2</sub>SiO<sub>3</sub> solution then assorted for 1 hour. The  
211 aggregates were then air dried for 24 hours.

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### 214 2.3 Physical performance of CRA

215 All treated CRA(s) were assessed for their specific gravity, water absorption, bulk density  
216 in accordance with IS 2386-Part 3, aggregate crushing value, aggregate impact value in  
217 accordance with IS 2386-Part 4, and aggregate abrasion value. Additionally, the IS 383  
218 specifications for coarse aggregate were contrasted with the physical properties of CRA. To  
219 compare the effectiveness of single stage and two stage treatment on CRA, comparative  
220 analysis is performed. Figure 4 reveals the grading curve of natural aggregate and CRA and  
221 it can be detected that CRA follows the IS provisions for particle size ranging 10-16 mm  
222 whereas some deviation is noticed for other size fractions.

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## 226 2.4 Microstructure studies

227 SEM, EDAX, FTIR, XRD, and TG-DTA were used to study the microstructure of various  
228 aggregate samples. To prepare samples for the examination of the aforementioned  
229 investigations, natural aggregate, untreated CRA, and treated CRA were all collected,  
230 crushed, and sieved. To determine the change in chemical composition inside the picture  
231 border, elemental analysis was carried out using an EDAX analyzer. Images were taken  
232 using a scanning electron microscope (GEMINI 300, Carl Zeiss, Resolution: 0.7 nm @15  
233 kV, 1.2 nm @1 kV). Malvern PANalytical, with a deflection angle range of 4 to 80 and a  
234 scanning speed of 2/min, is used to do the XRD analysis at Central Research Facility (CRF),  
235 NITK. After that, the patterns were examined using the X'Pert High Score Plus software. A  
236 Bruker (Alpha II) instrument with a  $2\text{ cm}^{-1}$  resolution and a  $4,000\text{--}500\text{ cm}^{-1}$  wavenumber  
237 range was used for the FTIR analysis. TGA was done using a Rigaku TG-DTA 8122  
238 TG/DTA analyzer. Samples were put into the analyzer and heated at a frequency of  
239  $10^\circ\text{C}/\text{min}$  in a nitrogen purge atmosphere (purge rate:  $10\text{ mL}/\text{min}$ ) between  $25$  and  $900^\circ\text{C}$ .

## 240 3. Results and discussions

### 241 3.1 Physical properties of treated RA

#### 242 3.1.1 Specific gravity and bulk density

243 Figure 5 illustrates the specific gravity of treated and untreated CRA (UCRA), along with  
244 natural aggregates. It is apparent that HCL\_CRA and MS+SS\_CRA has slightly higher  
245 specific gravity than UCRA, while SS\_CRA shows slightly lower values to that of UCRA.  
246 Further, figure 6 represents the bulk density measurements of single and two stage treated  
247 CRA and it can be observed that treated CRA has higher bulk density as compared to UCRA  
248 but shorter value than natural aggregate.

249 The increase in specific gravity may be ascribed to the omission of adhering mortar from the  
250 HCL and the subsequent mechanical scrubbing treatment, that is characterized by its weak  
251 and porous nature. Moreover, by changing adherent mortar to CSH, sodium silicate solution  
252 improves the recycled aggregates. The foregoing finding is further supported by the increase  
253 in bulk density, as specific gravity indicates how dense an aggregate is. The thicker layer of  
254 sodium silicate that makes the aggregate denser is most likely the cause of the treated CRA's  
255 increased bulk density. Additionally, the development of CSH fractions leads to the  
256 development of a dense microstructure, which is confirmed by the SEM findings in this  
257 study. This inference agrees with one of research <sup>38</sup> which examined the impact of sodium  
258 silicate as a surface treatment approach for the creation of RA-infused self-compacting  
259 concrete. Since specific gravity and bulk density data show that single- and two-stage treated

260 CRA are of higher quality than UCRA, IS 383 <sup>46</sup> advises using dense aggregates in  
261 construction projects.

### 262 3.1.2 Water absorption

263 Figure 5 illustrates the water absorption of natural aggregate, UCRA, and treated aggregate.  
264 After treatment, a noteworthy fall is observed in the water absorption values of CRA.  
265 Furthermore, the difference of water absorption values between treated CRA and natural  
266 aggregate is minimal. This outcome may be attributed to the elimination of remnant mortar  
267 due to HCL immersion as the water absorption is lowest in case of HCL\_CRA. Moreover,  
268 the formation of dense coating of sodium silicate solution have clogged the pores on the  
269 surface of CRA. Calcium hydroxide and sodium silicate solution react to generate the water-  
270 based silicate gel (CSH gel), which is a solid matrix.

271 This inference is consistent with research findings from a study <sup>47</sup> which showed that the RA  
272 treated with HCL solution saw a decrease in water absorption levels, especially when  
273 compared to the UCRA. Pre-wetting is not necessary when RA has water absorption values  
274 less than 5%, according to IS 383 <sup>46</sup>, henceforth, pre-wetting is no longer necessary when  
275 using single or two stage treated CRA in concrete applications.

### 276 3.1.3 Aggregate crushing value, impact value and abrasion value

277 Abrasion, crushing, and impact resistance tests are typically used to determine the excellence  
278 of the aggregate and its ability to withstand failure due to handling and mixing <sup>36</sup>. Figure 7  
279 displays the aggregate parameters for different CRA specimens in addition to the control  
280 aggregate. It is detected that two-stage treated CRA has somewhat better values than NA for  
281 crushing, impact, and abrasion than UCRA. It is clear from this observation that two stages  
282 of treatment are preferable to one stage for CRA. The observed pattern could be attributed  
283 to the deterioration and removal of older cementitious residue following mechanical  
284 cleaning. The Los Angeles abrasion of recycled aggregate (RA) had a significant positive  
285 impact in terms of the elimination of the aged cementitious remains from the surface of the  
286 RA particles as a consequence of abrasive motion. The aged mortar present on the surface  
287 of RA exhibits micro-cracks and pores, which result in a porous and weak interfacial zone  
288 (ITZ) between the surface of RA and the new mortar. By extracting the aged mortar from  
289 the surface of RA, the quality of the interfacial transition zone (ITZ) is improved <sup>48</sup>. Applying  
290 sodium silicate also improves the performance of the aggregates in terms of crushing,  
291 impact, and abrasion value by closing the holes and microcracks inside the aggregate. This  
292 study is consistent with observation <sup>49</sup> which shows that adding sodium silicate treatment  
293 reduces the aggregate crushing value.

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## 3.2. Microstructural studies

### 3.2.1 Scanning electron microscopy (SEM)

The SEM descriptions with relative magnifications for UCRA, HCL\_CRA, SS\_CRA and MS+SS\_CRA are shown in the Figs. 8 (a)-(d) respectively. It can be detected that URAC specimen shows irregular and non-uniform surface with large number of voids and minimum to nil occurrence of favorable hydration products. The occurrence of remnant cementitious fractions on the CRA surface, which resulted to the development of a porous and delicate microstructure, is responsible for this discovery. When it comes to treated CRA specimens like HCL\_CRA, SS\_CRA, and MS+SS\_CRA, CSH is more common and there are rare cracks that are the result of mechanical abrasion cycles. A supplementary CSH is created as a consequence of the reaction between adhering mortar and sodium silicate, especially for SS\_CRA and MS+SS\_CRA specimens, which results in a dense and uniform surface coating. However, for HCL\_CRA, an uneven surface morphology is illustrated due to the strong acidic nature of hydrochloric acid. Furthermore, the surface is found to show substantial damage as a consequence of adverse acid attack owing to HCL treatment.

### 318 3.2.2 Energy dispersive X-ray spectroscopy (EDAX)

319 The EDAX investigation of UCRA, HCL\_CRA, SS\_CRA and MS+SS\_CRA are shown in  
320 the figures 9(a)-(d) respectively. To quantify the elemental composition, area to point  
321 analysis is performed for each treated CRA and UCRA sample. Based on results from the  
322 EDAX analysis, Figure 10 shows the calculated atomic weight ratios of calcium to silicate  
323 as well as a comprehensive elemental configuration. It is commonly known that for dense  
324 microstructure, the Ca/Si ratio usually goes below 2<sup>4</sup>. Further, the Al/Ca atomic ratio is  
325 computed from EDAX data using comparable computations to the Ca/Si atomic ratio for  
326 various treatment strategies. This variation is shown in figure 11. According to a study, the  
327 Al/Ca ratio has an influence on the amount of CH crystals and with increasing Al/Ca ratio,  
328 the CH crystals decreases and results in dense microstructure<sup>4</sup>. It can be observed that the  
329 Ca/Si atomic ratios of all the samples are below 2, indicating a dense microstructure. This  
330 could be related to the right processing and treatment strategy that promoted increased CSH  
331 production. This is probably a good reason to use a multi-cycle method that can convert  
332 CH crystals into CSH, which reinforces the microstructure even more. It is to be noted that  
333 for UCRA, Ca/Si ratio is lower than two stage treatment but higher than single stage  
334 treatment, whereas Al/Ca ratio is higher than two stage treatment and lower than single  
335 stage treatment. This may be accredited to the fact that multi cycle processing approach  
336 has removed a substantial volume of adhered old mortar that has further densified the  
337 microstructure. However, with two stage treatment that initiates with mechanical  
338 scrubbing, the CRA specimen is again subjected to repeated abrasion cycles and that has  
339 created some microcracks that can be observed in SEM image of the sample (Figure 8 (d)).  
340 These microcracks created rare void spaces and the same is reflected in the form of slightly  
341 higher Ca/Si ratio and lower Al/Ca ratio.

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### 346 3.2.3 Xray diffraction (XRD)

347 The XRD peaks of UCRA, HCL\_CRA, SS\_CRA and MS+SS\_CRA are shown in the figure  
348 11. It can be detected that calcium silicate hydroxide were visible in the XRD pattern for the  
349 treated CRA samples. The highest intensity in the CSH peaks is detected for two stage treated  
350 CRA (MS+SS\_CRA) with respect to single stage treated CRA (HCL\_CRA, SS\_CRA). The

351 strong peaks in the URCA are detected at an angle of 26.46, 20.71 (Quartz, SiO<sub>2</sub>), 27.76°,  
352 49.99° (Calcite, CaCO<sub>3</sub>), 29.425° (unhydrated CSH). XRD peaks of HCL\_CRA and  
353 MS+SS\_CRA established an exaggerated peak of Calcium silicate hydroxide (27.78°) as  
354 compared to UCRA. However, SS\_CRA showed a comparatively fewer intense CSH peak  
355 (27.78°) with respect to other treated CRA specimen but still the CSH peak intensity is 1.55  
356 times the CSH peak intensity of UCRA. This conclusion agrees with the findings of SEM  
357 analysis for SS based treatment about the formation of auxiliary CSH. Henceforth, the XRD  
358 findings are giving clear evidence that two stage treatment is superior than single stage  
359 treatment approaches.

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### 362 3.2.4 Fourier-transform infrared spectroscopy (FTIR)

363 The FTIR spectra of NA, HCL\_CRA, SS\_CRA and MS+SS\_CRA specimens is illustrated  
364 in figure 12. In treated CRA samples, it was discovered that the main peak at wavelength  
365  $3640\text{ cm}^{-1}$ , that confirms to the O-H stretching bond of portlandite, was absent. According  
366 to a study <sup>50</sup> based on FTIR analysis on RA, this observation was made. This has been  
367 accredited to the phases of calcium carbonate that were created when portlandite and ambient  
368 carbon dioxide reacted. The additional water molecules in the specimens instigated the  
369 stretching vibration of O-H amid  $1600$  and  $1660\text{ cm}^{-1}$ <sup>50,51</sup>. In addition, the peak interval  
370 between  $1450\text{--}1470\text{ cm}^{-1}$ ,  $1000\text{--}950\text{ cm}^{-1}$ ,  $780\text{--}770\text{ cm}^{-1}$  and  $595\text{--}570\text{ cm}^{-1}$  resembles to the  
371 asymmetric stretching vibration of C-O bond, Si-O bond, symmetric stretching vibration of  
372 Si-O bond and unhydrated cement respectively. The inclusion of SS\_CRA and HCL\_CRA  
373 progressively weakens the distinctive peaks at  $975\text{ cm}^{-1}$ ,  $773\text{ cm}^{-1}$ , and  $585\text{ cm}^{-1}$ , indicating  
374 that the calcite and CH were absorbed in the interaction between the CRA, sodium silicate,  
375 and HCL. The calcium-HCL and calcium-sodium silicate complex can be produced by the  
376 CRA's calcite and CH reacting with sodium silicate and HCL, according to the findings of  
377 the FTIR study.

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380 3.2.5 Thermogravimetry analysis (TGA)

381 The thermogravimetry (TG) curvature demonstrates the presence of thermogravimetric mass  
382 loss during the heating evolution from 25°C to 900°C, while the derived of  
383 thermogravimetry (DTG) curve displays the temperature borders for the breakdown of  
384 certain compounds.

385 Figure 13 shows several endothermic peaks in the specified temperature range. The  
386 temperature limits that correspond to the loss of free water molecules, ettringites, and  
387 gypsum are 25–50 °C, 50–120 °C, and 120–150 °C, respectively. These ranges can be  
388 additionally divided from the main endothermic peak up to 200 °C. Between 400 and 500  
389 °C is the temperature range at which calcium hydroxide (CH) is dehydroxylated; at this  
390 point, a second notable endothermic peak was seen. The third notable peak results from the  
391 decarbonisation of CaCO<sub>3</sub> (CC) at temperatures between 600 and 800 °C<sup>4</sup>.

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394 It should be noted that the following equations—equations 2, 3, and 4 at specific temperature  
395 boundaries—are considered in this inquiry in order to control the reduction in mass  
396 proportion from TG-DTG. Figure 14 illustrates computed results for various chemical  
397 elements from TGA analysis. The percentage of decomposed calcium hydroxide (CH%) is  
398 represented by the symbols in the following equation, whereas the percentages of bound  
399 water and calcium carbonate that have formed. M<sub>CH</sub>, M<sub>H<sub>2</sub>O</sub>, M<sub>CC</sub> and M<sub>CO<sub>2</sub></sub> represents the  
400 molecular weight of calcium hydroxide, water, calcium carbonate and carbon dioxide  
401 respectively. The remaining symbols, %W<sub>CH</sub> and % W<sub>CC</sub> represents the weight loss of  
402 calcium hydroxide and calcium carbonate respectively.

403 
$$CH\% = (\%W_{CH}) \times \left(\frac{M_{CH}}{M_{H_2O}}\right) = (\%W_{CH} \times \frac{74}{18}) \quad (2)$$

404 
$$W_n\% = W_T - W_{CH} \quad (3)$$

405 
$$CC\% = (\%W_{CC}) \times \left(\frac{M_{CC}}{M_{CO_2}}\right) = (\%W_{CC} \times \frac{100}{44}) \quad (4)$$

406

407

408 It should be noted that the formation of stable CSH crystals as a result of triggered chemical  
409 reaction between CH phase with sodium silicate and HCL is indicated by the minor  
410 occurrence of the CH crystals for the HCL and mechanical scrubbing tracked by sodium  
411 silicate-based treatment approaches for CRA specimens. However, the CH crystals are

412 slightly higher in the case of the single-stage SS (sodium silicate) treatment method that  
413 further indicates an incomplete utilization of the CH crystals and lower volumes of CSH  
414 segments at microstructure stages. This might be owing to the reduction in the old  
415 cementitious remains in CRA samples due to multi cycle processing technology. As a whole,  
416 the TG-DTA analysis indicates that treatment techniques adopted for this experimental  
417 investigation forms favorable hydration products at microstructure level.

#### 418 **4. Cost Estimation**

419 In order to calculate the cost of generating the various processed and treated CRA specimens  
420 (UCRA, HCL\_CRA, SS\_CRA, and MS+SS\_CRA) taken into consideration in the present  
421 investigation, the elemental prices of all the materials were acquired from national and  
422 intercontinental marketplaces. The expenditure on the energy used by the corresponding  
423 machinery was calculated in order to account for the expenses associated with the multicycle  
424 jaw crushing and Los Angeles test machine. Table 2 displays the cost per metric tonne of  
425 sodium silicate and hydrochloric acid, given in Indian Rupees (Rs). In addition, the cost  
426 involved for producing various treated and processed CRA samples with respect to  
427 processed samples is also evaluated for distinguishing the induced cost among various single  
428 and multi-stage treatment methodologies.

429 As shown by the data in Table 2 particularly for treated CRA, the total cost induced is found  
430 out to be least for HCL\_CRA followed by SS\_CRA and MS+SS\_CRA. In comparison to  
431 untreated CRA, MS+SS\_CRA is observed to be most expansive treatment method. Even  
432 though it is estimated that two stage treatment is costly, the microstructure studies revealed  
433 that the same treatment method is found to show additional CSH alongside uniform surface  
434 characteristics with negligible void percentage. The supplementary formation of CSH is an  
435 encouraging feature for potential utilization of MS+SS\_CRA in concrete applications for  
436 satisfactory mechanical and durability performance, which will reduce the overall cost.

TABLE 2 Cost estimation for various treated CRA samples (Expressed in INR)

Particulars	Unit Cost (INR/ton)	UCRA	HCL_CRA	SS_CRA	MS+SS_CRA
Processing cycles	1550	155	155	155	155
HCL acid	1750	--	582	--	--
Sodium silicate	13500	--	--	1125	--
Mechanical scrubbing (INR/kWh)	240	--	--	--	1365
Total cost (INR)		155	737	1280	1520
Changes in Cost / UCRA sample		--	3.75 times higher cost	7.25 times higher cost	8.80 times higher cost
Remarks			Economical treatment	Moderate economic treatment	Expansive treatment

439 **Conclusion**

440 For the multi-cycle processed CRA, the current investigation examines the viability of single  
441 and two stage treatment approaches. The initial processing of demolished concrete debris  
442 involved multiple crushing cycles using a jaw crusher. This was tracked by single and two  
443 stage treatment approaches, such as immersion in a solution of sodium silicate and  
444 hydrochloric acid. Los Angeles abrasion and sodium silicate immersion were the two stages  
445 of the treatment for the processed CRA. Along with microstructural analyses using  
446 techniques including SEM, EDAX, XRD, FTIR, and TG-DTA, the practicality of processing  
447 and treatment methods was evaluated in terms of the physical attributes of the CRA. The  
448 research work's findings allow for the following interpretations to be made.

- 449 1. The multi cycle processing of CDW yields superior quality CRA that can be further  
450 used in the concrete mix.
- 451 2. The removal of the mortar component or surface coating methods to strengthen the  
452 adhering mortar are two ways to treat CRA, and it can be suggested that the latter  
453 method results in CRA of higher quality.
- 454 3. HCL treated CRA lowered the water absorption values by 78% and enhanced its  
455 specific gravity by 6% to that of UCRA.
- 456 4. MS+SS\_CRA specimens are found to show 6% additional bulk density, 30% higher  
457 impact resistance, 42% higher crushing resistance as compared to that of UCRA  
458 specimen.
- 459 5. FTIR results demonstrated that the treated CRA with sodium silicate and hydrochloric  
460 acid produces additional amount of hydration products.
- 461 6. TGA analysis proved that mechanical scrubbed followed by sodium silicate treated  
462 CRA induced the formation of stable CSH gel at microstructure level.
- 463 7. XRD study found highly intensified CSH peaks for mechanical scrubbed followed  
464 by sodium silicate treated CRA with respect to UCRA.
- 465 8. SEM images illustrated that treatment through sodium silicate provides uniform and  
466 compact surface characteristics by eliminating significant volume of pores, whereas,  
467 mechanical scrubbing followed by sodium silicate treatment results in the formation  
468 of CSH gel with partial cracking.
- 469 9. EDAX analysis revealed that the SS\_CRA samples and HCL\_CRA specimens had  
470 the lowest Ca/Si atomic ratios, which further suggests a dense microstructure, by  
471 81%.

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690

691 **Figure caption**

692 Fig. 1. C&D waste statistics across Indian states (Yellow-States with major  
693 waste, Grey-States with minor waste)

694 Fig. 2. Multi cycle processing technology for procuring CRA

695 Fig. 3. Two stage treatment process for CRA

696 Fig. 4. Grading Curve of NA and CRA

697 Fig. 5. Specific gravity and water absorption of CRA samples

698 Fig. 6. Bulk density of single and two stage treated CRA

699 Fig. 7. Various aggregate parameters for different CRA specimens

700 Fig. 8. SEM images of (a) UCRA (4.5K X), (b) HCL\_CRA (500X), (c)

701 SS\_CRA (3K X), (d) MS+SS\_CRA (9K X)

702 Fig. 9. EDAX images of (a) UCRA (b) HCL\_CRA (c) SS\_CRA (d)

703 MS+SS\_CRA

704 Fig. 10. Ca/Si and Al/Ca atomic ratio of CRA specimens

705 Fig. 11. XRD peaks of various treated and untreated CRA specimens

706 Fig. 12. Different peaks corresponding to FTIR analysis of CRA specimens

707 Fig. 13. TG analysis of CRA samples

708 Fig. 14. Quantification of chemical parameters for CRA samples

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711