

Mechanical and Durability Performance of Calcium and Silica-Rich Engineered Char in Recycled Aggregate Concrete Composites: A Comprehensive and Long-Term Curing Evaluation

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Abstract

This research work is devoted to understanding the behaviour of calcium and silica-rich char in recycled aggregate concrete composites. Although, the determination of absolute contribution by each type is one of the strenuous tasks to accomplish, however, the overall perspective in this area is presented for their mechanical and durability performance. Calcium rich engineered wood char produced higher strength performance than silica rich char. The long-term curing behaviour up to 56 days revealed that mechanical performance is slightly varied in contrast to 28 days. Similar behaviour was seen in flexural strength, water absorption and voids in which slight change was observed than control samples. Further in-depth research is required to determine the potential of engineered char in recycled aggregate concrete composites.

1. Introduction

Concrete footprint starts from the raw materials extraction till the distribution of concrete waste to the landfill sites, however, production of cement contains a significant role in carbon emissions into the atmosphere than the rest of the processes [1]. Concrete importance and varieties of applications make it near to impossible to reduce or even sustain at a certain level. The eventual growth of any area or country leads to further production of concrete and results in more carbon emissions. Cement industry alone is responsible for nearly 7% of total CO₂ emissions into the environment [2]. The supplementary cementitious materials tend to have the ability to partially replace the cement content while maintaining similar properties [2,3]. On the other hand, significant research is being done on geopolymer concrete to eliminate the cement content, considering fly ash as the major binder and produce the concrete of lower carbon footprint. Other binders also being investigated including blast furnace slag, metakaolin and their combination with the help of alkali activator [5].

Although the contribution of geopolymer concrete contains significant advantages, however, the limited availability of raw materials is also of concern. Thus, the suitable approach is to consider the introduction of modern pozzolanic materials in concrete which can reduce the cement content as well as maintain the consistent supply of raw materials. One of the recently recognised approaches is to consider the organic waste derived char obtained from different sources (poultry litter, forest, crop residues and industrial waste etc.). Char was reported to use in mortar and concrete composites and found to improve the properties according to their quantity and application. Akhtar et al., [6] reported an increase in flexural strength in conventional concrete and at another instant found to improve the overall properties of concrete when engineered for concrete applications. Char also tend to improve the properties of recycled aggregate concrete at several instants, improving the mechanical strength and reduce water absorption [7]. Filler effect of char is dominant when used without treatment, however, can as well hold pozzolanic and cementitious properties according to the processing during the pyrolysis process.

Cement is not alone to play the role of CO₂ emissions; aggregates are the other factor which is responsible for increasing CO₂ emissions. Although the contribution of CO₂ emissions by aggregates are minimal as compared to cement, however, while designing the project, the sustainability of the raw material is also needed to be considered. The consistent supply of aggregates to the concrete industry will eventually lead to the scarcity of the materials and thus requires to consider other possibilities to replace the concerned material. One of the

approaches is to reuse the demolished concrete which can be processed to reproduce the concrete of lower or similar quality. Many studies have been reported on the use of recycled aggregates (RA) in concrete production and reported positive results. Salesa et al., [8] presented a comprehensive review on the use of the multi recycled concrete produced from the pre-cast concrete industry. The use of two cycles of recycling was concluded to be optimal to maintain the strength of concrete, however, workability and bulk density is reduced. Huda et al., [9] reported the decrease in overall strength properties with the addition of three generations recycled aggregate concrete (RAC). However, the design strength could be reached at the age of 56 days.

The resultant properties of the recycled concrete usually depend on the properties of original concrete whose mortar is attached to the surface of aggregates. Aggregates derived from high strength concrete generally provides the possibility to produce normal strength concrete without significant impact on strength properties [2]. While RA utilisation brings some drawbacks regarding ultimate properties, however, it is noteworthy that environmental implications must be considered. Not to mention the fact that there are limited natural reserves available for aggregate extraction. Wijayasundara et al., [10] have recently presented the indirect external benefits to produce recycled aggregate concrete. It was concluded that 1 tonne of RA utilisation rather than NA, could prevent in 1.56 tonnes of waste landfill disposal with the transportation of the waste to landfill sites and a similar amount of NA extraction could also be prevented. Thus, there is a clear advantage from the environmental point of view, whereas, further work is necessary regarding the improvement of its properties through conventional as well as modern techniques.

The new materials are required which can enhance the overall properties and chemical structure of RAC with less burden on the environment. In this scenario, char is the ideal option which not only has the potential to reduce the use of raw materials but also can improve the properties when engineered for RAC production. Char produces properties of varying attributes and contain the potential to further change post production [11][12]. Therefore, this study presents the use of silica-rich (rice husk) and calcium-rich (wood chips combined with lime) char in recycled aggregate concrete and contributes towards the understanding of the potential to influence the properties of recycled aggregate concrete from both ends of the properties.

2. Materials and Methods

2.1 Materials

The following materials are used for the production of char concrete composites including cement, sand, recycled aggregates (9.5-19.0 mm), water reducer, calcium-rich wood char, silicon rich rice husk char and water. Calcium-rich char was produced from hardwood feedstock by prior mixing of wood chips with five wt. % of calcium hydroxide in water. The water was let it soaked into wood chips and then later pyrolysed at 650 °C for a residence time of 1 hour, and rice husk char was pyrolysed by Oliver Enterprises Philippines at 500 °C [7]. The properties of ordinary Portland cement can be referred at Akhtar et al., [6] and properties of rice husk char and calcium-rich wood char and recycled aggregates can be referred at Akhtar et al., [7]. The particle size distribution of both types of char was done by Malvern Mastersizer 3000 laser diffraction particle analyser with hydro G dispersion. The particle size of both types of char is given in Table 1.

2.2 Methods

Mix ratios were designed such that to replace the cement content with char up to 3% of total binder content based on weight. Both chars were investigated at 1% and 3% of total binder content for mechanical strength and water absorption potential (Table 2). Mixing of all the materials was followed by two-stage mixing approach as explained by Tam et al., [13] and followed by casting of the cylinders and beam samples. Cylindrical and beam specimens of dimensions (100 × 200 mm) and (100 × 100 × 500 mm) were cast respectively. All the samples were cast and cured according to the ASTM C192/192M [14] standard and tested for compressive and splitting tensile strength according to the ASTM C39/C39M [15] and ASTM C496/C496M [16] respectively. All the cylinders were capped before the compressive strength testing according to the ASTM C617/C617M [17] standard. Likewise, the flexural strength of the beam specimens was conducted according to the ASTM C78/C78M [18].

A number of separate samples were cast to determine the water absorption, and permeable voids in char added to concrete. These samples were tested according to the ASTM C642 [19] standard. Water absorption test was conducted after 56 days of curing in a humidity chamber maintained the temperature at 20 °C and 100% relative humidity. Concrete was characterized through the use of FTIR and XRD analysis after reaching 28 days to

complement the strength and durability results. The functional groups present in concrete were investigated by ATR-FTIR and spectra were collected in the range of 700 – 4000 cm^{-1} wavenumbers. The spectral resolution was kept constant at 4 cm^{-1} and scan rate of 100. Similarly, phase identification of concrete samples was done at a range angle 10 – 60 degrees with a step size of 0.02 at 0.1s per step. The current and voltage were kept constant at 30 mA and 40 kV.

3. Results and discussion

3.1 Compressive strength

The compressive strength of the cylindrical samples was tested after 7, 28 and 56 days of water curing in a humidity chamber at controlled conditions to determine the pattern of strength change with the addition of char. It can be seen from Figure 1 that all the samples showed high compressive strength at the age of 7 days than Control samples containing no char. On average, more than 10% increment in strength observed in R1C, R3C, R1S and R3S. R3S showed the highest improvement with 15% increase than Control samples. Most of the samples also showed significant improvement at the age of 28 days showing more than 8% improvement except R3C samples according to the Control samples. An average of 8.9 %, 9.2 % and 12.1% increment were observed in R1C, R1S and R3S samples respectively. R3C samples also exhibited nearly similar results to the Control with less than 1 MPa of difference between their values. Muduli et al., [20] reported an increase in strength performance with the addition of metakaolin in recycled aggregate concrete, however, 100% recycled aggregate concrete showed lower strength performance than natural aggregate concrete.

After the 56 days of curing in a fog room, strength pattern changes altogether. Although all the samples showed an increment in strength, however, R1C and R3S showed a relatively significant improvement. The average increase in strength was 15.4 % and 6.9 % exhibited by R1C and R3S samples respectively. There is a slight increase in strength by R3C and slight decrease by R1S samples with less than 0.25 MPa of a difference than Control samples. The strength change is quite significant as compared to previously reported studies [7] [6]. In higher replacement percentages, even though the strength increase is noteworthy at seven days, however, the difference reduces with the time. This might be due to early maturity in the strength attainment as compared to control samples.

3.2 Splitting tensile strength

The splitting tensile strength of the samples exhibited a contrasting behaviour in which most of the samples showed a reduction in strength at the age of 7 days (Figure 2). The R1C samples slightly improved the strength by 4.9% according to Control samples. Samples at 28 days changed the strength trend altogether, aligning with compressive strength values and improved the values for all of the samples. The improvement was recorded from 4-8% on average in all samples. Other studies also recorded an improvement in strength performance upon addition of natural pozzolanic materials, however, the reported strength in 100% RAC is less than NAC [20][21]. The highest increase was observed by R3C and R3S samples, showing more than a 7% increase. Likewise, most of the samples at 56 days also showed improved strength performance except R3S samples. The calcium-rich char at 56 days performed better than silicon rich char where both of the additions showed more than 5% improvement in strength. This agrees with the previous study presented by Bui et al., [22] in which 3 % silica fume at the water to binder ratio 0.39 produced lower values at 28 days than the increased addition of silica fume. However, it showed better results with higher replacement ratios reaching the values up to natural aggregate concrete. Silicon-rich char improved the strength; however, these samples were only capable of maintaining the strength according to control samples with $\pm 1\%$ of difference among their values.

3.3 Statistical analysis

The statistical analysis of the combined compressive and splitting tensile strength at 7, 28 days, 7, 56 days and 28, 56 days was presented in Figure 3. All of the statistical analysis showed a high coefficient of determination $R^2 > 0.98$ showing the dependence of the variables with each other. The regression of 7 days and 28 days combined strength analysis showed an R^2 value of 0.994 with p-value $1.86E-21$, showing the significance of the correlation. Similarly, the regression of 28 days and 56 days combined strength showed the R^2 value 0.985 with p-value $6.59E-18 < 0.5$. Linear regression of 7 days and 56 days also showed similar R^2 value with the very low p-value. A very high coefficient of determination and sufficiently low p-value predicted the reliable correlation between all the testing days. The correlation coefficient was also found to be > 0.99 between all of the concerned variables.

3.4 Flexural Strength

The bending strength of the char added RAC composites was measured after 56 days of the curing. It can be seen that most of the specimens are of similar or higher strength than control samples except R3S (Figure 4). The difference between R3S and control sample was minor

and noted to be in the range of 0.05 MPa. The rest of the samples showed strength increment between 2 - 4% than control samples. There is less variability in silicon-rich char, however, calcium-rich char exhibited consistency in the strength values.

3.5 Density and Water Absorption

The average density of the hardened concrete at the age of 7 days and 56 days is presented in Figure 5 for compressive strength as well as splitting tensile strength specimens. The density of the char added samples shown to be slightly less than their respective control samples at seven days. A similar trend was observed for 56 days old specimens except for R1S which showed a slightly higher density. The water absorption potential and volume of permeable voids were calculated using the separate samples to measure the scope of the change in structural behaviour. Water absorption was reduced in most of the samples at the age of 28 days. The absorption behaviour was as follows $R1S > R3C > R3S > \text{control} > R1C$ (Table 3). Similarly, the volume of permeable voids was also reduced in all of the char added samples up to 1%. The change in voids were as follows $R1S > R3S > R3C > R1C > \text{control}$. It is likely that change in strength and other properties could occur with the time span and further add the value to the concrete.

3.6 Fourier Transform Infrared (FTIR) spectroscopy

The FTIR spectra of the calcium and silicon rich char added concrete is presented with a control sample in Figure 6. The peaks were presented generally in similar patterns with no shift, however some peaks are dominant in the char added concrete composites. The peaks appeared in the spectra in the following regions 781 cm^{-1} , 995 cm^{-1} , 2326 cm^{-1} , 2364 cm^{-1} . Two earlier peaks at 781 and 995 cm^{-1} can be assigned to Si – O – Si band and Si – O band vibration [23][24]. The appearance of peaks in the range of $2347 - 2370\text{ cm}^{-1}$ is attributed to CO_2 presence in samples [25] in char added concrete which is absent in control samples. Thus, it indicates that char added concrete helps in the absorption of CO_2 from atmosphere resulting in further carbon sequestration during its life time. The rice husk char appeared to absorb higher CO_2 than wood derived char.

3.7 XRD analysis

X-ray diffraction analysis of the char added concrete was done at the age of 7 days and 28 days to compare the phase change behaviour. There are three main phases detected (i) calcium hydroxide, (ii) calcium carbonate and (iii) quartz (Figure 7). Quartz peaks were quite

distinctive with calcium carbonate. Calcium-rich char added concrete had shown higher peaks than control samples for quartz at the age of 7 days. On the other hand, calcium carbonate peaks are substantially higher in silica-rich char concrete than the rest of the samples. When examined at the age of 28 days, quartz peaks were lower in all char added concrete samples than control. This might be due to the conversion of silica to calcium silicate hydrate phase, which has lower detection through diffraction analysis due to amorphous behaviour. Similarly, calcium hydroxide peaks which were quite distinctive at the age of 28 days in char added to concrete, turns approximately disappeared at the age of 28 days, giving the further evidence of the reaction with silica.

3.8 Embodied carbon dioxide emissions

The impact of the char on embodied carbon dioxide emissions in concrete production contains significant value to evaluate its environmental footprint. Carbon abatement potential from slow pyrolysis was found in the range of 0.07 to 1.25 tonnes of CO₂ eq.t⁻¹ of feedstock utilised through the technology [26]. Wood waste was found to contain highest carbon abatement potential due to high char conversion and slow pyrolysis is the most feasible technology than fast pyrolysis and gasification. In another study, the carbon abatement potential of rice husk char with the generation of electricity was in the range of 0.86 CO₂ eq.t⁻¹ of feedstock [27].

The effect of char on overall embodied emissions of RAC is shown in Figure 8. There is a clear decrease with the increase of char content up to 6% of total emissions in this scenario. The use of cement is the major driver of the increase of carbon emissions [28]. The use of rice husk and wood char both produced similar results at respective addition of 1 and 3% of total binder content. There is a 5% increase in carbon emissions assumed in wood char scenario due to calcium enrichment of char. Further engineering of the char is required to optimise the properties of concrete with higher replacement of binder in concrete. This may lead to additional reduction of embodied carbon emissions and will pave the way towards green concrete production.

4. Conclusion

Char comparison of two different origins and composition evaluated in detail in recycled aggregate concrete. It is clear from the obtained results that calcium-rich wood char improved the overall properties of the concrete, however, the difference is marginal than rice husk char. Both char types improved the compressive, splitting tensile and flexural strength of the

concrete samples. The increment in strength performance up to 28 days were impressive for silica-rich char, however, further development was minimal as compared to calcium-rich char. Further studies could be conducted to explore the area of calcium induction on rice husks surfaces prior to pyrolysis for their in-depth effect in concrete composites. It is likely that further engineering the rice husk char could potentially enhance the concrete properties.

5. References

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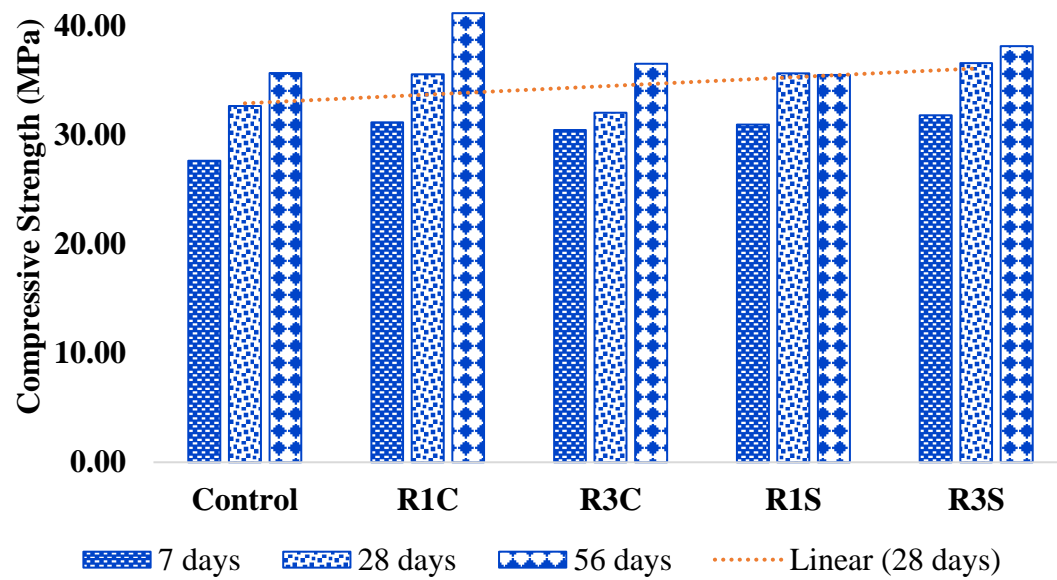


Figure 1 Compressive strength of recycled aggregate concrete with char addition of 1 and 3%

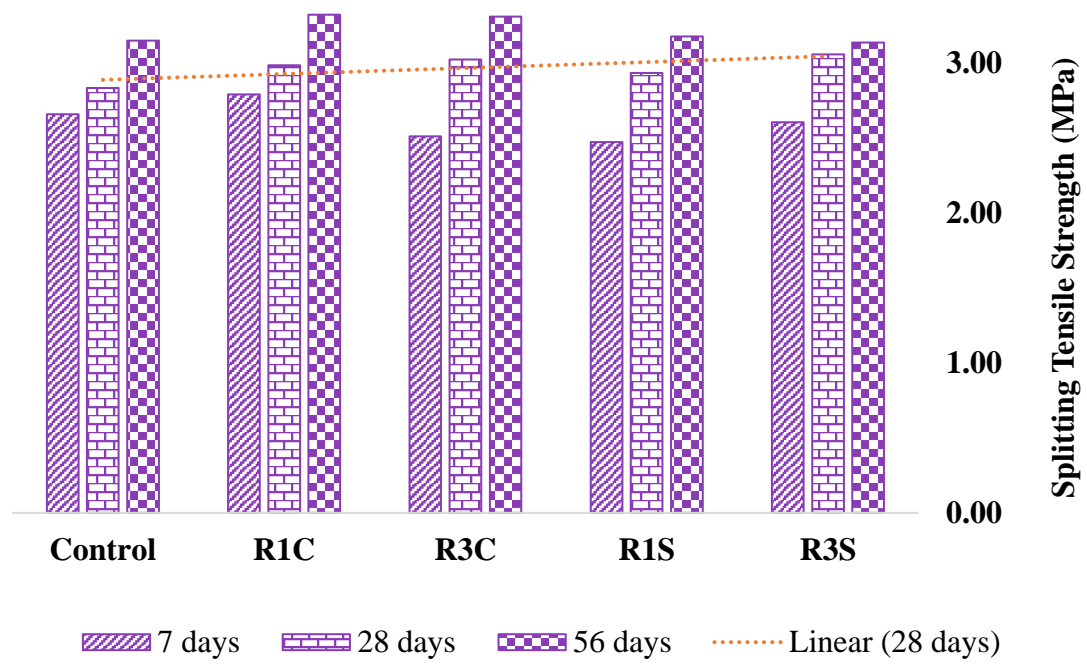


Figure 2 Splitting tensile strength of recycled aggregate concrete with 1 and 3% char addition

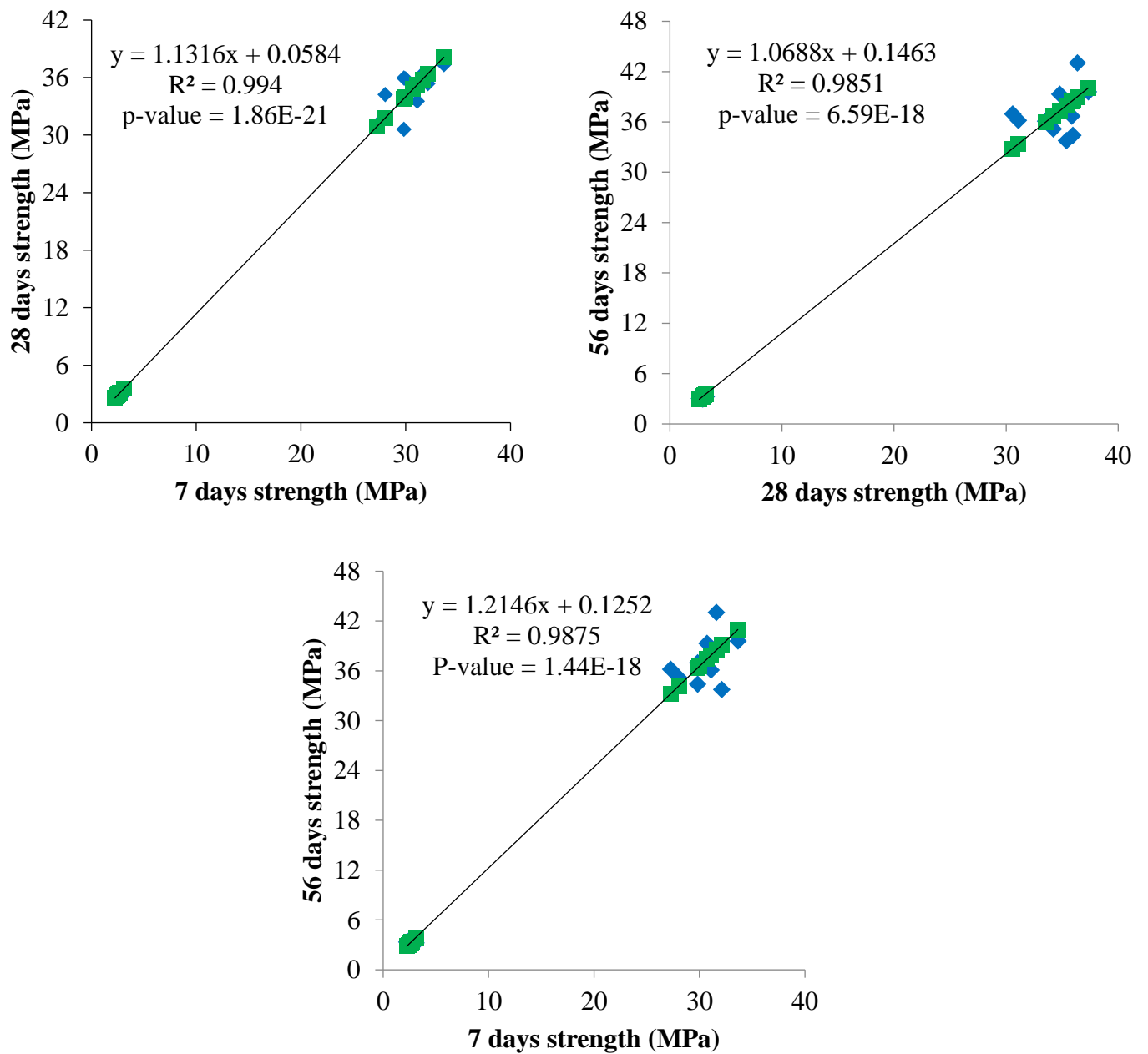


Figure 3 Statistical analysis of the overall strength comparison at the age of 7, 28 and 56 days

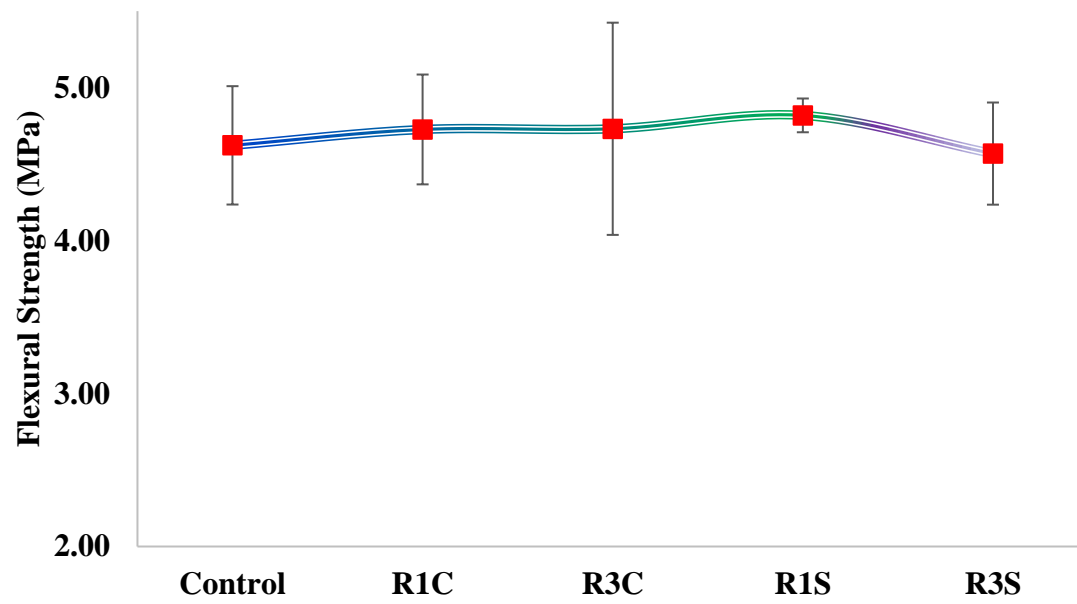


Figure 4 Flexural strength variation with char addition at the age of 56 days

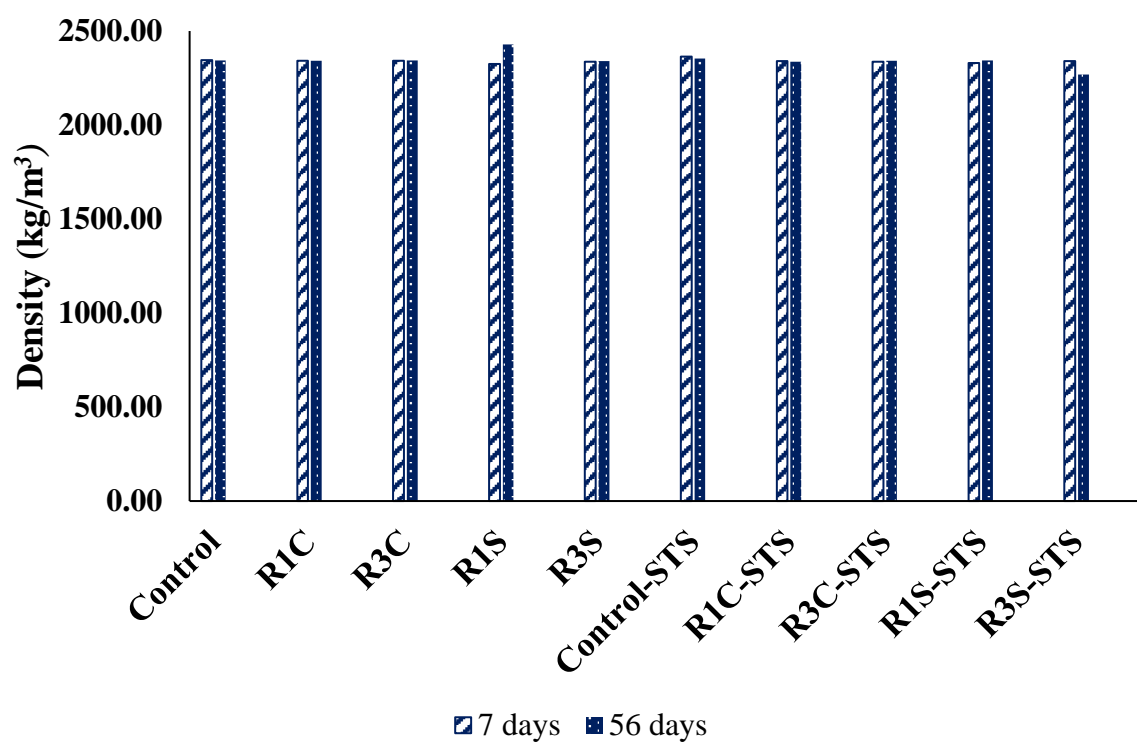


Figure 5 Density change from 7 days to 56 days of recycled aggregate concrete

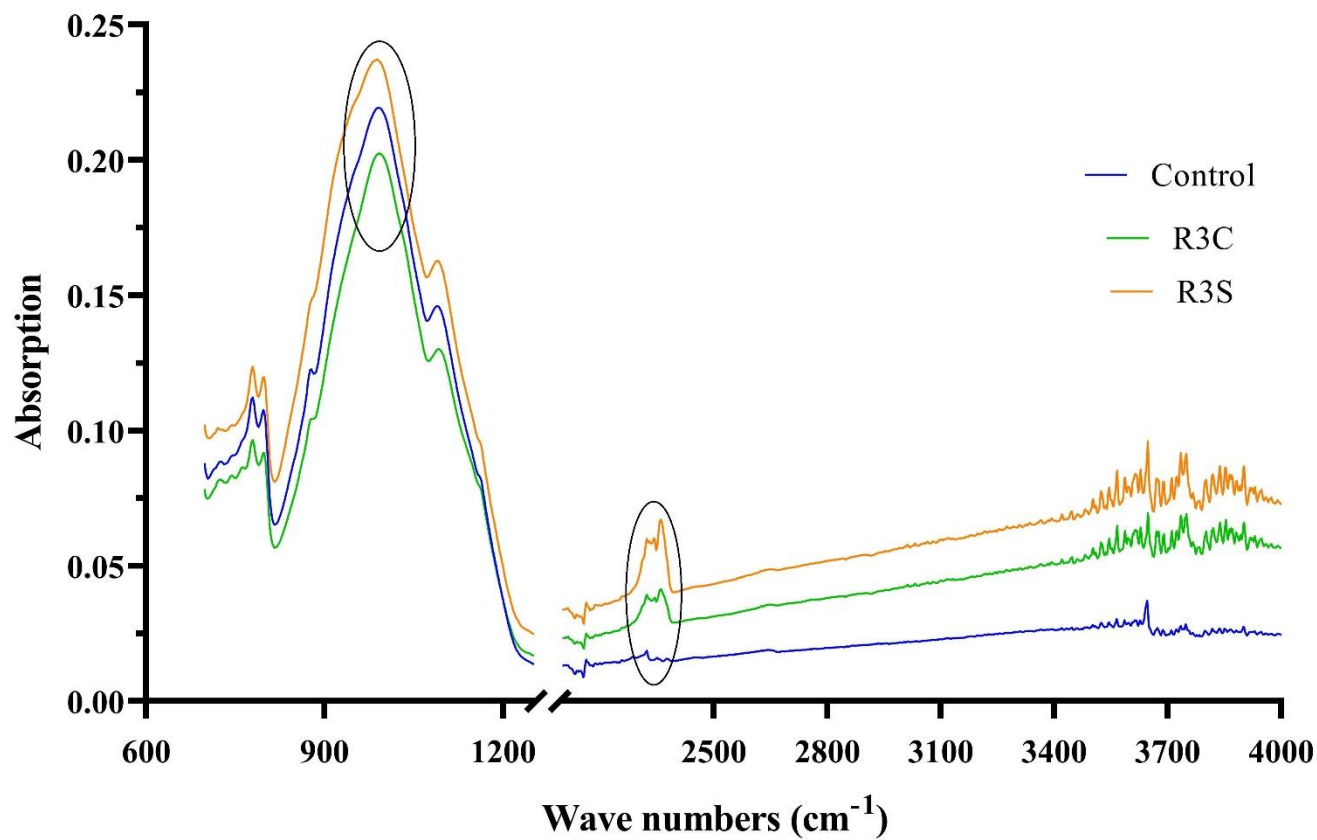


Figure 6 FTIR spectra of concrete at the age of 28 days

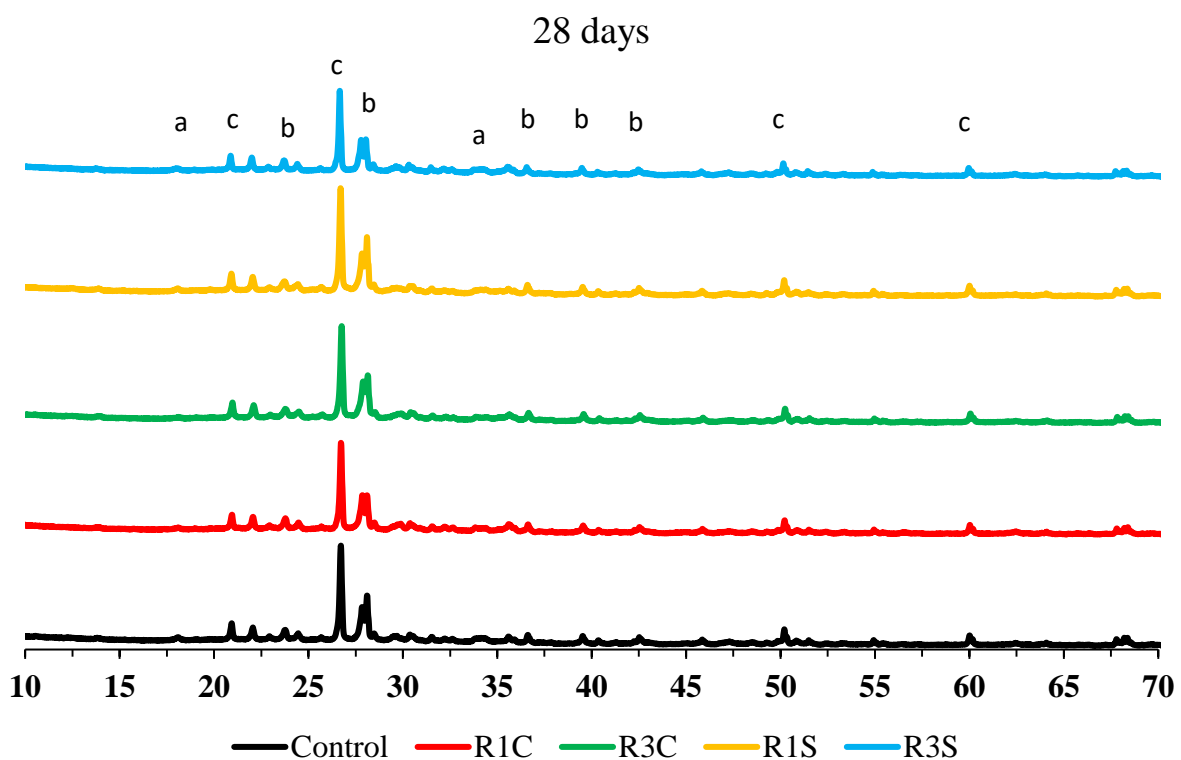
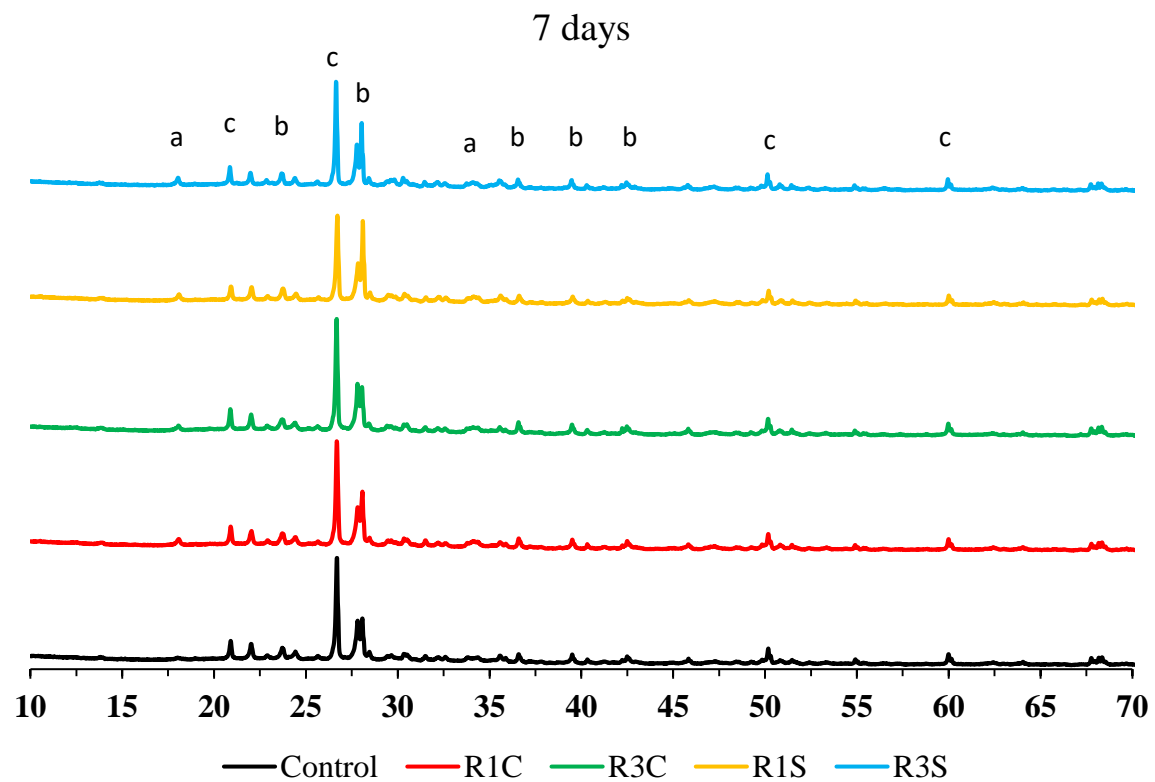


Figure 7 XRD analysis of the char added concrete composites at the age of 7 days and 28 days ($a = \text{Ca}(\text{OH})_2$, $b = \text{CaCO}_3$, $c = \text{quartz}$)

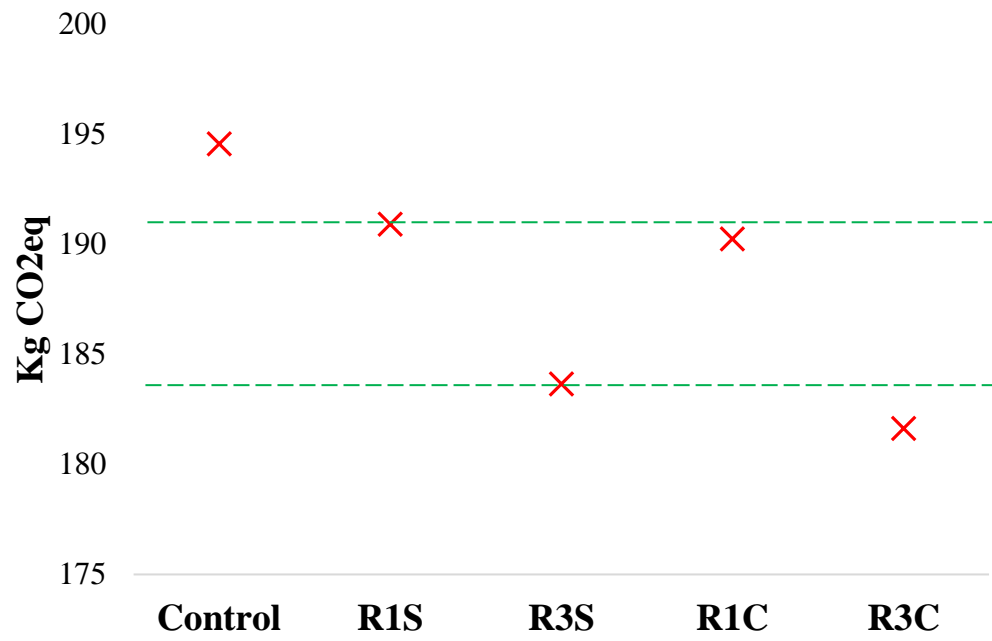


Figure 8 Embodied carbon dioxide emissions with the addition of rice husk and plain wood char at 1 and 3% of binder content

Table 1. Particle size distribution of wood char and rice husk char

Size (µm)	Wood char (%)	Rice husk char (%)
0.02-10	24.64	61.36
10-20	12.59	17
20-30	8.65	7.02
30-40	7.74	3.92
40-50	7.37	2.53
50-60	6.75	1.76
60-70	6.03	1.21
70-200	26.22	5.21

Table 2 Mix design of concrete for 1% and 3% addition of char in kg/m³

Samples	Cement	Sand	RA (9.5/19)	Wood Char	Rice Husk Char	Admixture	w/b
Control	450.4	822.52	917.99	-	-	6.12	0.37
R1C	445.9	822.52	917.99	4.53		6.12	0.37
R3C	436.9	822.52	917.99	13.53		6.12	0.37
R1S	445.9	822.52	917.99		4.53	6.12	0.37
R3S	436.9	822.52	917.99		13.53	6.12	0.37

Table 3 Water absorption and permeable voids of concrete

Samples	Water absorption (%)	Absorption after boiling (%)	Volume of permeable voids (%)
Control	6.60	7.12	15.84
R1C	6.92	7.34	15.82
R3C	6.18	6.73	14.72
R1S	6.12	6.62	14.56
R3S	6.24	6.54	14.71