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Compressive Strength and Resistance to Sodium Sulphate Attack of Concrete Incorporated with Fine Aggregate Recycled Ceramic Tiles

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ABSTRACT: In this experimental study, compressive strength and resistance to sodium sulphate attack of concrete incorporating recycled ceramic tiles (RCT) as fine aggregate were investigated. RCT was used as partial replacement for river sand at four levels (0%, 33%, 66%, 100%). Samples for sulphate resistance tests were immersed in 5% Na_2SO_4 solution for 180 days after they had been cured under water for 28 days, and were monitored for change in physical appearance, mass change and loss of compressive strength. From experimental results, RCT was found to be capable of producing light weight concrete compared to river sand. The results showed increase in compressive strength as the level of RCT content increased. On resistance to sulphate attack, sodium sulphate seems not to attack C-S-H bond which is produced in excess in RCT concrete, rather it attacks calcium hydroxide and calcium aluminate which are produced in equal amounts for both RCT and control samples. Hence, RCT might not play much direct role in concrete's resistance to strength loss due to sulphate attack. However, the residual compressive strength of the RCT samples after the attack was seen to be much higher than that of the control samples because of their initial higher strength before the attack. This shows that RCT can improve the properties of concrete when incorporated as fine aggregates.

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Concrete production comes with a huge environmental impact. This happens in two major forms. The first is the emission of greenhouse gases, which occurs during the manufacturing of Portland cement (PC). It is estimated that the production of one tonne of PC generates approximately one tonne of CO_2 to the atmosphere accounting for about 5% of global CO_2 emissions (Neville, 2011; Kannan *et al.*, 2017). The second negative impact of concrete production in our environment is the rapid reduction of the natural reserve of traditional crushed rock aggregate and river sand, leading to scarcity of these materials. To meet up with sustainable development requirements and the need for environmentally friendly concrete production, there are numerous literatures on the usage of industrial wastes and by-products as replacement for either aggregate or cement in concrete. Several materials have already been incorporated into concrete production in practice. These include materials like fly ash (Fasihihour *et al.*, 2022), ground granulated blast furnace slag (GGBS) (Ogirigbo and Black, 2017; Ogirigbo and Inerhuwa, 2017; Ambrose and Forth, 2018), silica fumes (Mehta *et al.*, 2020), recycled concrete (Kisku *et al.*, 2017), quarry sand (Verma *et*

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al., 2020; Ambrose *et al*., 2018) and others. Research has confirmed the feasibility of incorporating even many more waste materials. Such materials include: palm kernel shell, periwinkle shell, recycled glass (Keerio *et al*., 2020; Malek *et al*., 2020) recycled plastics, (Babafemi *et al*., 2018) and recent, ceramic wastes (Ikponmwosa and Ehikhuenmen, 2017; Samadi *et al*., 2020; Siddique *et al*., 2018; 2018b; Mohammadhosseini *et al*., 2019; Ambrose *et al*., 2021; 2021b; Onyia *et al*., 2023).

Ceramic wastes are generated by manufacturers of ceramic products such as ceramic tiles, porcelain, bricks, electrical insulators, sanitary wares and many others as a result of cracks, off-standard products, size discrepancies, production error, glazing fault, etc. They are also generated during the transportation and distribution of these products or as construction and demolition waste. Lots of ceramic wastes are produced globally and are currently not recycled in any substantial quantity (Elci, 2016; Awoyera *et al*., 2018), rather are deposited of in landfills. Unlike wastes like sawdust which are biodegradable (Etim *et al*., 2017), ceramic wastes are non-biodegradable (Ray *et al*., 2021; Medina *et al*., 2016). Therefore, incorporating them into concrete production will not only be of great benefit to the concrete industry, but will also go a long way in resolving the environmental issues allied with ceramic waste landfills. The incorporation of ceramic wastes as aggregate in concrete has been widely researched (Ambrose *et al*., 2021; 2021b; Rajawat *et al*., 2018; Awoyera *et al*., 2018; Dang and Zhao, 2019). However, most of the researches have been on strength and other mechanical properties (Onyia *et al*., 2023; Rajawat *et al*., 2018; Dang and Zhao, 2019) with very few data on durability properties. From a review of literatures on the durability properties of ceramic wastes aggregate based concretes, it is obvious that there are no research data on concrete incorporating ceramic waste tiles as fine aggregate. The few available data on durability properties considered the use of other ceramic wastes aggregate like sanitary wares and red bricks. However, different ceramic products are produced using different combinations and proportions of geomaterials (Elci, 2016) and are fired at different temperatures during their manufacturing processes (Ozkan *et al*., 2010). This means that their microstructure and intrinsic properties are different. It will therefore be improper to assume the performance of ceramic tiles aggregate concrete to be the same as others. In this study, compressive strength and resistance to sulphate attack of concrete incorporating recycled ceramic tiles (RCT) as fine aggregate was investigated.

MATERIALS AND METHODS

Materials: Five materials were used to prepare concrete samples for the experiments in this study. These included: cement, water, river sand (RS), recycled ceramic tiles (RCT) and granite chippings.

Binder: The cement used was Portland Limestone cement (CEM II), manufactured by United Cement Company of Nigeria in conformation to NIS 444-1 (2008) with strength class 32.5MPa. The chemical composition of the cement was determined via X-ray fluorescence (XRF).

Aggregates: RS and RCT were used as fine aggregate while granite chippings were used as coarse aggregate (CA). The RS used was acquired from a quarry site at Ikot Ekong, Akwa Ibom State, while the granite chippings were obtained from a mining site in Akamkpa, Cross River State, in Nigeria. To process the RCT, waste ceramic wall and floor tiles were acquired from a ceramic tile dealer in Uyo. These were tiles that had been manufactured properly but became damaged during transportation or handling (see Figure 1(a)). The waste ceramic tiles were first broken into tiny bits, then crushed and milled into the desirable size using a hammer mill and British Standard sieves respectively. The crushed RCT aggregate is as shown in Figure 1(b). The physical properties of the crushed RCT aggregate such as particle size distributions, specific gravity, and bulk density, were determined following the British standards; while the chemical composition was determined via XRF.

Fig 1: Recycled ceramic tile before (a) and after (b) crushing

Mix design: Mix design was based on an optimum mix derived in a previous study (Ambrose *et al*., 2021) for RCT fine aggregate concrete with target strength of 35MPa. The optimum mix for this criterion is as presented in Table 1 with a designation, M_{66}. The mix had 66.6% replacement of sand with RCT. Three other mixes were derived from the optimum mix by changing the percentage replacement of sand with RCT. The selected percentage replacements were 0%, 33.3% and 100% and the mixes were designated as M_0, M_{33} and M_{100}, respectively, as shown in Table 1. The water-cement ratio for M_{100} was slightly adjusted from 0.531 to 0.577 since the mix was not workable at the former water-cement ratio. M_0 served as the control mix since it had 100% sand as fine aggregate, while mix M_{100} had 100% RCT as fine aggregate.

Table 1*:* Mix proportions for production of test samples

Mix Designation	% Replacement of sand with RCT	Real Component Ratios				
		Water	Cement	Sand	RCT	CA
M_0	0	0.531	1.000	1.372	0.000	2.742
M_{33}	33.3	0.531	1.000	0.915	0.457	2.742
M_{66}	66.6	0.531	1.000	0.458	0.914	2.742
M_{100}	100	0.577	1.000	0.000	1.372	2.742

Production of concrete samples: Concrete mixing and curing was done in accordance with BS EN 12390-2 (2009). Batching was done by weight using the design mix design in Table 1. For each batch of mix, constituents were manually mixed with the aid of trowel and shovel. The required weights of cement, sand and RCT were initially mixed to obtain a homogeneous mix. Coarse aggregate was then added and further mixing was carried out. Water was then added to the mix, and mixing continued until a consistent mixture was obtained. For all sample production, a thin layer of grease was added to the inner surfaces of the mould, to prevent adhesion of the concrete to the mould. The concrete mixture was placed into 100mm cubes and left to cure under air in the laboratory for 24 hours. Thereafter, the concrete cubes were demoulded and placed in curing tans to cure under water till the day of testing.

Test Methods

Compressive strength tests: The compression testing machine of 2000kN testing capacity was used for all compressive strength tests. The test was performed in accordance with BS EN 12390-3 (2009). The test sample for each test was placed in between the two steel platens (30mm thick) of the machine. Progressive compressive loading was then applied to the sample till failure occurred. The load at failure for each sample was taken and the compressive strength was computed by dividing the failure load by cross sectional area of the sample.

Sulphate resistance test: Tests on sulphate resistance were conducted on 100mm concrete cubes using sodium sulphate solution similar to the procedure used elsewhere (Mohammadhosseini *et al*., 2019; Tang *et al*., 2019). Samples were immersed in 5% (50g/l) sodium sulphate solution at laboratory temperature (23 ± 2°C) for a total of 180 days after initial 28 days of wet curing. They were monitored for visual appearance, mass loss or gain and loss of compressive strength. Samples remained completely immersed in the solution until their respective test dates. The sodium sulphate solution was stirred on a regular basis throughout the period. The initial 28 days of curing was to mimic situations where precast concrete is used. Testing was carried out on the 14th, 28th, 56th, 120th and 180th day from the date of the first immersion. Before immersion, all sample were uniquely labelled and weighed. The mass was recorded as initial mass (M_a). On each test date samples were weighed again and weight was recorded as M_t before the compressive strength test. Two samples were tested for each mix on each test age. Mass gain or loss (M_Δ) was obtained in % as:

$$M_{\Delta} = \frac{M_t - M_o}{M_o} \times 100 \qquad (1)$$

Compressive strengths were determined using the initial cross-sectional area to evade measuring any change in dimension. An identical set of samples were also completely immersed in water for the same periods and used as control samples. Loss of compressive strength was therefore computed with reference to the compressive strength of the control samples.

RESULTS AND DISCUSSION

Materials: Table 2 and Figures 2 and 3 show the results of the preliminary tests carried out on aggregates used in this study while Table 3 presents the chemical compositions of RCT and cement used. From the results of bulk density and specific gravity tests in Table 2, it could be seen that RCT has a lighter weight compared to the river sand. This is because the specific gravity and bulk density values of RCT were lower than those of RS. This has also been observed in earlier studies (Elci, 2016; Ikponmwosa and Ehikhuemen, 2017). It therefore means that RCT will produce light weight concrete than RS and this is of great advantage to engineers and other concrete users because it will reduce the self-weight of concrete elements in structures. Particle size distribution curves in Figure 2 shows that both RS and RCT curves are within the boundaries of fine aggregate grading curves according to BS 882:1992 (Neville, 2011). Values of C_u and C_c in Table 2 also shows that the range of particle size is wider in RCT than in RS and CA. RCT can also be said to be well graded because its C_c is between 1 and 3 while its C_u is greater than 6 (Ambrose *et al*., 2019).

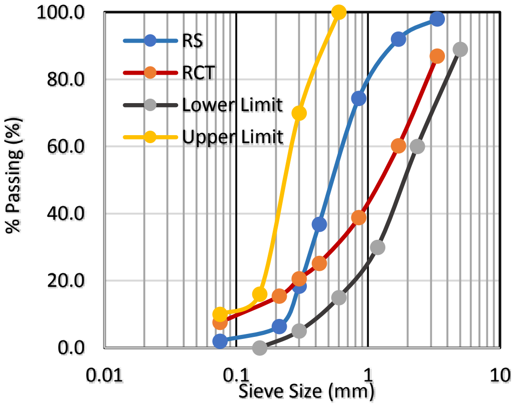

Fig 2: Particle size distribution curve for fine aggregates

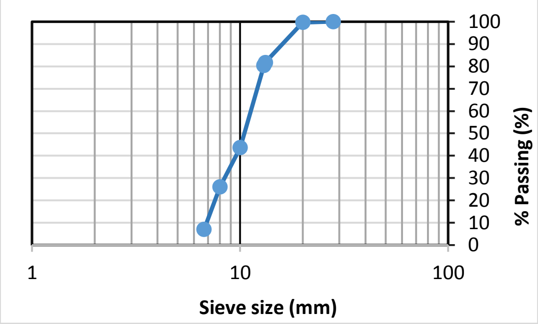

Fig 3: Particle size distribution curve for CA

Table 2: Physical properties of aggregates

Property	Sand	RCT	CA
Specific gravity	2.61	2.40	2.39
Bulk density (kg/m^3)	1635	1373	1386
Uniformity coefficient (C_u)	2.85	17	1.84
Gradation coefficient (C_c)	0.73	1.78	0.87

Compressive strength: Results of the compressive strength of concrete samples at varying test ages and levels of replacement of sand with RCT are presented in Figure 4. M_{100} samples had the best performance in terms of gain of compressive strength with age. They were followed by M_{66}, M_{33} and M_0 samples respectively. This trend was consistent at all test ages – at the 7th, 28th and 90th day. This indicates that the use of RCT as fine aggregate increases concrete strength and this increase is directly proportional to the level of replacement of conventional fine aggregate with RCT. Similar improved strengths have been reported in earlier studies on concrete samples incorporating ceramic waste as fine aggregate (Siddique *et al*., 2017; Elci, 2016) although a few studies have also reported reduction in strength of ceramic waste aggregate concrete samples compared to control samples (Alves *et al*., 2014).

Table 3*:* Chemical composition of cement and RCT

Compound	% Composition by mass	
	Cement	RCT
Iron Oxide (Fe_2O_3)	2.25	3.07
Aluminum Oxide (Al_2O_3)	4.73	17.50
Silicon dioxide (SiO_2)	19.84	66.13
Calcium Oxide (CaO)	70.32	5.70
Manganese Oxide (MnO)	0.01	0.58
Magnesium Oxide (MgO)	1.47	2.14
Zinc Oxide (ZnO)		0.42
Sulfur trioxide (SO_3)	0.03	-
Sodium Oxide (Na_2O)	0.08	0.09
Potassium Oxide (K_2O)	0.72	1.02
LOI (Loss of Ignition)	1.01	3.30

Two major factors are likely to be the reason for the increase in the strength of concrete as a result of the incorporation of RCT as fine aggregate. These factors are directly related to some inherent properties of ceramic waste aggregates. Generally, most ceramic waste aggregates are porous and are characterised by high water absorption (Alves *et al*., 2014; Elci, 2016). For this reason, RCT absorbs more mixing water than sand and this reduces the actual quantity of water available for hydration. This will then be the same as using a lower water-cement ratio which usually leads to higher strength. The second factor is linked to the angular and irregular shape of ceramic waste aggregates and their rough surface texture (Siddique *et al*., 2017).

Aggregate's shape and surface texture in a way affects aggregate-cement paste bonding and consequently, concrete strength (Mkpaidem *et al*., 2022; Zegardlo *et al*., 2016). Angular and irregularly shaped aggregate materials usually produce stronger bonding than round shaped aggregate materials. On the other hand, aggregates with rough surface texture perform better than aggregates with smooth texture. Therefore, the irregular and angular shape of RCT combined with the roughed surface texture of its particles improves aggregate-cement paste bonding and hence, strength.

Resistance to sulphate attack

Visual appearance: Samples exposed to sodium sulphate medium exhibited no noticeable change in their physical appearance, throughout the 180 days of exposure. This was at variance with reports from previous studies like Mohammadhosseini *et al*. (2019) and Tang *et al*. (2019) which reported cracks, spalling, warping and loss of materials (at the corners) of samples exposed to sodium sulphate medium. However, the exposure periods in their studies were 18, 14 and 22 months respectively as against 180 days (approximately 6 months) in the present study. Therefore, it takes longer a period for visual signs of deterioration to occur in concrete exposed to sodium sulphate medium.

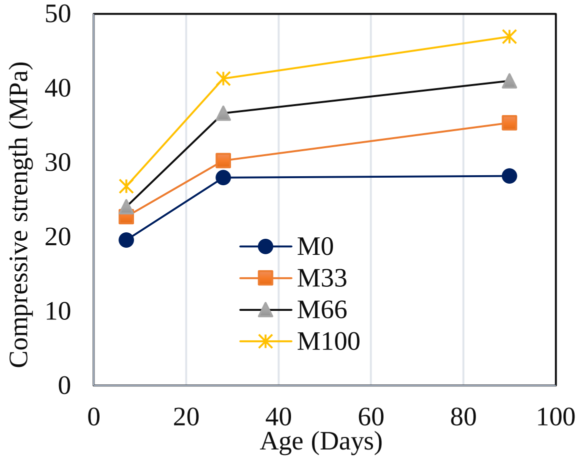

Fig 4: Compressive strength of concrete at varying age and level of RCT content

Mass change: Change of mass for samples exposed to 5% Na_2SO_4 solution was generally low and almost insignificant. As shown in Figure 5, values of mass loss or gain were within 0.2% of their respective initial mass before immersion except for M_{100} samples which exhibited up to about 0.5%. This is similar to the results obtained by Tanwar *et al*. (2021) on concrete samples. Values of mass change obtained by Tang *et al*. (2019) as at 6 months (about 180 days) exposure period to sodium sulphate medium were also similar. They were less than 0.2% of their initial mass. The low mass change obtained in this study in addition to the fact that there was no noticeable change in the physical appearance of samples shows that sulphate attacks on all samples was minimal. This could mean that the concentration of the sodium sulphate solution was very mild for such test duration compared to the strengths of the concrete samples. However, 5% of sulphate solution is the common concentration used in most studies involving resistance of concrete to sulphate attack (Bing *et al*., 2007; Mohammadhosseini *et al*., 2019; Samadi *et al*., 2020; Aziez and Bezzar, 2017; Tang *et al*., 2019; Tanwar *et al*., 2021) and this seems to represent its most common concentration in soils and groundwater where sulphate attack on concrete is common. Most studies reporting high values of mass change use mortar as test samples. This could be seen in Mohammadhosseini *et al*. (2019) and Samadi *et al*. (2020), which recorded up to 2.5% and 4.5% mass change respectively on mortar samples. Mortar samples would certainly contain a higher percentage of hardened cement paste than concrete samples because of the omission of coarse aggregate in mortar. Since sulphate attack is usually on hydration products on cement pastes, its effect will be more severe in the mortar than on concrete, thereby causing higher mass change. The same explanation could be given to the results of Ikumi *et al*. (2019) and Aziez and Bezzar (2017) which recorded up to 1.4% and 2.5% mass change respectively. From these, it is obvious that tests on the resistance of concrete samples to sulphate attack produces less effect than those on mortar samples and therefore should be carried out for much longer duration. Nevertheless, the slight change of mass is caused by decomposition and leaching out of hydration products within the cement paste into the solution. Even with the insignificant percentage change in mass, M_{66} samples still recorded slightly lower values than M_0 and M_{33} samples.

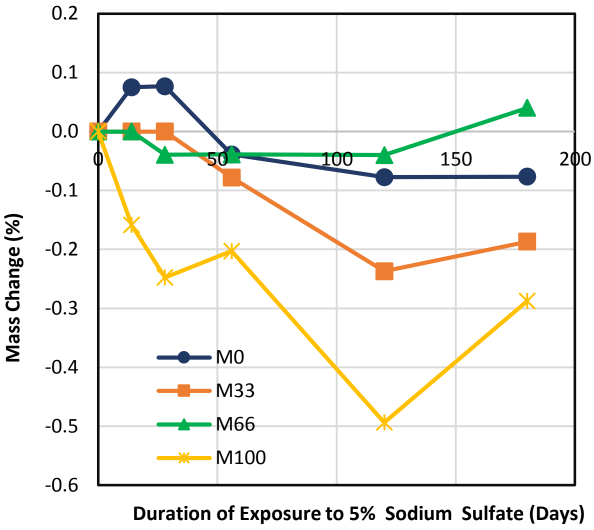

Fig 5: Mass change for samples exposed to Na_2SO_4

This improved resistance of RCT concrete samples could be due to their refined and stable microstructure caused by the formation of additional C-S-H during the hydration process (Samadi *et al*., 2020; Mohammedhosseini *et al*., 2019). M_{100} samples recorded the highest mass loss at all exposure durations. This is most likely to be linked with the poor workability of M_{100} samples in fresh state. These samples with 100% RCT as fine aggregate had the least workability. It is possible that full compaction was not achieved on these samples and this creates more voids that ease the penetration sodium sulphate solution into the concrete samples. Therefore, in using RCT as fine aggregate, the mix should be carefully designed to achieve appropriate workability that will aid in obtaining maximum compaction.

Loss in Compressive Strength: Figure 6 shows the loss of compressive strength for samples exposed to sodium sulphate for 180 days. Loss of compressive strength was measured in relation to their respective control samples. There was an obvious loss of strength for both control and RCT samples and this was caused by sodium sulphate attack on calcium-based hydration products. Sulphates decompose the products of hydration and form new compounds (Neville, 2011). This gradually weakens aggregate-cement paste bonding and hence reduces concrete strength. Microstructural analysis on concrete incorporating ceramic waste shows that the pozzolanic property of ceramic wastes (Elci, 2016), causes more $Ca(OH)_2$ to be used up in the formation of additional C-S-H during the hydration process. This in turn creates excess C-S-H and gives such concrete a more compacted and stable microstructure (Samadi *et al.*, 2020; Mohammedhosseini *et al.*, 2019). At first, it was surprising that the percentage reduction in compressive strength due to the Na_2SO_4 attack was similar for both RCT concrete samples and control samples according to Figure 6. It was expected that RCT samples would record much less percentage decrease in compressive strength compared to control samples. However, Neville (2011) has stated that different sulphates attack different products of hydration and further explained that while Na_2SO_4 attacks $Ca(OH)_2$ and calcium aluminate, $MgSO_4$ attacks C-S-H in addition to $Ca(OH)_2$ and calcium aluminate. It therefore seems that Na_2SO_4 does not attack C-S-H which is produced in excess in ceramic based concrete. Hence RCT seems not to play much direct role in concrete's resistance to Na_2SO_4 attack. In the studies of Mohammadhosseini *et al.* (2019) and Samadi *et al.* (2020) where improved resistance (to Na_2SO_4 attack) of mortar with ceramic aggregate as fine aggregate was reported, it should be noted that there was also a replacement of cement with ceramic waste powder. Hence, the improved performance could have been more from the effect of cement replacement than the use of ceramic waste aggregate. In another study by Siddique *et al.* (2018) where improved resistance to sulphate attack was also reported, it should be noted that samples were exposed to $MgSO_4$ medium and not Na_2SO_4. Since $MgSO_4$ attacks C-S-H, the additional C-S-H will have a significant resistance to sulphate attack. Nevertheless, it should be noted that the plots in Figure 6 were percentage of the initial 28th day strength of samples. The actual residual strengths of RCT samples were still far greater than those of the control sample at all exposure durations as shown in Figure 7. Figure 7 shows the compressive strengths of control samples and their respective residual strengths after exposure to Na_2SO_4 Solution. The residual compressive strength at all exposure durations was directly proportional to the level of RCT content as fine aggregate. With this, there is still an added advantage of using RCT as aggregate for concrete exposed to a sodium sulphate environment.

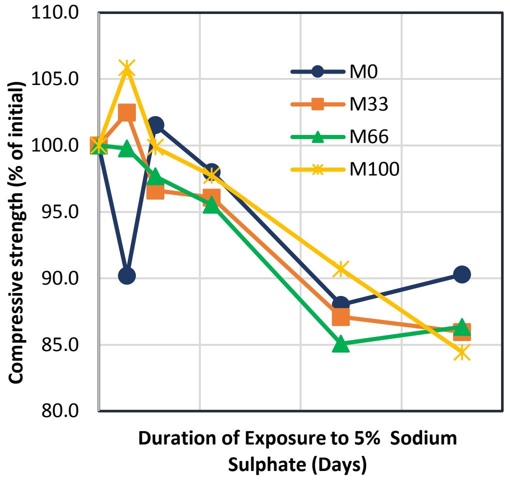

Fig 6: Compressive strength loss (with respect to control samples) for samples exposed to 5% Na_2SO_4

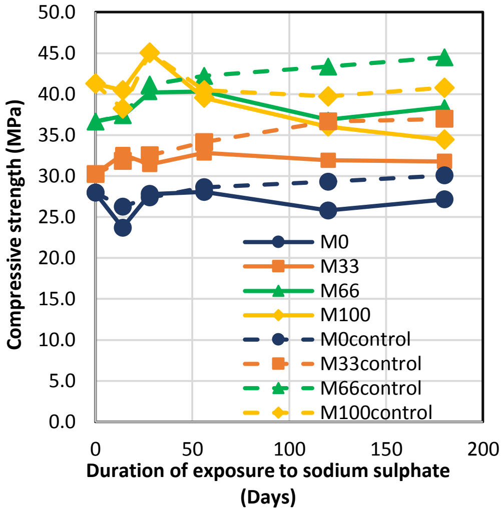

Fig 7: Compressive strength of controls samples and residual compressive strength of samples exposed to Na_2SO_4

Conclusion: The results of this study have shown that RCT is capable of producing light weight concrete compared to river sand. Compressive strength increased as the level of RCT content increased, due to the pozzolanic nature and some intrinsic physical properties of the RCT. On resistance to sulphate attack, it was seen that RCT might not play much role in concrete's resistance to strength loss due to sodium sulphate attack. However, the residual compressive strength of the RCT samples after the attack was still seen to be better than that of the samples without RCT.

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