

Eco-friendly chopped tire rubber as reinforcements in fly ash based geopolymer concrete

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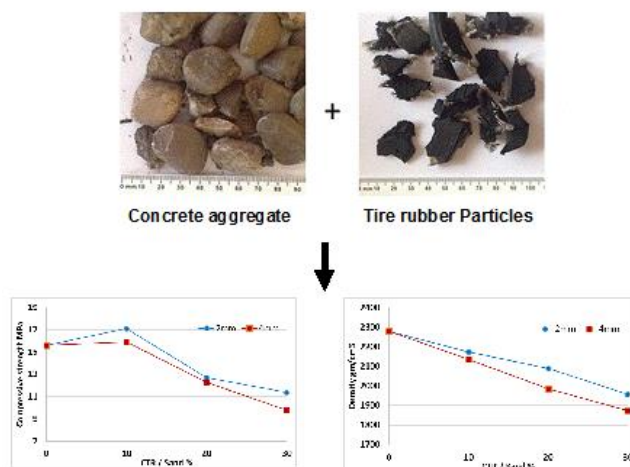
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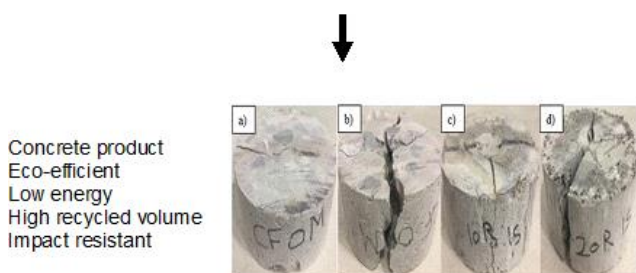
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Graphical abstract



The 28d compressive strength and density at various CTR to sand ratios.



Abstract

One of the major challenges faced by researchers is to recycle industrial wastes in a manner that reduces their environmental impact in nature. An experimental study was carried out to determine the suitability of using chopped tire rubber as reinforcements in green and sustainable geopolymer concrete, with the purpose of using them as nonstructural products. The geopolymer mixture was made by mixing of fly ash powder, fine aggregate, and Superplasticizer in Na₂SiO₃/NaOH solution. Mixtures were divided into four different groups, with

constant water to fly ash ratio of 0.12 and alkaline dosage of 45% by weight of fly ash, based on the recycled chopped tire rubber (CTR) content: 0, 10, 20, and 30% by volume of fine aggregate with two maximum sizes (2 and 4 mm). Hardened properties of resulted geopolymer like compressive strength, density; and ultrasonic pulse velocity were examined at 28d. Besides that, X-Ray diffractometer and Scanning Electron Microscope were used in order to observe the microstructure of the resulted geopolymer concrete. In view of the consequences for this study, it is preferable to replace no more than 10% of fine aggregate in geopolymer concrete by CTR. In addition, according to SEM photographs, increasing the CTR content more voids will be pronounced and thus, decreasing the mechanical performance.

Keywords: Fly ash, geopolymer, strength, microstructure, UPV, chopped tire rubber CTR, XRD, SEM.

1. Introduction

It is well known that using waste materials in the manufacturing of building units such as geopolymer concrete, is an opportunity to reduce their environmental impact. "Geopolymer is an alumino-silicate reactive materials with strongly alkaline solutions" (Nurrudin *et al.*, 2018). Geopolymer, has properties such as low consumption of raw resources, no CO₂ emission, less energy consumption, low production costs, and low heat of hydration. These properties make geopolymer discover incredible applications in numerous fields of industry like civil engineering (Al-Shathr *et al.*, 2019).

Davidovits stated that geopolymerization might be too close to zeolite formation, although the geopolymer microstructure is amorphous to semi-crystalline rather than crystalline. He also stated that geopolymers have a three-dimensional structure and belong to the group of zeolites and feldspathoids. The designation of geopolymers based on aluminosilicate is called poly (sialates) involving an amorphous network of AlO₄ and SiO₄ tetrahedra linked interchangeably by sharing all the oxygens in the form of silicon-oxo-aluminate (-Si-O-Al-O-) and abbreviated as sialate. The presence of positive ions, such as Na⁺, K⁺, and

Ca⁺⁺, in the framework is necessary to balance the negative charge of Al³⁺ in IV-fold coordination with oxygen. This polymeric response is similar to the formation of zeolites and zeolite precursors. The same findings for fly ash geopolymer concrete production were also documented by Komljenovi (Komljenović *et al.*, 2010) even when sodium hydroxide is prepared with sodium silicate (water glass) for making ready solution and then added to powder fly ash.

Bernal *et al.* (2011) studied the addition of slag to Metakaolin based geopolymer concrete. Slag caused as 0, 20, 40, 60 and 80 percent replacement of metakaolin modified the compressive strength increased as compared with geopolymer concrete synthesis by metakaolin only, the optimum percentage was 60 percent.

In a related study, the detrimental effect created by the addition of waste fibers is due to the initiation of more pores and microcracks into the matrix. An increase in pores may be because of the poor workability of a larger fiber volume fractions, which made compaction difficult (Shanthini *et al.*, 2016). The increased number of microcracks with high fiber volume fractions is due to fibers touching one-another, resulting in fibers that are poorly bonded or even unbounded in the matrix and this creates weak zones (Kumaravel and Sivakumar, 2018).

Parveen *et al.* (2018) investigated the influence of incorporating alccofine on the properties of fly ash based geopolymer concrete at ambient temperature. The results showed that specimens prepared with alccofine have an important impact on the polymerization of the geopolymer concrete, that lead to improve the strength and microstructural characteristics.

This study is an attempt mainly to understand the microstructural and mechanical performance of sustainable fly ash geopolymer concrete with and without chopped tire rubber as a replacement of fine aggregate. The second important objective is to find a direct relationship between the investigated mechanical properties and UPV. So, it become too easy to estimate strength in terms of direct pulse transmission. In addition to reduce the harmful environmental impacts of some industrial wastes.

2. Materials

The raw materials used to produce geopolymer are fly ash powder, natural sand, coarse aggregate, super plasticizer, tap and distilled water and alkaline activator (Sodium Silicate + Sodium Hydroxide). The chemical composition of the fly ash is presented in Table 1, which indicated that they are compatible with the requirements of ASTM C-311 (ASTM C311/C311M) and ASTM C-618 (ASTM C-618) Class F specifications with strength activity index of 118 % at 7d. The alkaline solution was prepared using Sodium silicate (Na₂SiO₃), fabricated in the United Arab Emirates. The concentration of the Na₂SiO₃ depends on the ratio of Na₂O to SiO₂ anticipated. Commercial sodium hydroxide (NaOH), with 99 % purity in flake form was utilized. The solids should be dissolved in distilled water to form an activator with the desired molar concentration (10 M). A total of 12% (by weight of FA) was added to increase the homogeneity

of the producing geopolymer. Quartz-based natural sand that conforms to I.Q.S No.45 (Iraqi Standard No. 45), zone 3 was employed as a fine aggregate.

Table 1. Chemical composition of fly ash*

Constituent	Fly Ash (%)	Limits of ASTM C-618/05
CaO	1.20	
SiO ₂	49.6	≥ 70 %
Al ₂ O ₃	44.9	
Fe ₂ O ₃	4.00	
SO ₃	0.18	≤ 5
NaOH+KOH	---	
Loss on Ignition	2.4	≤ 6 %
Fineness	20 %	≤ 34 %

*Chemical tests were made by the National Center for Geological Survey and Mines, Baghdad, Iraq

Tables 2 and 3 show the grading of chopped rubber and fine aggregates used in this paper, and the chemical composition of CTR respectively. Rheobuild SP1 (commercially known as Master RHEOBULD SP1) high-range water reducer superplasticizer was utilized in all mixtures. Rheobuild SP1 is designed specially to impart rheoplastic qualities to concrete. It is a modified Sulphonated naphthalene based chemical aqueous solution, purchased from Sika Company in Iraq. The properties of Rheobuild SP1 are followed the ASTM C 494 (ASTM C494/C494M and ASTM 494/C 494M) Type F. Crushed gravel of 10 mm maximum size obtained from Al-Nebai quarry (middle of Iraq) was used as the coarse aggregate in all mixes. The results show that coarse aggregate conforms to the Iraqi Standard IQS 45 (Iraqi Standard No. 45).

Table 2. Grading of fine aggregate and chopped tire rubber*

Item	Sieve size (mm)						
	10	4.75	2.36	1.18	0.60	0.30	0.15
Fine aggregate	100	95	78	56	43	15	6
Chopped Rubber	100	100	97	83	28	7	0
Tires							

*Tests were made by the Concrete Laboratory in Karbala Technical Institute, Karbala, Iraq

Table 3. Chemical composition of chopped tire rubber*.

Rubber hydrocarbon (SBR)	47.7%
Carbon black	30.7%
Acetone extract	15.6%
Ash	2.1%
Residue chemical balance	3.9%
Density	620 kg/m ³

*Results according to the manufacturer

3. Experimental procedure

Since there is no committee that specify the mix proportions of geopolymer concrete, so the mix used in this work shown in Table 4 as previously proposed by Ali *et al.*

(2019). Three concrete samples were chosen for each test carried out for 28d to investigate compressive strength, density and UPV. The compressive strength and density tests were done using 100 mm cubes according to BS 1881: Part 116 (BS 1881: Part 116) and BS 1881: Part 114 (BS 1881: Part 114) standards respectively. Meanwhile, ultrasonic pulse velocity test was done using 100 mm cubes specimens according to BS 1881: Part 203 (BS 1881: Part 203) standard. After casting the specimens, hot curing technique was used in water of 60 °C for 48 hrs followed by moist curing until the testing day.

Table 4. Mix proportions

Mix No.	Fly ash (kg/m ³)	Fine aggregate		SP (kg/m ³)	Coarse aggregate (kg/m ³)	Na ₂ SiO ₃ / NaOH by w.t of FA (%)	CTR by vol. of sand
		kg/m ³	m ³				
Control		720	0.465				0
M10	400	647	0.418	8	1100	45	10
M20		576	0.372				20
M30		505	0.326				30

4. Results and discussion

The replacement of chopped tire rubber as a percentage volume of fine aggregate, to geopolymer concrete mixtures after 28d have been investigated and discussed. However, the increase in the compressive strength shown in Figure 1, of specimens containing 10 % chopped tire rubber was more than that of control due to the low voids as the scanning electron microscope prove later. Chopped tire rubber which is beneficial for bridging of micropores and hence, enhancement of the final strength by 9.6 % and 2.0 % for 2 mm and 4 mm respectively. On contrasts, it is obvious from results that as the percentage of chopped tire rubber increases more than 10 %, the compressive strength decreases for mixes M20 and M30 as compared to that of control. The reduction in compressive strengths at 20 % and 30 % of CTR replacement were of (18.6 and 26.9) % and (21.2 and 37.1) % for 2 and 4 mm respectively. This could be attributed to the low specific gravity of CTR and/or to the increase in the number and size of micropores. The same results were reported by Maranan *et al.* (2015).

Figure 2 shows the relationship between the oven dry densities for all geopolymer concrete mixes containing chopped tire rubber as compared to control mix. The results illustrate that geopolymer mixes containing CTR have lower oven dry density relative to that of control. Furthermore, increasing the 2 mm chopped tire rubber to fine aggregate ratios by 10, 20 and 30 % decreases the oven dry density of 4.6, 8.3 and 14.1 % respectively. While, the reductions became of 6.3, 12.9 and 17.8 % as the chopped tire rubber maximum size was increased to 4 mm. This is due to the low specific gravity of the used chopped tire rubber and more initiated pores in internal structure. However, the densities of geopolymer concrete are close to density of normal concrete, which varies in the range of 2200-2500 kg/m³. This is in agreement with the pervious findings by Al-Shathr *et al.* (2016).

In addition, in order to characterize their reaction products, geopolymer concrete samples from each of the four mixtures were analyzed using X-ray diffractometer (XRD) analysis. The textural study of the fractured surface for the samples was performed using (SEM Model: TESCAN-VEGA/USA) with tungsten source and detector X-Flasb 5030, which operates at a voltage of 1–20 kV with a range of between 10 and 80,000 - magnification, at a work distance from 1 to 10 mm.

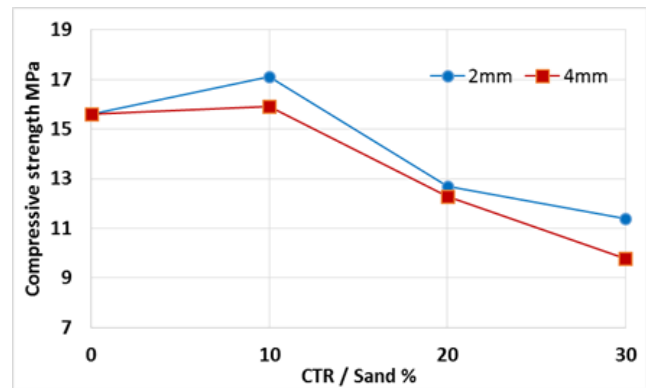


Figure 1. The 28d compressive strength at various CTR to sand ratios

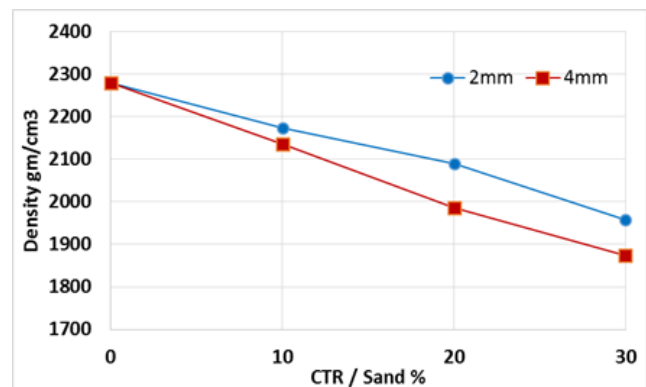


Figure 2. The 28d density at various CTR to sand ratios

The UPV test is used to estimate the uniformity and quality of geopolymer concrete, and the present of voids. Figure 3 presents the relationship between ultrasonic pulse velocity and chopped tire rubber of different maximum size (2 and 4 mm). From this figure, there is almost a linear decrease in the pulse transmission of studied specimens with the increase in the chopped tire rubber to sand ratios. Like density, the replacement of 2 mm CTR to geopolymer

concrete mixtures by 10, 20 and 30 % by volume of sand resulted in a decline of the UPV for all specimens after 28d by 9.6, 14.0 and 20.5 % respectively. However, the reductions became of 15.8, 18.3 and 31.4 % as the chopped tire rubber maximum size was increased to 4 mm. Similar findings were obtained by Kim and Kim (Kim and Kim).

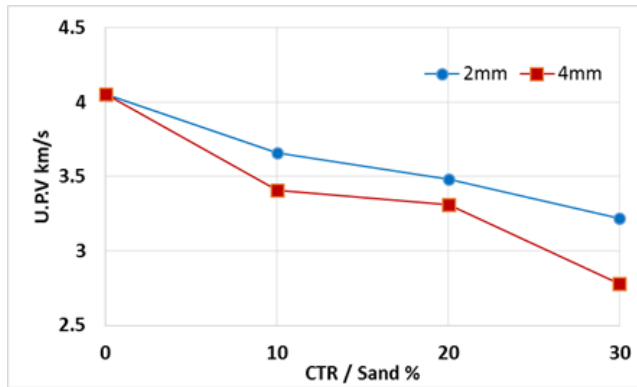


Figure 3. The 28d U.P.V at various CTR to sand ratios

The following equations shown in Figure 4 may be suggested to fit the relationship between compressive strength and Ultrasonic Pulse Velocity for fly ash geopolymer concrete associated with good accuracy. Equation 1 is used for geopolymer concrete with 2 mm CTR maximum size, meanwhile, equation 2 is used for geopolymer concrete with 4 mm CTR.

$$f_c = 3.45e^{0.3941v} \tag{1}$$

$$f_c = 4.05e^{0.3486v} \tag{2}$$

where:

f_c = Compressive strength in MPa, v = direct pulse velocity in km/s.

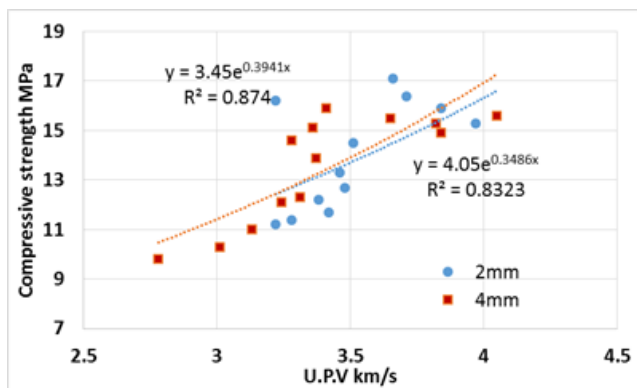


Figure 4. Predicted equations for compressive strength of geopolymer concrete with UPV at 28d

The lower fitting curve (2 mm) shows better-fit nonlinear regression curve. This curve minimizes the sum-of-squares of the vertical distances of the points indicating good relevance between experimental data and the theoretical data. In this curve, the sum of squares of those distances equals 0.8323. The upper fitting curve (4 mm) of the figure

shows the R^2 of 0.874. According to Mulholland and Hibbert (1997), R^2 with a value > 80% is usually considered a strong relationship between any two variables. Additionally, Kurtoğlu *et al.* (2018) described the expression which had R^2 of 0.84 in their work as a good relationship. Therefore, these formulas may be appropriate for geopolymer concrete.

X-ray powder diffraction is a powerful technique to study semi-crystalline materials like geopolymer. According to Figure 5, semi crystalline peaks were noticed in all of the investigated mixes. Moreover, for all the specimens, well-defined diffraction peaks of Quartz (Q) were observed at $2\theta = 12.2^\circ, 28.1^\circ$ and 32.3° . All the other lower peaks correspond to the Aluminum Mullites, Merwinites, and Calcium silicates are also noticed. The highest peaks intensities were observed at $2\theta = 28.1^\circ$ for control mix due to the presence of Quartz. After the addition of CTR, highest peaks intensities were observed at $2\theta = 32.3^\circ$ for M10, M20 and M30 respectively. Amorphous peaks intensities of geopolymerization were also observed and was not easy to indicate. The percentages of amorphous silicates were more in the case of M30 mix. It shows that at higher percentage of CTR in the geopolymer concrete indicates less crystallinity. The same trend was found by Parveen *et al.* (2018).

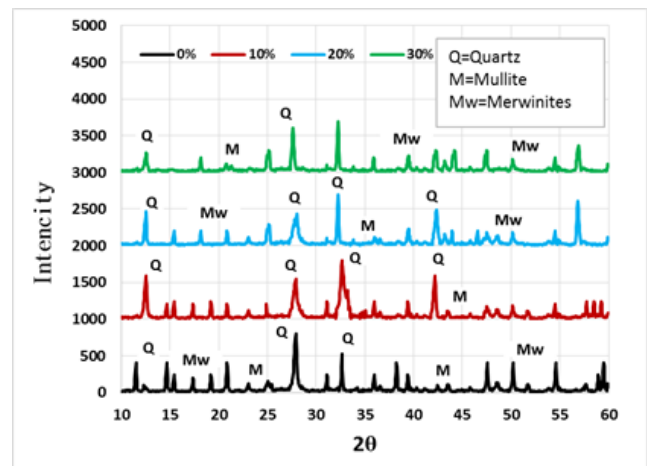


Figure 5. X-ray patterns for geopolymer concrete at 28d and 2 mm maximum size of CTR

Scanning electronic photographs of different specimens prepared in this investigation were carried out in order to study the reactants of fly ash and to authenticate the internal microstructure of geopolymerization. The SEM photographs of control, M10, M20, and M30 with 2 mm chopped tire rubber, at 28d, are shown in Figure 6 (a to d). The aggregate matrix interface of the fracture surfaces for samples with chopped tire rubber do not possess any indication of cracking like that found in control specimen in Figure 5-a. It is also obvious from Figure 6-b that the addition of CTR enhanced the microstructure of geopolymer for M10 associated with thicker ITZ. This may be due to the more rough surface texture of the chopped tire rubber which leads to good interlocking with geopolymer matrix. In addition, increasing the CTR to 20 and 30% as shown in Figures 6-c and 6-d initiate additional

voids in the internal microstructure and this is definitely the main reason for density and compressive strength reduction at hardened state. Same observations were noticed by Embong *et al.* (2016) and Gandoman and Kokabi (2015).

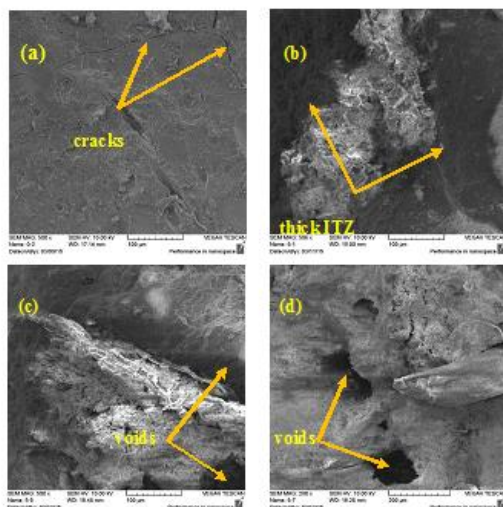


Figure 6. SEM photographs of geopolymer concrete at 28d (a) control, (b) M10, (c) M20 and (d) M30 of 2 mm CTR

5. Conclusions

Based on the materials used and the results obtained, conclusions can be drawn:

1. Higher compressive strength was gained using chopped tire rubber as a replacement by volume of the fine aggregate of not more than 10 %. On contrast, higher fractions (20 and 30%) of chopped tire rubber, lower compressive strength, density and UPV were observed.
2. Reducing the CTR maximum size from 4 mm to 2 mm resulted in an increase in the mechanical performance of fly ash based geopolymer concrete by 20%. The geopolymer concrete made with CTR has a lower density than normal concrete by 19 %. Its bulk density ranged between 1873-2174 kg/m³ when the CTR/sand range from 10-30 %.
3. The employment of CTR as a partial replacement by volume of sand was found to a maximum reduction in the ultrasonic pulse velocity of geopolymer concrete by 20.5 and 30.4 for 2 and 4 mm CTR respectively at 28 days.
4. It was found that Quartz was the highest reaction peaks and lower peaks correspond to the Aluminum Mullites, and Merwinites, are also noticed. Trace amounts of calcium silicates were also found in the final product.
5. The XRD examination shows highest peaks intensities were observed at $2\theta = 28.1^\circ$ for control mix due to the presence of Quartz. After the addition of CTR, highest peaks intensities were

observed at $2\theta = 32.3^\circ$ for M10, M20 and M30 respectively.

6. SEM analysis revealed that the fracture surfaces for samples with CTR do not possess any cracking like that found in control. Further, increasing the CTR to 20% and 30% produces voids in the internal microstructure of geopolymer concrete.

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