



Article Effect of Rubber Heat Treatment on Rubberized-Concrete Mechanical Performance

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Abstract: To eliminate the unfavorable effect of the accumulation of end-of-life car tires on the environment, many studies have been conducted to recycle those tires in concrete as a partial or full replacement of its natural aggregates. However, the produced rubberized concrete suffers from low compressive strength due to low adhesion at the rubber/cement interface. Pre-treating of rubber surfaces before use in concrete is the most effective way to overcome this adverse effect on the concrete strength. Several studies introduced different methods to enhance rubberized-concrete strength through pre-treating rubber particles, especially when using a high content of rubber in concrete. This study presents the results of experimental work on the effect of heat treatment on crumb-rubber-concrete mechanical performance. Rubber contents of 40%, 60% and 80% of sand volume were the variables in this study. Workability, density, compressive strength, and impact resistance were the measurements in this experimental work. The results showed that using saturatedsurface dry (SSD) rubber can eliminate the adverse effect on concrete slump when using a high rubber volume or the heat-treated rubber. Using heat-treated rubber at 200 $^\circ$ C for 2 h as 40%, 60%, and 80% displayed compressive strength recoveries of 14.9%, 10.4% and 9.7%, respectively. Heat treatment of 40%, 60%, and 80% rubber contents increased the impact resistance for ultimate failure by 57%, 28%, and 7%, respectively, compared with those of the control mix. The thermal treatment enhanced the impact resistance at ultimate failure by 37%, 28%, and 15%, respectively, for mixes containing 40%, 60%, and 80% rubber contents compared with those of as-received rubber.

Keywords: rubber concrete; thermal treatment; workability; compressive strength; impact resistance

1. Introduction

Concrete structures subject to severe loading conditions are critical structural components. Many of these structures have been severely damaged or collapsed under severe loading due to inadequate strength, ductility, or toughness [1–9]. The low energy dissipation of conventional concrete structures is one of the main reasons behind this severe damage. Therefore, more ductile and energy-dissipative materials and systems are highly desirable to reduce this damage [10]. Crumb-rubber concrete (CRC) offers a ductile and energy-dissipative material that may be an alternative to the conventional concrete in concrete structures. CRC is a class of concrete in which crumbed scrap-tire rubber partly replaces concrete mineral aggregates. Using rubber in concrete can enhance its ductility, durability, damping ratio, impact resistance, and toughness [11–18]. However, it reduces its compressive strength, tensile strength, and modulus of elasticity [19–23]. The surface nature of rubber and its low hydraulic conductivity are the major reasons for the rubber-concrete's low strength, as they both cause poor adhesion at the cement/rubber interface [14,24]. In addition, the rubber contains zinc stearate, which is a part of tire formulation and it also causes poor adhesion of rubber to the surrounding concrete matrix. This zinc stearate creates a layer of soap that repels water [25–28].

Due to the large amount of tire rubber waste generated every year, the management of this type of waste became an environmental crisis due to the dumping of end-of-life tires



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Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). into landfills [29–31]. An accumulation of tires can catch fire easily, which is costly and difficult to extinguish [32,33]. Therefore, it is an urgent demand to decrease the number of tires disposed in landfills by recycling end-of-life tires in daily use product such as concrete. A significant number of studies have been carried out to investigate the performance of rubber in concrete as a partial or full substitute of its natural aggregates. This can reduce the amount of rubber entering landfills and conserve natural resources such as sand and stones [34,35].

Several approaches have been examined to reduce or eliminate the rubber deficiencies in concrete, such as pre-treating the rubber particles before using them in concrete, and/or adding some external additives as general enhancers for concrete mechanical properties. However, the experimental findings regarding the effectiveness of these approaches have been quite inconsistent and, in some cases, conflicting in the research literature to date. Balaha et al. [36] showed that CRC properties improved as cement content increases up to 400 kg/m³. However, the slump was negatively affected when using 400 kg/m³ compared with using 300 kg/m³ cement content. Using a 15% silica fume (SF) and Sodium Hydroxide (NaOH) solution pre-treatment of rubber particles increased concrete slump by 77% and 7%, respectively, increased compressive strength by 18% and 15%, respectively, and increased tensile strength by 9% and 6%, respectively. Youssf et al. [25] found that the losses in CRC compressive strength with higher cement content were less than when using lower cement content. In addition, when using pre-treated rubber, while the concrete slump and tensile strength decreased by 25% and 13%, the compressive strength and modulus of elasticity increased by 15% and 12%, respectively, compared to non-treated rubber. No effect was observed in their results when using SF except a slight increase in the compressive strength at a rubber content of 20% by sand volume. Other researchers have also reported success in improving the concrete compressive strength of CRC through a range of pre-treatment and additive methods including: Eldin and Senouci [37]; Pelisser et al. [19]; Güneyisi et al. [38]; Mohammadi et al. [39]; Su et al. [40]; and Hamza and Ghedan [41].

There have also been a number of studies that have reported negligible improvement or even a lowering of compressive strength despite pre-treatment or the use of additives. For example, Raffoul et al. [42] tried two different rubber pre-treatments. The first one was pre-washing with water and then air drying, and the second one was pre-coating with SF paste for 20 min before mixing with other concrete constituents. Their results showed that not only did their pre-treatment methods marginally affect the CRC strength, but they also resulted in the reduced flowability of the concrete. Other researchers who reported negligible improvements in compressive strength, even though they used pretreatments that were basically the same as those reported in the previous paragraphs, include: Deshpande et al. [43]; Tian et al. [44]; Li et al. [45]; Turatsinze et al. [46]; and Albano et al. [47].

Tian et al. [44] observed that rubber pre-treatment by inorganic salt Calcium Chloride $(CaCl_2)$ improved the mechanical properties of CRC; however, organic, acidic, and alkaline solutions did not effectively enhance CRC properties. Huang et al. [48] showed that rubber pre-treatment by a silane coupling agent followed by a cement paste coating could increase compressive strength by up to 110%. Dong et al. [49] used a similar method but their results showed only a 10–20% strength enhancement in concrete incorporating coated rubber compared to that with uncoated rubber. Abdulla and Ahmed [50] showed that rubber pre-treatment by Sulfuric acid (H₂SO₄) increased the rubberized mortar compressive strength by 2 times, but it negatively impacted other properties of the cement mortar. Xiong et al. [51] observed a noticeable improvement in the microstructures of cement hydrates at the rubber/cement interfacial transition zone when using a silane coupling agent solution (0.5–1.0% concentration) for pre-treatment. He et al. [52] showed that the oxidation and sulphonation of rubber particles significantly improved compressive strength by 48.7%. Akinyele et al. [53] noted that rubber in concrete affects not only mechanical but also chemical properties. They showed that increasing rubber in concrete decreased Ferrous

iron, Oxygen, Calcium, Aluminium, and Silicon elements; however, it increased Carbon and Sulphur elements which act as impurities during the hydration process.

Of the rubber pre-treatment methods, heat treatment was shown to be highly capable of recovering CRC strength losses, as reported by Abd-Elaal et al. [54], who investigated the influence of the heat treatment of rubber particles on CRC characteristics. They employed four different crumb-rubber sizes (0.425, 0.6, 1–3, and 2–5 mm) and replaced three different amounts of concrete fine aggregate (10%, 20%, and 40%) by volume. They found that rubber heat treatment at 200 °C for 2 h improved the CRC compressive strength by 25%, 40%, and 128% for 10%, 20%, and 40% crumb-rubber content, respectively. Furthermore, the size of crumb-rubber particles was discovered to have a substantial impact on the efficiency of heat treatment. The improvement was greater in the smaller particles than in the bigger ones. When compared to concrete with untreated rubber, the rubber treatment dramatically increased the compressive strength by 40%, 28%, 18%, and 3% when using rubber sizes of 0.425, 0.6, 1–3, and 2–5 mm, respectively.

As per the literature above and to the best of the researchers' knowledge, limited research has been carried out on the heat treatment of rubber in crumb-rubber concrete, especially with high rubber content. The proposed CRC mixes in this study offer several potential advantages over conventional concrete including saving natural resources, disposing of end-of-life tire rubber, and improving the performance and ductility of concrete subject to severe loadings.

2. Experimental Program

The cement used in this investigation was Elswiz Portland cement, Type: CEM-I 42.5 N according to Egyptian Standards ES 4756-1:2013 [55]. Size 10 mm dolomite stone with 2.63 specific gravity and 1560 kg/m³ bulk density was used as a coarse aggregate in this study. River sand with 5 mm size, 2.54 specific gravity, and 1730 kg/m³ bulk density was employed as a fine aggregate in the concrete. The rubber particles used were from "Nagaa Hamady", and their size varied between 0.125 mm and 2 mm, and were used to replace the concrete fine aggregate. The specific gravity and unit weight of the rubber used was 0.97 and 530 kg/m³, respectively. Figures 1 and 2 show the particle-size distribution of the rubber, sand, and dolomite stone used in this study. To improve the workability of concrete mixtures, superplasticizer (SP) type F according to ASTM C 494 [56] standards was used as a concrete high-water reducer. As per the manufacturer's datasheet, the superplasticizer has a specific weight of 1.08.



Figure 1. Particle-size distribution of fine aggregates (crumb rubber and sand).



Figure 2. Particle-size distribution of coarse aggregates (dolomite).

In the initial stage of this experimental study, crumb rubber was used as received in concrete mixes to investigate the required mixing procedures and workability. However, it was observed that the small-sized rubber used was adversely affecting the concrete workability due to its high water absorption. Therefore, it was decided to use the fine rubber in a saturated surface dry (SSD) condition. The fine rubber was pre-soaked in water for 24 h and then the rubber surface was left to air dry before mixing in concrete. Foil trays were used to thermally treat the rubber crumbs. The foil trays were partially filled with rubber to a thickness of 10 mm and then were inserted into an electric oven with a capacity of 60 L, a power of 2000 watts, and a temperature control of up to 250 °C. The oven had dimensions of 645 mm \times 430 mm \times 465 mm and contained four stainless-steel heating elements. After placing the rubber crumbs in the oven for 2 h, they were taken out and the cohesive parts were broken up to get ready for mixing in concrete.

Seven mixtures were designed and prepared according to Egyptian Standards ECP 203-2020 [57] to investigate the behavior of concrete containing heat-treated rubber. The Egyptian standard methodology followed the absolute-volume approach in designing the concrete mixtures. In this approach, the concrete mix is designed for a total volume of 1 m³, in which the summation of the absolute volumes (ingredient weight/ingredient specific gravity) of all concrete ingredients should equal the total absolute volume of concrete (1 m³). The concrete mixtures were designed using the absolute-volume method and are shown in Table 1. Rubber contents of 40%, 60% and 80% were the variables in this experimental investigation. The control mix contained natural aggregates, cement, SP, and water. Crumb rubber (as received) was used to partially replace sand in mixes F40, F60, and F80 with ratios of 40%, 60%, and 80%, respectively. The effect of the heat treatment of crumb rubber was evaluated in mixes F40T, F60T and F80T, which contained 40%, 60% and 80% heat-treated rubber, at 200 °C for 2 h.

Workability, density, compressive strength, and impact resistance were the measurements in this experimental work. All tests carried out in this study were according to the appropriate Egyptian Standards. A standard slump cone (100 mm \times 200 mm \times 300 mm) was used to measure the workability of each mix according to ES 8411-2:2020 [58]. The cone was filled by fresh concrete in three layers and each layer was compacted manually using a steel rod. The density was measured by weighing the mass of the concrete cube and dividing the mass by the cube volume according to ES 8411-6:2020 [59]. Three standard 150 mm cubes were used to measure the fresh density, hardened density, and compressive strength per mix. The concrete cubes were tested for compressive strength according to

F80T

ES 1658-6:2020, 2018 [60] on a compression machine with 200 ton capacity in which each cube was centralized within the machine axis and tested on well-finished cube faces. The impact resistance was measured according to ACI 544 [38] at a concrete age of 28 days. Ten discs from each mix were prepared with dimensions of 150 mm diameter and 50 mm thickness. The impact resistance was calculated as the total number of blows needed to split the concrete disc into two halves.

Mix	Dolomite	Fine Aggregate (kg)		Cement	SP	Water	Heat	
Code	(kg)	Sand	Rubber	(kg)	(kg)	(kg)	Treatment	
Control	1070	890	0	360	3	144	_	
F40	1070	534	131.8	360	3	144	No	
F60	1070	356	197.7	360	3	144	No	
F80	1070	178	263.6	360	3	144	No	
F40T	1070	534	131.8	360	3	144	Yes	
F60T	1070	356	197.7	360	3	144	Yes	
F80T	1070	178	263.6	360	3	144	Yes	

Table 1. Composition of concrete mixes (per 1 m³).

SP: Superplasticizer.

3. Results and Discussion

Table 2 displays the measured properties of the tested mixes including: slump, density, compressive strength, and impact resistance. The workability was measured once per mix, and both density and compressive strength were measured using three specimens per mix. The impact resistance was measured using ten specimens per mix. The average of each property with the corresponding standard deviation (SD) are presented in Table 2. The following sections will discuss the effect of the rubber content and rubber heat treatment on the measured concrete properties.

Mix _ Code	Slump (mm)		Fresh Density (kg/m ³)		Hardened Density (kg/m ³)		Compressive Strength (MPa)		Impact Resistance (Blow)		
	As Received	Heat Treated	Value	SD	Value	SD	Value	SD	First Crack	SD	Ult Crack
Control	245	245	2470	16.3	2350	24.5	43	2.94	9	2.59	14
F40	55	220	2250	20.4	2170	16.3	16	0.94	10	2.98	16
F60	24	215	2130	4.1	2070	8.2	11	3.09	8	2.21	14
F80	13	215	2020	13.9	1930	9.8	9	0.47	7	2.26	13
F40T	_	225	2255	12.2	2175	4.1	20	1.25	15	3.91	22
F60T	_	225	2133	2.4	2073	6.5	14	1.25	13	2.83	18

1930

16.3

Table 2. Measured properties of the concrete mixes.

SD: Standard deviation.

20.4

3.1. Workability

2025

215

The performance of the developed concrete in its fresh state was estimated by measuring the concrete slump, which leads to the identification of concrete workability. In stage 1, where rubber was used as received, the measured slump values for Control, F40, F60 and F80 mixtures were 245 mm, 55 mm, 24 mm and 13 mm, respectively, as shown in Figure 3. Using 40%, 60%, and 80% rubber contents decreased the concrete slump by 77%, 90%, and 95%, respectively. It can be inferred that there was a general reduction in slump values when rubber particles were used to replace sand. This is mainly ascribed to the higher water absorption of the rubber particles compared to that of sand. During the concrete mixing, the finer rubber particles absorb water to achieve the saturated surface dry

12

1.25

10

3.45

15

SD

3.90

5.65

5.80

2.40

6.21

4.75

3.60



(SSD) condition. This resulted in reducing the free water, thus making the overall concrete mixture less workable.

Figure 3. Slump values of the produced concrete with as-received rubber.

In stage 2 (rubber in SSD condition was used), the slump results of all the tested mixtures were very close regardless of the use of treated or untreated rubber. The lowest slump value was 215 mm for mixtures F60, F80, and F80T with a difference of 30 mm from the control mixture and a difference of 10 mm from the highest slump value recorded for concrete contained rubber. Figure 4 presents the measured slump values of the produced concrete with treated rubber. It can be observed from the figure that the thermally treated rubber could slightly increase the concrete slump. This could be due to the evaporation of chemicals from the rubber particles while heating, which were replaced with water when-soaking rubber to achieve the SSD condition. The water replacing the chemicals helped in increasing the movability of the rubber particle within the concrete mix and, hence, the slump increased.



Figure 4. Slump values of the produced concrete with SSD rubber.

3.2. Fresh and Hardened Density

The fresh density of concrete is an excellent predictor of hardened-concrete performance. The measured fresh density of concrete is plotted in Figure 5. The figure shows reductions in the fresh density of the concrete when using crumb-rubber aggregate. This is because the crumb rubber has a relatively low specific gravity. Using 40%, 60%, and 80% rubber contents decreased the concrete fresh density by 9%, 14%, and 18%, respectively. It has been observed that the fresh density slightly increased with the heat treatment of the rubber. When the dry chemicals and fibers (which are part of the rubber surface) burnt and evaporated from the rubber particles while heating and were replaced with water while soaking, the water, as a liquid, could have increased the overall weight of the rubber aggregate and, hence, the fresh density increased.





The measured hardened density of the produced concrete after 28 curing days is presented in Figure 6. It can be shown that using crumb rubber as a fine aggregate in producing concrete reduced the hardened density regardless of the heat treatment conducted. Using 40%, 60%, and 80% rubber contents decreased the hardened-concrete density by 8%, 12%, and 19%, respectively.



Figure 6. Hardened density of concrete mixes.

3.3. Compressive Strength

The concrete strength was determined at 28 days for all mixes in this study. The measured compressive strength values for Control mix and rubber–concrete mixes F40, F60 and F80 made with as-received rubber were 43 MPa, 16 MPa, 11 MPa, and 9 MPa, respectively, as shown in Figure 7. This means that increasing crumb-rubber content decreases concrete compressive strength. For example, with rubber content increases as 40%, 60% and 80%, the compressive strength decreased by 63%, 74% and 79%, respectively. This is consistent with a previous study by Batayneh et al. [61]. The reduction in the

concrete strength after incorporating rubber is related to several reasons, such as: (1) rubber has a Poisson's ratio approximately twice as high as that of concrete and has a Young's modulus as low as a 1/3 of that of concrete, which results in early concrete cracking due to the large difference in the concrete-materials deformations; (2) the low modulus of elasticity of rubber particles produces high internal tensile stresses that are perpendicular to the direction of the applied compression load, which cause early failure in cement mortar [62]; and (3) rubber has a specific gravity lower than concrete, causing rubber migration to the top surface of the concrete during concrete mixing, resulting in a non-homogeneous mix [22]. Figure 7 also shows an improvement in the compressive strength when using heat-treated rubber at 200 °C for 2 h. The mixes including treated rubber F40T, F60T, and F80T displayed compressive strength recoveries of 14.9%, 9.3% and 8.8%, respectively. The strength recovery is the ratio between the strength gained (by pre-treated rubber) to the strength lost when using as-received rubber. When heat-treated rubber is used, the strength recovery is attributed to the relatively higher bond between treated rubber particles and the surrounding cement paste. The heat-treated rubber particles have a stronger bond with the surrounding cement than that showed by the as-received rubber, which shows clear bond weakness at the rubber/cement interface. Figure 8 shows the adhesion at the rubber/cement interface before and after heat treatment. The observed relatively stronger adhesion is attributed to the ability of thermal treatment to burn out the unwanted impurities that are attached to the rubber particles [54]. These impurities consist of cords, steel and fibers [61,63]. Although processing the crumb rubber includes removing all constituents but rubber, it can still have remnants from those impurities within the rubber particles. Those impurities develop an immediate barrier against good contact with surrounding concrete materials. Consequently, it adversely affects the crack-bridging effect of rubber in rubberized concrete [64,65]. By removing these impurities by burning them out, the cement hydration and its penetration to the rubber surface is improved and, hence, there is better adhesion between them which resulted in better compressive strength.



Figure 7. Effect of rubber content on concrete compressive strength.



Figure 8. Microscopic scan of the rubber/cement interface before and after heat treatment.

3.4. Impact Resistance

The concrete impact resistance was determined at 28 days for all mixes in this study through a drop-weight test. The measured impact resistance values for Control mix and rubber concrete mixes F40, F60 and F80 compared with as-received rubber were 9 blows, 10 blows, 8 blows, and 7 blows, respectively, for the first crack and 14 blows, 16 blows, 14 blows, and 13 blows, respectively, for ultimate failure, as shown in Figure 9. This means that the impact energy for both the first crack and ultimate failure increased when replacing 40% of the sand volume with fine-crumb rubber, and then decreased when using 60% and 80% rubber content less than that of Control mix. Replacing 40% of concrete sand with rubber increased the impact energy by 10% for the first crack and 14% for ultimate failure. Increasing the replacement ratio to 60% and 80% decreased the impact energy by 11% and 22%, respectively, compared with that of Control mix. The increase in the impact energy when using 40% rubber can be attributed to the flexibility of the rubber material, which can help concrete to absorb the impact loads and delay its failure. However, with the increase in the rubber content, the reduction in concrete compressive strength became pronounced and the weak points (locations of rubber particles) within the concrete matrix became more connected, which caused the earlier failure of the surrounding cement paste and, hence, less impact resistance. This is in good agreement with Al-Tayeb et al. [66], in which using fine rubber in concrete was able to increase the impact resistance up to a certain replacement level, when the impact resistance decreased with increasing rubber content.



Figure 9. Impact resistance for CRC with as-received rubber.

Impact-resistance values for rubber–concrete mixes with heat-treated rubber at 200 °C for 2 h F40T, F60T, and F80T were 15 blows, 13 blows, and 10 blows, respectively, for the first crack and 22 blows, 18 blows, and 15 blows, respectively, for ultimate failure, as shown in Figure 10. Results show an increase in impact energy values by 66%, 44%, and 11% for mixes containing thermally treated rubber at 40%, 60%, and 80% rubber content, respectively, for the first crack and by 57%, 28%, and 7%, respectively, for ultimate failure compared with those of Control mix. Although the impact resistance decreased when increasing the rubber content beyond 40%, all the mixes with thermally treated rubber showed a higher impact resistance than that of Control mix. This can be attributed to the effect of the thermal treatment of rubber, which could relatively enhance the bond at the rubber/cement interface, which delayed the concrete failure under the impact load and, hence, had higher impact resistance.



Figure 10. Impact resistance for CRC with heat-treated rubber.

By comparing the impact resistances of CRC mixes that had as-received rubber with those of CRC mixes that had heat-treated rubber (Figures 9 and 10), it can be observed that the heat treatment at 200 °C for 2 h enhanced the impact resistance at the first crack by 50%, 62%, and 42%, respectively, for mixes containing 40%, 60%, and 80% rubber contents, and enhanced the impact resistance at ultimate failure by 37%, 28%, and 15%, respectively.

4. Conclusions

In this study, the influences of using untreated and heat-treated crumb rubber as a partial replacement of a fine aggregate were measured on crumb-rubber concrete mixes with high rubber contents. The following conclusions can be drawn:

- 1. Incorporation of as-received crumb rubber in concrete with contents of 40%, 60%, and 80% decreases its slump by 77%, 90%, and 95%, respectively. However, using saturated-surface dry (SSD) rubber showed an insignificant effect on concrete slump regardless of the rubber volume used, or the heat treatment conducted.
- Increasing the untreated rubber content to 40%, 60% and 80% decreased the compressive strength by 63%, 74% and 79%, respectively. Using heat-treated rubber (at 200 °C for 2 h) of 40%, 60%, and 80% displayed compressive strength recoveries of 14.9%, 10.4% and 9.7%, respectively.
- 3. Using 40% as-received rubber content increased the impact resistance by 14% for ultimate failure. Increasing the rubber content to 60% and 80% decreased the impact energy by 11% and 22%, respectively, compared with that of Control mix. Heat treatment of 40%, 60%, and 80% rubber contents at 200 °C for 2 h increased the impact

resistance by 57%, 28%, and 7%, respectively, for ultimate failure, compared with those of Control mix. The thermal treatment enhanced the impact resistance at ultimate failure by 37%, 28%, and 15%, respectively, for mixes containing 40%, 60%, and 80% rubber contents compared with those of the as-received rubber.

5. Future Recommendations

It is recommended that future studies try different rubber heat-treatment conditions and other rubber-treatment methods to compare the practicality and economics for use in the concrete market. In addition, the use of magnetized water as a replacement for concrete-mixing water in producing rubberized concrete is recommended. This can be a promising additive to improve the characteristics of rubberized concrete in addition to heat-treatment effects.

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