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PROPERTIES AND MICROSTRUCTURE OF RUBBERISED SILICA FUME CONCRETE

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ABSTRACT

This study investigated nine mixes containing 0 or 10% treated and untreated rubber as fine aggregate replacement and 0 or 15% silica fume as cement replacement. The fresh and mechanical properties were addressed, and these results were correlated using non-destructive testing, including rebound hammer and Ultrasonic Pulse Velocity (UPV). Two curing methods were used: normal and heating to a standardised 105°C. In addition, microstructure characterisation was carried out using X-ray Diffraction Analysis (XRD) and Scanning Electron Microscope (SEM). The XRD showed that the rubber must be washed after the pre-treatment of NaOH to avoid ettringite formation in the concrete pores. The results revealed that combining the crumb rubber pre-treatment and silica fume inclusion enhanced the mechanical properties, especially the compressive strength. The flexural strength reduction was less than or equal to 25%. However, the heat curing for 24 hours exhibited a strength loss of less than 8%, comparable to the expected strength loss for the control mix. The rebound hammer numbers closely correlated linearly with the compressive strength of the mixes. Poor agreement in predicting elastic modulus, compressive, flexural, and tensile strengths utilising the adopted equations from codes and guidelines used for predicting the mechanical properties as functions in concrete strength and modifications are recommended for practitioners.

Keywords: *Rubberised concrete, untreated and treated crumb rubber, non-destructive testing (NDT), microstructure, mechanical properties, silica fume*

INTRODUCTION

Rubber extracted from waste tyres, commonly called crumb rubber (CR), can be used in concrete as a partial replacement for aggregate, reducing its carbon footprint [1]. Several researchers [2]-[12] investigated rubberised concrete properties containing percentages of CR with findings related to low density and the possibility of utilising it against impact resistance loads. Gesoğlu and Güneyisi [2] explained the compressive strength reduction by the poor interfacial transition zone (ITZ) of the untreated crumb rubber in concrete. Segre and Joekes [3] and Raghavan et al. [4] reported that using a NaOH solution for treating the rubber before inclusion in concrete increased the bonding strength between the cement paste and the CR. This was confirmed by Khorrami et al. [5] while exploring the microscopic structure of the concrete matrix of treated rubberised concrete. On the contrary, Albano et al. [6] and Copetti et al. [7] did not find that rubber pre-treatment enhanced the strength of rubberised concrete. Jalal et al. [8] and Copetti et al. [7] reported that the 10% silica fume inclusion caused a slight increase in the strength for mixes containing 10% or 15% rubber replacement of coarse aggregate and did not with the untreated rubber, especially at later test ages.

Marques et al. [9] and Bengar et al. [10] reported another shortcoming: declining mechanical properties for samples containing up to 15% rubber as an aggregate replacement subjected to elevated temperatures up to 800°C. Saberian et al. [11] found an increase in the strength of the mixes with up to 2% CR rubber addition when heated above the melting point (286°C). Mousa [12] and Dihr et al. [13] prepared concrete with 3% or 5% CR and heated it to 105°C until a constant weight was reached with no further water evaporation. Fawzy et al. [14] tested samples with up to 16% CR after heating them to 70°C for 4 hours to simulate high daily temperatures. Others report non-destructive testing (NDT) on rubber concrete [6],[15]-[16] and correlate their results with the compressive strength for unheated concrete [17]-[18] and for those heated to 600°C [19] and 800°C [20]. However, NDT studies did not cite effects on rubberised concrete heated to a temperature of 105°C.

Generally, the rubber is allowed as fine aggregate replacement at a maximum of 10% [17],[21], with no clear design guidelines being developed for structural applications. Therefore, an empirical equation is required for estimating the mechanical properties of rubberised concrete [22], as the overestimation could jeopardise the entire structure's integrity. Suksawang et al. [23] and Nihal et al. [24] reported that the C/S ratio (coarse-to-fine-aggregate ratio) ratio is a key factor in evaluating the elastic modulus and the tensile strength. This study assesses the rubberised concrete while pretreated and untreated crumb rubber with silica fume under heated and unheated conditions. It also validates the adopted equations by codes and guidelines for predicting their mechanical properties.

EXPERIMENTAL PROGRAM

Materials and Mix Design

Cement

The cement used was Ordinary Portland Cement type CEM I, grade 42.5 MPa (N/mm^2), manufactured as per EN 197-1. The specific gravity is 3.15.

Silica Fume

The silica fume was an additive manufactured by Sika Inc. © as per EN 13263-1. The physical and chemical properties are found in the manufacturer's product sheet.

Sodium Hydroxide

The sodium hydroxide (NaOH) was prepared using 40 g sodium hydroxide pellets with a compound formula of HNaO and a 2.13 g/cm^3 density dissolved in 250 ml of distilled water. Thus, for 8 M NaOH, eight times 40 g weight of flux should be added in 1 litre to reach 8 M NaOH.

Fine and Coarse Aggregates

Sieve analysis of the fine and coarse aggregate used in the mix design is shown in Figure 1. The properties were determined according to ASTM C117, ASTM C127, and ASTM C128. The specific gravity is 2.60 and 2.65, with materials finer than 75 µm (sieve No.200) for fine aggregate is 2.53% and a maximum coarse aggregate size of 25 mm.

Treated and Untreated Crumb Rubber

Figure 1 shows the sieve analysis and grade size distribution for the CR. The CR particle size ranged from 0.6 to 4.75 mm. The rubber pre-treatment was performed by submerging the rubber in 8 M NaOH before mixing it into the concrete for 30 minutes. Previous researchers [5]-[10] followed this procedure.

Concrete Mix Design

The concrete mixes were designed following the ACI 211.1 [25]. Eight mixes containing CR and an additional control mix, without rubber, were designed for the test program. Table 1 presents the concrete mix design for a one-meter cube. The targeted slump and grade were 20 to 40 mm and 30 MPa with a water-to-cement ratio 0.4. The eight mixtures include 10% fine aggregate replacement by treated or untreated CR. For the mixes with silica fume, 15% silica fume cement replaced the exact weight of cement. As shown in Table 1, mixes 1 and 3 represent the use of untreated CR at 10% of fine aggregate replacement, while mixes 2 and 4 contained similar content for treated CR replacement. Mixes 3 and 4 contained 15% cement replacement by silica fume. The letter "H" was added to the code for heat-cured samples, e.g., "Mix 1H".

Sample Preparation

The concrete mixes were mixed in a mechanical mixer and poured into the 54 cube specimens of dimension 150 mm tested for compressive strength at 7- and 28 along with Rebound Number determination; in addition, 27 cylinder specimens of diameter 150 mm and height 300 mm for indirect splitting tensile strength and direct Ultrasonic Pulse Velocity (UPV) testing at 28 days.

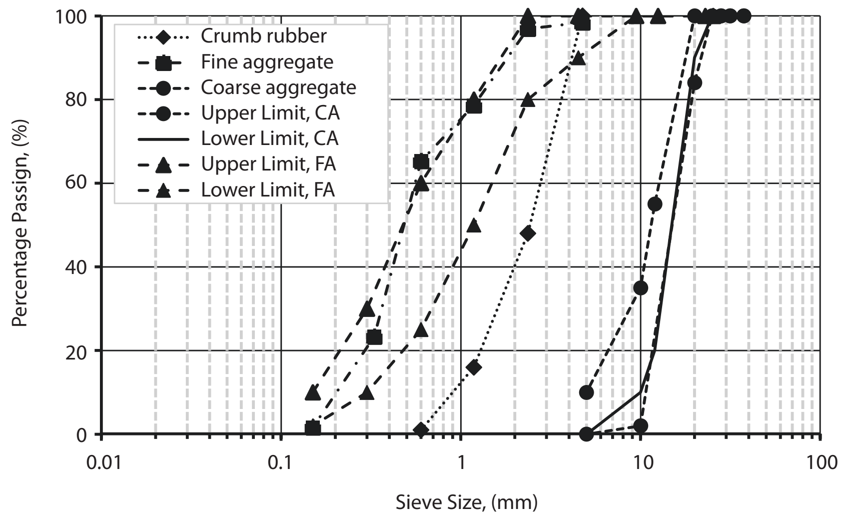


Figure 1 Sieve analysis of the crumb rubber, fine aggregate, and coarse aggregate used

Table 1 Concrete quantities used for concrete mix design in 1 m^3

I.D.	Crumb Rubber%	Silica fume%	Water, Kg	Cement, Kg	Coarse Aggregate, Kg	Fine Aggregate, Kg	Crumb Rubber, Kg	Silica fume, Kg	Curing Method#	Rubber treatment
Mix 0	0	0	200	500	1024	631	0	0	Normal	N/A
Mix 1	10%	0	200	500	1024	567.9	26.69	0	Normal	Non treated
Mix 1H	10%	0	200	500	1024	567.9	26.69	0	Heated	
Mix 2	10%	0	200	500	1024	567.9	26.69	0	Normal	Treated*
Mix 2H	10%	0	200	500	1024	567.9	26.69	0	Heated	
Mix 3	10%	15%	200	425	1024	567.9	26.69	54.76	Normal	Non treated
Mix 3H	10%	15%	200	425	1024	567.9	26.69	54.76	Heated	
Mix 4	10%	15%	200	425	1024	567.9	26.69	54.76	Normal	Treated*
Mix 4H	10%	15%	200	425	1024	567.9	26.69	54.76	Heated	

* Rubber treated in NaOH for 30 minutes
# Curing method applied at 28 days only

Finally, 27 prism specimens, 500 mm in length and 100 mm in the cross-section were prepared for flexural strength testing at 28 days, along with a UPV indirect test for concrete homogeneity. The mixes were compacted onto the vibrating table. The specimens were cured in water tanks for 7 and 28 days until testing as per BS EN 12390-2 after 24 hours of casting. Heat-cured samples were heated to 105°C at 27 days of age to obtain reliable results [13]. They were then tested with unheated samples at the age of 28 days.

Test Methods

A slump and density test for the fresh and hardened concrete mixes was conducted as per ASTM C143 and ASTM C642 at 28 days on unheated samples. After curing, the specimens were dried in the laboratory and then tested to show a typical cube, cylinder, and prism specimen during compression, splitting tensile, and flexural testing, respectively, using the universal testing machine of capacity 2000 kN as per BS EN 12390-3, ASTM C496, ASTM C78, as shown in Figure 2.

(a) (b) (c)

Figure 2 The (a) cube, (b) cylinder, and (c) prism specimens for compressive, splitting tensile, and flexural strengths determination till failure

NDT was conducted using rebound hammer number (RN) and UPV measurements and assessed according to the ASTM C805, ASTM C597, and [26]-[29], as shown in Figure 3.

(a)

(b)

Figure 3 The Schmidt hammer and UPV

EXPERIMENTAL RESULTS AND DISCUSSION

Slump

The slump results for the mixes are reported in Table 2. The inclusion of CR, treated or untreated, leads to a slump reduction compared to the control mix (Mix 0). The results show that the mixes with fine aggregate replaced by untreated CR (mix 1 and mix 3) had a lower slump reduction of 15% and 32% than those with treated rubber (mix 2 and mix 4) of 28% and 36, respectively. The inclusion of silica fume exhibited a higher reduction, as in mix 3 and 4, which are relevant to mix 1 and 2. Therefore, rubber pre-treatment and the inclusion of silica fume caused a reduction in mix workability, while the untreated rubber increased the reduction.

Parveen et al. [30] argued that the decreased workability due to the addition of CR could be attributed to the rough surface of the CR particles, which increases the friction between the CR and other ingredients in the concrete. Youssf et al. [31] also found that rubber pre-treatment further reduced the slump of the concrete as the surface roughness increased compared to non-treated rubber. They attributed this to the erosion of the rubber surface during pre-treatment, which slowed the movement of rubber particles within the concrete matrix. The extreme fineness of silica fume (20,000 m^2/kg) leads to its high water demand and hence reduces the workability of silica fume concrete [32].

Table 2 Typical slump and density values

Mix	Slump - mm	The density of hardened concrete at 28 days ρ (g/cm^3)
0 (control)	24.0	2.50
1	20.0	2.40
2	17.0	2.40
3	16.0	2.44
4	15.0	2.44

ElNemr [33] reported that adding silica fume to mortar reduced the workability by 16%, on average, relative to the control. Youssf et al. [31] reported an 88.2% reduction in a slump when 15% silica fume partially replaced cement in the mix. However, the reduction in a slump was only 25% in the mixes containing 20% rubber and 15% silica fume. Mahmod et al. [19]

reported a reduction in a slump by 8% with 10% crumb rubber inclusion. The rubber they used was not treated with NaOH.

Güneyisi et al. [34] partially replaced, by volume, fine and coarse aggregate with crumb rubber. The rubber used was untreated, and superplasticisers were added to enhance its workability. Besides, a reduction of 27.5% in a slump at 10% rubber inclusion with w/c=0.4, similar to the current study, they also reported reported a 34.8% slump reduction for the mix, where 15% silica fume partially replaced the cement. Similarly, Mavroulidou and Figueiredo [35] reported a 19% reduction in a slump when 10% rubber replaced fine aggregates. In contrast, Kaloush et al. [36] reported an 80% reduction in a slump with 16% rubber as fine aggregate replacement. The varied slump may depend on silica fume utilisation or rubber inclusion or treatment, with enough evidence that slump reduction with rubber is expected to occur.

Density

The density results of the mixes are shown in Table 2. The concrete density was slightly reduced by 4 and 2% for the mixes without and with silica fume, respectively, due to the inclusion of CR by 10%. However, the inclusion of silica fume decreased this reduction, as evident in mixes 3 and 4. In conclusion, the rubber treatment did not significantly affect the density.

Pham et al. [37] prepared mixes with 15% rubber by volume and concluded that the effect of rubber on density was negligible. In contrast, Ramli and Dawood [38] noted that partially replacing cement with 10% silica fume with superplasticisers led to a 13% increase in concrete density. Both of these reports align with the current study's findings. Siddique and Naik [9] suggested that the non-polar nature of rubber particles repels water and entraps air on the rubber surface, increasing the air voids and thus decreasing the density.

Mechanical Properties of Rubberised Concrete

Table 3 reports the mechanical properties of specimens at 28 days and only compressive strength at 7 days. The mixes developed their strengths since the 7 days' compressive strength values were approximately 76% of those at 28 days. The influence of the treated and untreated CR inclusion, the silica fume addition, and heat application on the mechanical properties of concrete are discussed in the following sections. The failure is comprised of those without rubber with less brittleness due to rubber chips acting as a bridge to control cracks, as shown in Figure 2.

Effect of Crumb Rubber Inclusion

Table 3 shows the average compressive strength results of the cube specimens at age 7 days. It can be observed that mixes 1, 2, 3, and 4 showed a reduction in compressive strength by 33%, 30%, 23%, and 20% compared to the control mix (Mix 0), respectively, at 28 days. Table 4 shows that the reduction in compressive strength was 34%, 31%, 27%, and 24%, respectively. Mavroulidou and Figueiredo [35] reported 32% and 40.9% loss of compressive strength at 7 and 28 days with 10% untreated rubber replacement without silica

Table 3 Compressive strength results

Mix, ID	Compressive strength - Age (MPa)		Rebound number, (x)	Predicted compressive strength (y) ($y = 0.8222 (x) + 11.791$) ($R^2 = 0.9488$)	Mechanical properties - strengths at 28 days (MPa)	
	7 days*	28 days			Splitting tensile	Flexural
0	30.0± 0.6	40.50±1.5	45	39.70	3.30±1.0	5.85±0.9
1	20.0±2.6	26.90±0.9	34	27.20	2.21±0.2	4.30±0.2
1 H	20.0±1.6	25.0±2.7	31	23.70	1.65±0.0	3.69±0.3
2	21.0±2.6	28.0±1.7	35	28.30	2.28±0.2	4.42±0.2
2 H	21.0±1.6	25.9±5.0	34	27.20	1.85±0.1	3.92±0.3
3	23.0±2.1	29.4±3.7	36	29.40	2.32±0.2	4.51±0.3
3 H	23.0±0.5	27.3±3.7	35	28.50	1.75±0.2	3.88±0.3
4	24.0±2.1	30.8±4.7	38	31.70	2.40±0.1	4.64±0.3
4 H	24.0±0.5	30.0±3.1	35	29.40	1.86±0.1	4.09±0.1

* The cube specimens for 7 days compressive strength were cured under the condition

fume. The results of Jalal et al. [8] indicated a loss in compressive strength of 46.0% and 46.1% for mixes with 10% untreated CR and without silica fume and a loss of 43.6% and 48.1% for mixes with 10% silica fume at 7 and 28 days, respectively.

From Table 3, the tensile strength was reduced by 33.0%, 31.0%, 29.8%, and 27.2% for Mixes 1, 2, 3, and 4, respectively, compared to the control mix (Mix 0). Ataria and Wang [40] reported a reduction of 21.4% in the tensile strength of concrete with 10% treated CR and recycled coarse aggregates. Gerges et al. [41] showed a 25-40% reduction in splitting tensile strength for mixes of different strengths containing 10% CR. Ganjian et al. [42] noted that the tensile strength of rubberised concrete should be higher than that without rubber due to the rubber's inelastic behaviour.

Similarly, Table 3 indicates that Mixes 1, 2, 3, and 4 exhibited a flexural strength loss of 26.4%, 24.5%, 23.0%, and 20.7%, respectively, compared to Mix 0. For a mix with 10% untreated rubber, Abusharar [15] reported a loss in flexural strength of 20.0% compared to the mix without rubber. The reduction in flexural strength due to rubber inclusion was less than that exhibited for compressive strength in the current study and Abusharar [15]. They argued that CR behaves like springs and delays the widening of flexure cracks, leading to a slight improvement in flexural behaviour. Akshay and Sofi [43] observed the former behaviour and recorded a 19 and 8% reduction for compressive and flexural strengths at 10% CR inclusion.

Effect of Heat Curing

The results in Table 3 show that heating slightly reduces compressive strength. Compared to the unheated samples, the heated samples lost 6.9%, 7.5%, 6.9%, and 2.5% of their compressive strength for mixes 1, 2, 3, and 4, respectively.

Tufail et al. [44] reported losses of compressive strength of plain concrete for heated specimens with and without CR inclusion. Their data show that a 21.0 MPa plain concrete without rubber, heated to 95 ± 2°C for two hours, reduced compressive strength by 7.8% in a mix containing limestone as coarse aggregates. Data from Shen and Xu [45] indicates that for 60 MPa concrete, the compressive strength is expected to be reduced by approximately 10% when heated at 105°C for one day. Behnood and Ziari [46] found that heating 60-80 MPa concretes, with and without silica fume, for 3 hours at 100°C resulted in a compressive strength loss of approximately 15%. Sancak et al. [47] reported that mixes with and without silica fume exhibited comparable strength losses due to exposure to 100°C. However, Moghadam and Izadifard [48] reported that a plain concrete mix and one with 10% silica fume reduced compressive strength by 28.11 and 14.68%, respectively, after being subjected to 100°C for 6.5 hours. For the concrete grade in the current study (20-30 MPa), Mix 4, containing treated rubber and silica fume, exhibited better residual strength after heating compared to the other samples.

For the tensile strength results, heating caused a reduction of 25.3, 18.9, 24.4, and 22.5% for mixes 1, 2,3, and 4, respectively. The corresponding reduction for flexure was 14.3, 11.3, 13.9, and 11.8 for mixes 1, 2, 3, and 4, respectively. Fawzy et al. [14] found that increased CR content from 0% to 16% by sand volume led to a decrease in splitting strength of 6.4% to 9%, respectively, at 70°C. The reduction in splitting strength for the same mixes was 17% to 21% when the concrete was heated to 200°C. For the same mixes, Fawzy et al. [14] reported that the flexural strength was reduced by 4%-5.6% when the concrete was heated to 70°C and by 19.8%-37.3% at 200°C compared with unheated concrete containing rubber 0%-16%, respectively. It can be seen that the exhibited reductions in mechanical properties in the current investigation due to heating are of the same magnitude order (concrete grade of 33 MPa and cement content of 400 kg/m^3) as those reported by Fawzy et al. [14].

Effect of Crumb Rubber Pre-treatment with NaOH

The mixes with treated CR (mixes 2 and 4) exhibited limited improvement in mechanical properties compared to their counterparts containing untreated CR (mixes 1 and 3) when normal or heat curing was applied, see Table 3. For example, for the unheated samples, the improvement in compressive strength at 28 days was 4.2% and 4.8%, respectively, for the samples with and without silica fume. The improvement in the splitting tensile strength was 3.2% and 3.4%, and the flexural strength was 2.8% and 2.9% for the samples with and without silica fume, respectively. The improvements in compressive, splitting tensile, and flexural strengths for the heated samples were 3.6% and 9.7%; 12.1% and 6.3%; 6.2% and 1.4% for the mixes with and without silica fume, respectively.

Copetti et al. [7] reported similar findings that rubber pre-treatment did not significantly affect the mechanical properties of their concrete. On the same note, Youssf et al. [31] reported approximately 17% improvement in compressive strength and 15% in splitting tensile strength with rubber pre-treatment. However, Muñoz-Sánchez et al. [49] noted a 2.4% improvement in 28-day flexural strength for mixes with CR treated with NaOH. Similarly, Balaha et al. [50] and Chiraz et al. [51] showed approximately 13% and 15% improvement in compressive strength in rubber concrete containing NaOH-pretreated CR, respectively. Saloni et al. [52] attributed this improvement to the treatment modification of the CR surface. Hence, the conflicting reports on the pre-treatment benefits depend on how it is applied, the duration, and whether the CR is washed with water after any pre-treatment.

Effect of Silica Fume Inclusion in Mixes with Crumb Rubber

From Table 3, the mixes (mix 3 and 1, mix 4 and 2) containing silica fume exhibited some improvement in mechanical properties compared to their counterparts without silica fume under normal or heat curing at 105°C. For compressive strength at 28 days of the unheated mixes, silica fume inclusion enhanced the strength by 9.3% in untreated rubber mixes. In contrast, the improvement was 9.9% for the mixes with treated rubber. For the heated mixes, the improvement was 9.4% and 15.8% for untreated and treated rubber, respectively.

For the splitting tensile strength, the improvements were 5% and 5.3% for unheated mixes containing untreated and treated rubber, respectively. For the heated samples, the improvements in splitting tensile strength were 6% and 0.05% for samples with untreated and treated rubber, respectively.

For the flexural strength, the enhancements were 4.9% and 5% for unheated mixes with untreated and treated rubber, respectively. In contrast, the observed enhancement for the heated mixes was 5.1% and 4.3% for mixes with untreated and treated rubber, respectively. Adding silica fumes benefitted compressive strength more than the other mechanical properties. However, in general, the enhancements were minor. Güneyisi et al. [34] reported a 9.6% and 12.5% increase in compressive and tensile strengths, respectively, with 15% silica fume in a mix with 10% rubber. The CR replaced fine aggregate in their mixes, and crumb rubber replaced coarse aggregate. When no rubber was utilised in the mixes, the compressive and tensile strength increased due to silica fume inclusion of 10.8% and 14.6%, respectively.

It appears that silica fume offered a lower contribution to mechanical properties for the rubber mixes. A similar variation was noted by Li et al. [53], who found that adding 10% silica fume to a mix without rubber increased the compressive strength by 41.6%, whereas when the same mix contained 20% CR, the increase in compressive strength was only 35.7%. Sun and Young [54] illustrated that, through a mix with 18% silica fume, by exploring the hydration process, which showed that only 44.1% of the silica fume had reacted at 28 days, and thus, the strength at this age is not profound. Indeed, Gesoğlu and Güneyisi [2] found that silica fume continued to contribute to the strength of rubberised concrete with extended curing of up to 90 days, which supports the silica fume effect on rubberised concrete in this study.

Results of NDT of Rubberised Concrete

Rebound Number Results

Figure 3 shows the relationship between the RN and compressive strength results for the test samples. From Table 3, the RN values recorded at 28 days for the mixes with 10% CR varied between 31 and 38, depending on rubber treatment, heat curing, and silica fume inclusion.

Mohammed et al. [17] reported an RN value at 28 days of 33 for a comparable mix to those in the current investigation. Akshay and Sofi [43] reported an RN value 39 for the 10% CR rubber mix. These values are similar in magnitude order to the current study. However, Kumar and Dev [29] reported an RN of 48 for a mix with 10% CR, but their mix had a higher strength of higher RN. A similar was deduced by Shaaban et al. [21]-[22],[55], evaluating the compressive strength and correlating the strength with air content that was deduced by permeability testing of concrete. From Figure 4, the RN is reasonably correlated with compressive strength values for the mixes in the current investigation, regardless of the considered parameters.

Ultrasonic Pulse Velocity (UPV)

Figure 5 shows the direct UPV test results. From Figure 5a, the test mixes exhibited different classification

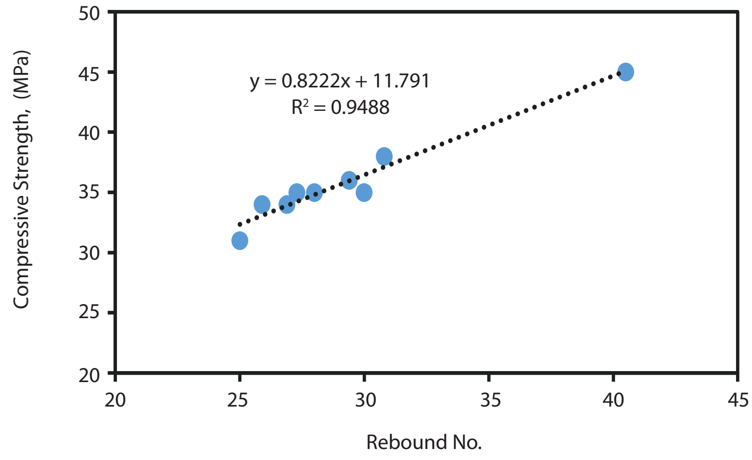
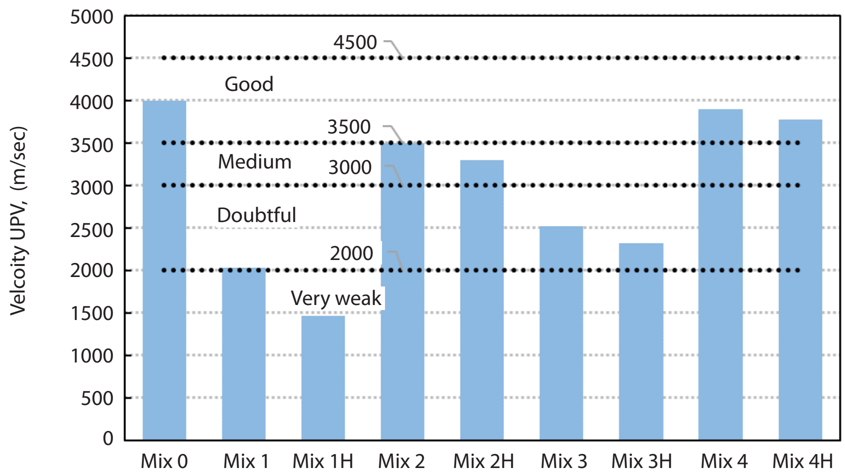
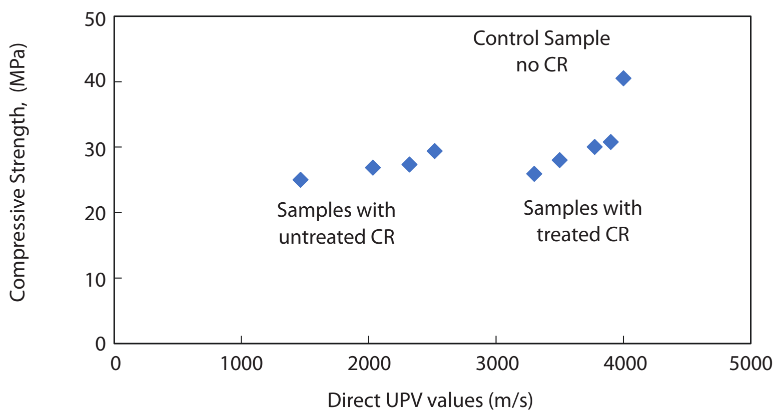


Figure 4 Correlation of rebound number with compressive strength at 28 days

qualities [26]-[29] as per BS 1881: Part 203. The inclusion of CR reduced the UPV by 49.1%. Figure 5b shows that the UPV results for the test mixes could not be correlated with the compressive strength on one trend line. A distinct difference is seen between mixes with untreated CR and those with treated CR.



(a) UPV values for the different test mixes showing classification for concrete quality



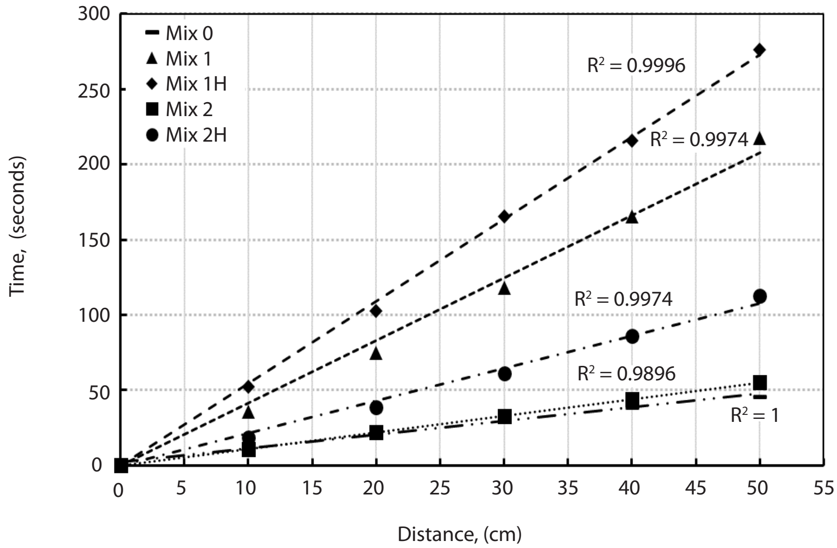
(b) Scatter diagram for the compressive strength and UPV values

Figure 5 Experimental results of direct UPV

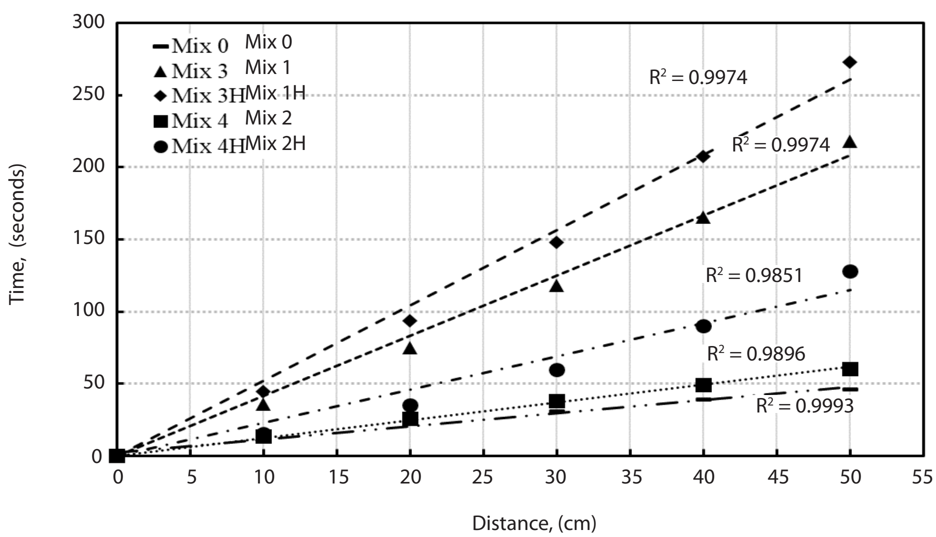
The indirect UPV test is applied to study the CR mixes' homogeneity, as shown in Figures 6a and 6b. The inverse of the slope of the lines showed high sensitivity to the mixed variables. Generally, applying heat curing lowered the UPV values or increased the wave propagation time in mixes 1H, 2H, 3H, and 4H, compared to the unheated samples (Mixes 1, 2, 3, and 4).

Albano et al. [6] found a reduction in UPV of 56% with similar CR content. In general, heating to 105°C did not affect the classification of any of the mixes; however, rubber treatment and/or the inclusion of silica fume had an evident impact on the classification. Rubber treatment affected the UPV values by 72.1% and 54.9% for the mixes without and with silica fume, respectively. These values contradict the findings of Najim and Hall [56], who reported no improvement in UPV with CR treatment, which will be discussed in the SEM results.

In the current study, the inclusion of 15% silica fume increased the UPV by 23.8% and 11.4% for mixes containing untreated and treated CR, respectively. Gesoğlu and Güneyisi [2] found that with 10% silica fume inclusion, the UPV was increased by 8% at 28 days in mixes with untreated rubber. The low improvement in UPV can be attributed to the lower percentage of silica fume compared to the current study. This leads



(a) Without silica fume



(b) With cement replacement by 15% silica fume

Figure 6 Indirect UPV for homogeneity testing to more pores in the matrix, as rubber is partially replaced for combined fine and coarse aggregates in their mixes. Mohammed et al. [17] also found that the UPV test was more realistic in evaluating the quality of the mixes with CR.

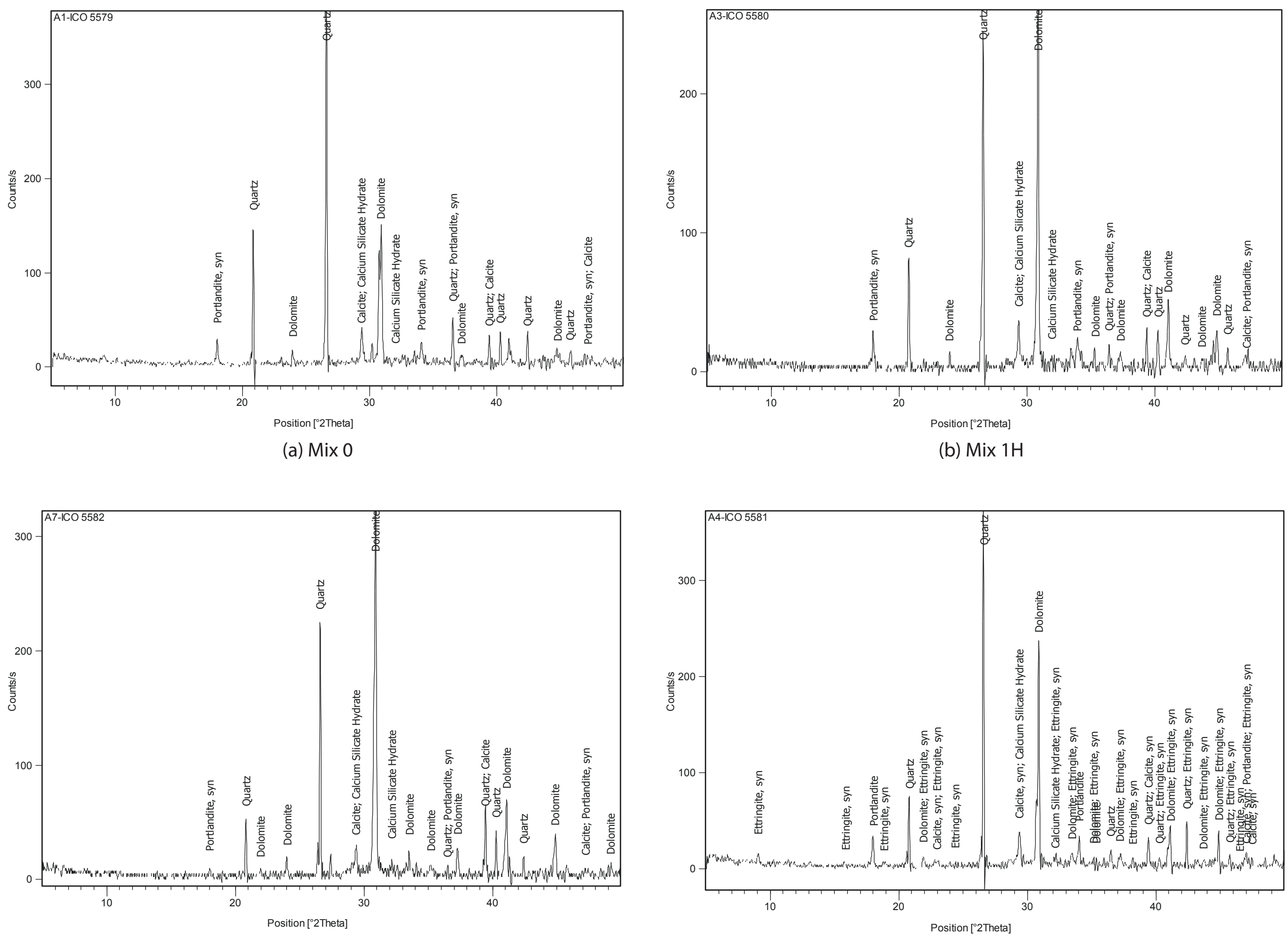
This reduction in UPV values was attributed to the voids and cracks due to heating, while improvement in mixes with silica fume and treated CR, which were evident in UPV results as corresponding to discontinuities of mortar porosity causing a delay in wave propagation [52].

Morphology for Limited Mixes

X-RAY Diffraction

Figures 7a, 7b, 7c, and 7d show X-ray diffraction for Mix 0, Mix 1H, Mix 2, and Mix 3H, respectively. Figure 7a, for the control mix without CR, shows the usual formation of Calcium Silicate Hydrate (C-S-H) and Portlandite (C-H). In addition, the usual peaks characteristic of quartz are present due to fine aggregates. In contrast, the Dolomite peaks are attributed to the coarse aggregate utilised, forming approximately 43% of the mix by weight, while the peaks characteristic of C-H and C-S-H were weak, indicating the presence of un-hydrated cement particles. This behaviour is reflected in the moderate strength of 40 MPa exhibited by Mix 0 at 28 days with 0.4 w/c mix containing 500 kg/m^3 of cement.

Figure 7b for Mix 1H, which contained untreated rubber and was heated for one day before testing, shows similar trends as Fig. 7a for Mix 0; however, it has slightly higher peaks of C-S-H and C-H than Mix 0. The latter indicates somewhat better hydration of the cement due to the heat treatment; however, the exhibited strength was still lower than Mix 0 due to the inclusion of CR.



(a) Mix 0

(b) Mix 1H

(c) Mix 2

(d) Mix 3H

Figure 7 XRD of rubberised concrete specimens

Figure 7c, for Mix 2 contained treated rubber and was not heated, shows that the characteristics of C-H were higher than the corresponding peaks for samples Mix 0 and Mix 1H. However, the peaks characteristic of C-S-H were lower than the corresponding peaks in samples Mix 0 and 1H. Furthermore, more significant amounts of ettringite (E) were observed. The increased C-H and the appearance of ettringite are probably due to the deposition of NaOH from treating the CR after adding it to the mix without washing.

Finally, Figure 7d, for the heated Mix 3H containing silica fume and untreated rubber, shows lower peaks characteristic of quartz and C-H. In contrast, the C-S-H phase's peak characteristics were higher than Mix 0; thus, it indicates that silica fume reacted with C-H, forming more C-S-H gel. In addition, lower quartz was exhibited because of the dilution effect of adding silica fume. However, the presence of CR still adversely affected the mechanical properties, and the silica fume contribution to the mechanical properties was less than expected. Mernerdaş et al. [57] developed a mix with the same type of cement and identical proportions as the current study and achieved nearer to 70 MPa cylinder compressive strength at 28 days. This behaviour indicates better hydration of the cement due to the heat treatment. In addition, a lower volume of quartz was present because of the dilution effect of adding silica fume.

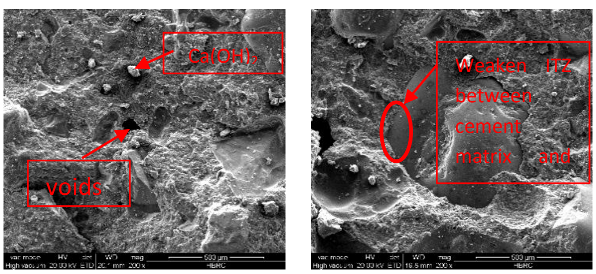
Scanning Electron Microscope (SEM)

Figures 8a, 8b, 8c, and 8d present the SEM for Mix 0, Mix 1H, Mix 2, and Mix 3H, respectively, similar to those of the XRD tested above. Figure 8a, for the control mix without CR, exhibits many voids resulting from unhydrated particles, inadequate mixing, or interlayer and capillary pores in the matrix. In addition, Figure 8a shows the weak bond between the cement matrix and fine aggregate particles reflecting poor microstructure, which is noticed through the mechanical properties of the control mix.

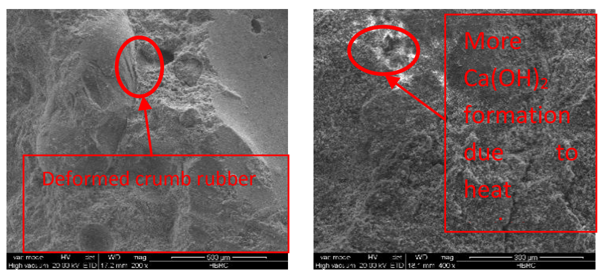
Figure 8b shows the SEM for Mix 1H, where the CR was untreated, and the mix heat cured at 105°C for 1 day. The CR particles were deformed from the figure, and white Ca $(OH)_2$ deposits were found in the matrix due to increased hydration.

Figure 8c shows the SEM of Mix 2, in which the NaOH was deposited as CR was treated. The deposits filled the voids created by the poor hydration around the CR particles. However, these deposits are not a substitute for hydration products.

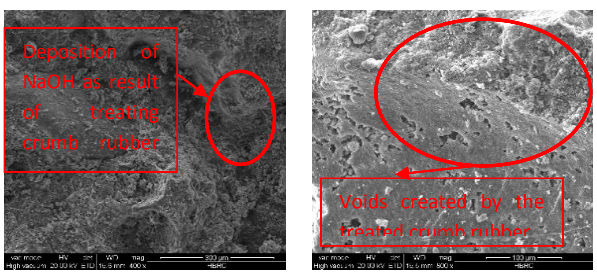
Finally, Figure 8d shows the SEM for Mix 3H, which was heat-cured and contained untreated CR and 15% silica fume. From Figure 8d, it can be seen that a better bond between the CR and cement matrix was formed. This behaviour may be attributed to the activation of



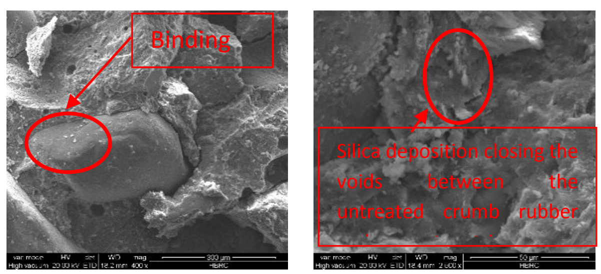
(a) Mix 0



(b) Mix 1H



(c) Mix 2



(d) Mix 3H

Figure 8 Scanning electron microscope (SEM) for specimens; (a) Mix 0, (b) Mix 1H, (c) Mix 2, and (d) Mix 3H the silica fume reaction, compensating for the CR size reduction during heat curing. Thus, this behaviour was reflected in the slight increase in strength in the mixes containing silica fume.

The X-ray micro-tomography images of Saberian et al. [11] showed that heating below the melting point of rubber caused shrinkage of CR particles, leading to a change in their shape. Therefore, voids were created, leaving empty spaces. Their results noted an increase in porosity due to the shape modification of the rubber particles upon heating. Wang et al. [58] concluded that the addition of rubber inhibited the cement hydration, thereby reducing the formation of C-S-H through their extensive exploration of the interface between sand and cement paste and between CR and cement paste. They explained this formation by the hydrophobicity of the rubber surface, leading to water retention around the CR, slowing hydration reactions, and poor bond between cement paste and rubber, similar to the findings reported by Albano et al. [6]. This conclusion contradicts the findings of Segre and Joekes [3], reported that the bond between treated and washed CR and the cement paste is improved compared to that of a mix containing untreated CR. Sugapriya and Ramkrishnan [59] support this finding and reported that raw rubber, possessing a smooth surface, exhibited a poor bond with the surrounding matrix. In contrast, treated rubber, where the zinc stearate layer has been removed, possessing a rougher surface, bonded well with the matrix. However, after pre-treatment with NaOH, rubber washing is essential to ensure good bonding with the matrix.

The deposition of NaOH in the pores of the treated samples in the current investigation significantly improved the UPV results by reducing the internal spaces in the matrix. This explains the varied enhancement of UPV in the current study compared to the observations of Najim and Hall [56]. Similar to the current study's findings, Li et al. [53] found that the bonding surface between the rubber and cement paste was weak, but partially replacing the silica fume helped to improve this weakness. The silica fume filled the gaps in the ITZ zone between the CR and cement paste, leading to a compact transition zone.

Design Codes, Guidelines, and Proposed Equations for Mechanical Properties Prediction

Relating concrete properties such as elastic modulus, tensile, and flexural strengths to compressive strength empirically is widely adopted by codes and guidelines [60]. These relationships are vital in defining material properties for ordinary concrete, whether in reinforced concrete structures design applications or using finite element analysis. Thus, the applicability of concrete relations is explored for the validity of rubberised concrete.

Table 4 of the addressed AAS concrete mixes were converted to equations originally developed for cylinder strength. From Table 4, AS3600 code equations underestimated the splitting tensile strengths by 14% on average. However, the other code and guidelines equations overestimated the tensile strengths of rubberised concrete. The overestimation for Eurocode 2, Fib model code, ACI-363, and ACI 318 ranges between 33 to 55%. For flexural strength, the codes and guidelines, Eurocode 2, Fib model Code, ACI-363, and ACI 318 underestimated the flexural strengths of rubberised concrete by 40 to 45% expected for code ACI-363 and Fib model Code are underestimated by 17 and 7%, respectively. For elastic modulus, only Eurocode 2 and Fib model Code consistently overestimated the experimental values, with 15 and 37%, respectively, while AS3600, ACI-363, and ACI 318 underestimated the elastic modulus by 46, 10 and 25%, respectively. These results highlight the significance of developing equations tailored specifically for rubberised concrete. However, the elastic modulus equations proposed by codes and guidelines have a coefficient of variation of 2%, except for the Fib Model Code.

In the current study, it is clear that the crumb rubber and silica fume replacement influence the proposed equation by codes and guidelines [60] and large discrepancies regardless of the heat at 100°C or water curing or whether the crumb rubber is treated or untreated. No clear trend is obvious for the codes and guidelines in predicting the tensile and flexural strengths, in addition to the elastic modulus expected for the AS3600 and ACI 318 in which one underestimated and the latter overestimated the precision of only tensile strength at a reasonable increase.

Table 4 Ratios between the predicted and experimental values of elastic modulus, tensile, and flexural strengths

ID	EXP. @ 28 days	Eurocode 2	Fib model	AS3600	ACI 363	ACI 318
			Splitting Tensile Strength			
Mix 0	2.37	1.22	1.15	0.65	1.06	1.01
Mix 1	2.21	1.38	1.32	0.76	1.24	1.17
Mix 1H	1.65	1.84	1.75	1.00	1.64	1.56
Mix 2	2.38	1.37	1.32	0.77	1.27	1.20
Mix 2H	2.33	1.37	1.31	0.76	1.25	1.19
Mix 3	2.02	1.53	1.45	0.84	1.37	1.30
Mix 3H	1.50	2.10	2.01	1.17	1.91	1.81
Mix 4	2.22	1.44	1.39	0.81	1.32	1.25
Mix 4H	1.86	1.72	1.65	0.96	1.57	1.49
	Average	1.55	1.48	0.86	1.40	1.33
	SD	0.26	0.25	0.15	0.24	0.23
	COV	17%	17%	17%	17%	17%
	Flexural Strength					
Mix 0	7.98	0.36	0.57	0.32	0.50	0.33
Mix 1	7.24	0.42	0.67	0.38	0.60	0.40
Mix 1H	4.48	0.68	1.07	0.61	0.96	0.64
Mix 2	7.57	0.43	0.69	0.41	0.64	0.42
Mix 2H	5.13	0.62	0.99	0.58	0.90	0.60
Mix 3	5.74	0.54	0.85	0.49	0.77	0.51
Mix 3H	3.53	0.89	1.42	0.82	1.29	0.85
Mix 4	6.48	0.49	0.79	0.46	0.72	0.47
Mix 4H	4.20	0.76	1.22	0.71	1.11	0.73
	Average	0.58	0.92	0.53	0.83	0.55
	SD	0.16	0.26	0.15	0.24	0.16
	COV	29%	29%	29%	29%	29%
	Elastic Modulus					
Mix 0	22635	1.19	1.06	0.57	0.93	0.78
Mix 1	24512	1.16	1.02	0.53	0.91	0.73
Mix 1H	24173	1.17	1.03	0.54	0.92	0.74
Mix 2	27316	1.11	0.96	0.53	0.87	0.73
Mix 2H	25809	1.15	1.00	0.54	0.90	0.74
Mix 3	25493	1.12	0.99	0.53	0.88	0.73
Mix 3H	25172	1.16	1.02	0.56	0.91	0.77
Mix 4	26426	1.12	2.07	0.54	0.88	0.75
Mix 4H	26120	1.14	3.19	0.55	0.89	0.75
	Average	1.15	1.37	0.54	0.90	0.75
	SD	0.02	0.72	0.01	0.02	0.02
	COV	2%	53%	2%	2%	2%

CONCLUSIONS

Crumb rubber and silica fume inclusion reduced the slump by 16, 42, 20, and 11.7% for untreated and pretreated rubber, respectively. A 10% crumb rubber inclusion reduced the density by 4%; however, the silica fume inclusion increased it. Treated and untreated crumb rubber reduced the compressive strength at 7 days by 25 and 28%, respectively. The silica fume inclusion enhanced the mechanical properties of those treated crumb rubber better than those of untreated ones. Heat curing at 105°C for 24 hours exhibited a strength loss of less than 8%, comparable to the control, which is reduced by silica fume inclusion. The RN and UPV values were linear trending when correlated with the compressive strength, with no sensitivity to any of the studied parameters. However, UPV was improved with rubber pre-treatment and silica fume inclusion, showing good homogeneity while indirectly UPV testing. The XRD showed the Ettringite formation due to NaOH deposition in the pores, which adversely affected the mechanical properties. The SEM observed the rubber pre-treatment and silica fume inclusion reduced voids in the matrix reflecting their compaction as per UPV. No addressed codes and guidelines estimated the elastic modulus, tensile, and flexural strengths regardless of the current parameters.

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