Optimisation of rubberised concrete with high rubber content: an experimental investigation

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Abstract

This article investigates experimentally the behaviour of Rubberised Concrete (RuC) with high rubber content so as to fully utilise the mechanical properties of vulcanised rubber. The fresh properties and short-term uniaxial compressive strength of 40 rubberised concrete mixes were assessed. The parameters examined included the volume (0 to 100%) and type of mineral aggregate replacement (fine or coarse), water or admixture contents, type of binder, rubber particle properties, and rubber surface pre-treatments. Microstructural analysis using a Scanning Electron Microscope (SEM) was used to investigate bond between rubber and concrete at the Interface Transition Zone (ITZ). This initial study led to the development of an "optimum" RuC mix, comprising mix parameters leading to the highest workability and strength at all rubber contents. Compared to a non-optimised concrete with 100% replacement of fine aggregates with rubber, the compressive strength of concrete with optimised binder material and moderate water/binder ratio was enhanced by up to 160% and the workability was improved significantly. The optimisation proposed in this study will lead to workable high rubber content RuC suitable for sustainable high-value applications.

Keywords: *rubberised concrete; tyre rubber; mix optimisation; sustainability; microstructure.*

1 Introduction

Worldwide tyre production is approximately 1.5 billion units/year and it is estimated that, for every tyre placed in the market, another tyre reaches its service life and becomes waste [1]. Over 300 million tyres reach their service life every year in the EU alone, i.e. practically one waste tyre per person. Tyres used in the automotive industry are made with 70-80% highly durable vulcanised rubber, which cannot be easily recycled. The inadequate disposal of rubber from scrap tyres is hazardous to the environment and human health and, as a result, stringent environmental legislations have been introduced to manage such "waste". The EU directives prohibit the disposal of scrap tyres in landfills and favour the reuse of waste materials ahead of recycling to minimise energy consumption (Landfill Directive 1991/31/EC [2] and Directive 2008/98/EC [3], respectively). This has increased efforts towards generating new applications for vulcanised rubber from scrap tyres [4-12]. In the past two decades, numerous studies have investigated the reuse of recovered tyre rubber in concrete to replace fractions of its mineral aggregates [5-12]. Whilst rubber is a valuable material with high strength, durability and elasticity, it can have a detrimental effect on some of the fresh and hardened mechanical properties of concrete.

In general, previous literature on the characteristics of RuC mixes is contradictory, highlighting the difficulty of achieving suitable mixes for construction. Whilst some researchers have reported satisfactory workability at all rubber contents and sizes [13, 14], others have measured zero slump at 50% [15] or 80% [16] aggregate replacement by volume. Previous experimental work often measures concrete workability through slump [17, 18]. Workability, however, is defined by the ease of mixing, placing and consolidating fresh concrete while maintaining adequate concrete homogeneity [19], and therefore, the overall stability (i.e. segregation and bleeding) of the fresh RuC mix has to be taken into account. Due to the relatively low density of rubber compared to mineral aggregates and cement, RuC cylinders with inadequate mix proportioning, consolidation or handling can exhibit a high concentration of rubber at the top upon vibration [20, 21]. The increase in porosity and entrapped air content (up to 30% at 25% rubber replacement by volume [20]) is conceivably the main reason behind the poor fresh performance of RuC [22]. Such increase may be attributed to rubber hydrophobicity, irregular shape, rough texture, contamination, interlock among rubber particles and excessive friction with cement paste [23, 24]. Other factors include flocculation among fine rubber particles, particle gradation and moisture content [22].

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The compressive strength of RuC reduces by up to 90% at high levels of rubber replacement (e.g. 100% sand replacement) [25]. The lower compressive strength of RuC can be attributed to the relatively high Poisson's ratio of rubber particles (nearly 0.5), the high porosity of the composite and the weak rubber-cement paste bond (or Interfacial Transition Zone, ITZ) [26, 27]. Other factors that reduce RuC strength include segregation, lower overall stiffness of the composite and casting and consolidation techniques [28]. Whilst such reduction is well documented in the literature [14, 17, 24, 25, 29-31], strength seems to be influenced by rubber content, size and properties, as well as mix parameters and proportions (i.e. water to binder ratio (w/b), type of chemical admixture and binder material). As a consequence, results from compressive strength tests on RuC cylinders are difficult to compare due to their large scatter (Figure 1).
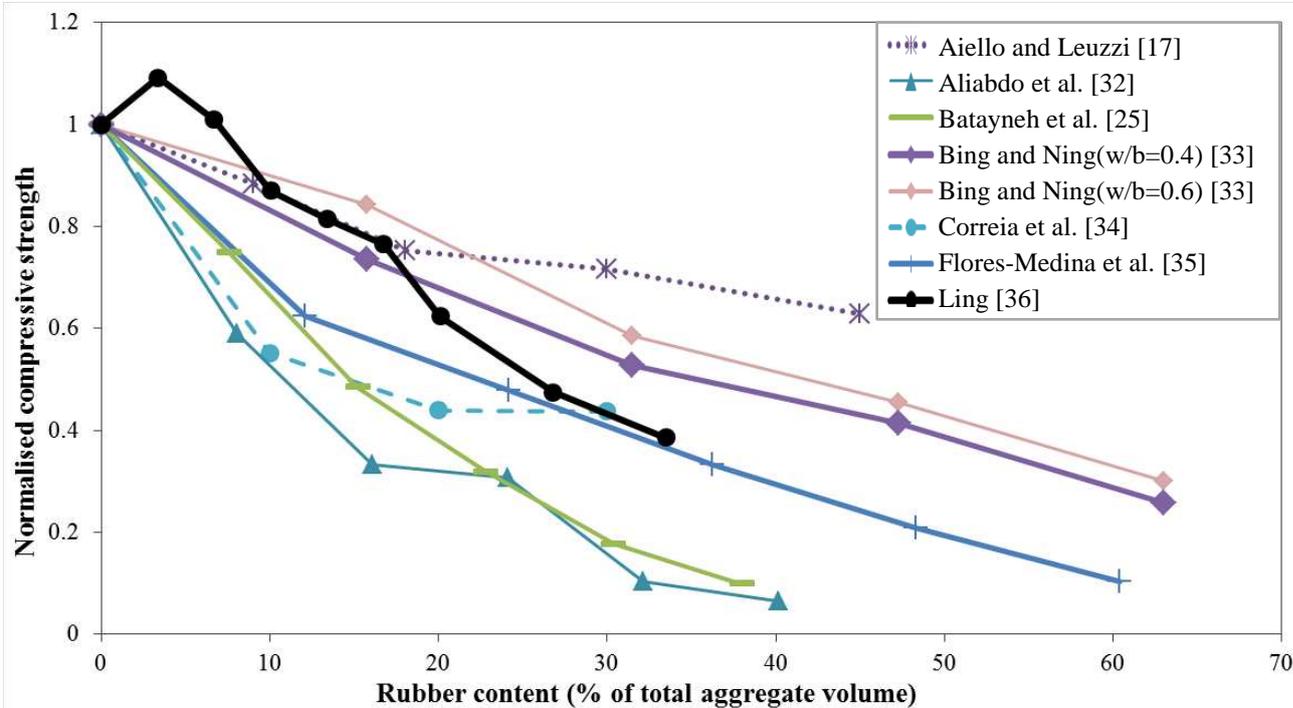
Figure 1: Normalised concrete compressive strength versus rubber content (data from [17, 25, 32-36])

Whilst rubber hydrophobicity and surface texture are known to weaken the bond between rubber and cement paste, the level of bond and load transfer at the rubber-cement paste interface is still unknown. Microstructural analysis of RuC revealed higher porosity in the matrix at the rubber-cement paste ITZ, as well as a larger ITZ, when compared to conventional concrete [37, 38]. In fact, the ITZ between rubber aggregates and cement paste increased from 6.65 μm to 13.44 μm at 10% and 50% sand volume replacement, respectively [38]. However, w/b was often varied with rubber content [38], which could

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possibly affect the hydration kinetics, mix porosity and ITZ density and width. Scanning Electron Microscopy (SEM) images have shown a lack of bonding (gap) between the rubber and cement paste at their ITZ, as well as limited hydration products surrounding the rubber particles [37-39]. Conversely, other studies show that rubber bonds well to the cement matrix [30, 40]. This good rubber-cement paste bond has been attributed to interlock at the rough surface of rubber particles [40].

It has been reported that zinc stearate (used to extend tyre service life in many developing countries) increases rubber hydrophobicity and leads to a porous and weak rubber-cement interface [41]. To improve rubber-cement paste chemical/physical bonding [18], several rubber pre-treatments have been investigated such as washing with water [21, 35, 42], polyvinyl alcohol [43], NaOH [13, 41, 44, 45], $Ca(OH)_2$ [46], silane coupling agents [47], organic sulphur compounds [48] or acid [40], as well as partial oxidation of the rubber surface [49], exposure to UV radiations [50] or pre-coating with cement [51], mortar [26], silica fume [39], limestone [52] or sand [45]. Despite some success in rubber pre-treatments (strength increase in the range of 3-40% [18, 26, 41, 51, 52]), results are often scattered and inconclusive, particularly when mixes with pre-treated rubber are not compared to mixes with as-received rubber [35, 42]. The effects of the pre-treatments on the concrete hydration reaction and long term durability have not been investigated. The pre-treatments are also often costly and time-consuming, and can only be justified if concrete performance is enhanced.

The significance of achieving an "ideal" packing of the concrete constituents on its rheology, durability and mechanical properties has been highlighted in the literature [53]. The packing of granular particles is influenced by their shape, texture, specific gravity, moisture condition and mixing, placing and consolidation techniques. To date, an appropriate method for characterising rubber particle properties does not exist, possibly due to the different types of rubber, levels of contamination and the lack of standard tests. For instance, the specific density of rubber reported in the literature varies between 0.5 and 1.3 [7, 28, 54]. The reported water absorption values vary between "negligible" [27, 55] up to 42.1% [33]. Nevertheless, rubber particles are broadly characterised with a flaky and elongated shape, a rough surface (i.e. high friction coefficient) and hydrophobicity that is likely to affect its packing with conventional aggregates [21, 56]. Due to their high surface area to weight ratios, it is also likely that ultra-fine rubber particles interact by surface and inter-particular forces [57]. To limit the influence of rubber size on

concrete particle packing, mineral aggregates are often replaced with rubber particles of similar grading [58].

Based on the previous discussion, it is evident that the lack of consensus in the literature, insufficient understanding of RuC performance and adverse effects of rubber on concrete properties limit the development/use of rubber in structural concrete applications. To date, the use of RuC has been mainly limited to:

1) Non-structural applications such as road barriers [7], thin overlays [8], concrete panels [9], paving blocks [29, 31] and applications for thermal and acoustic insulation [5, 6], and

2) Low-medium compressive strength structural concrete with reduced weight and increased ductility, as well as resistance to vibrations, impact and cyclic loads [6, 10-12].

To minimise the negative impact of rubber on concrete strength, the use of small volumes of rubber (up to 25% of the total mineral aggregates) is often proposed [16, 59, 60]. This inhibits the benefits that high-quality rubber can have on the concrete toughness and ductility [61, 62]. The use of large amounts of rubber in concrete can also have a positive environmental impact by reusing materials that would otherwise be considered waste. Therefore, from a structural and environmental perspective, further research is needed to mitigate the negative impact of large amounts of rubber on concrete characteristics.

This article investigates experimentally the behaviour of RuC with high rubber content so as to fully utilise the excellent mechanical properties of vulcanised rubber. The article describes an experimental programme that examines the parameters that influence the performance of RuC and describes a mix "optimisation" exercise. Subsequently, the study presents the main experimental results and analyses the factors influencing the fresh performance and compressive strength of RuC. Microstructural observations from scanning electron microscope (SEM) images are also presented and discussed. This research is part of the ongoing EU-funded collaborative research project Anagennisi (http://www.anagennisi.org/) that aims to develop innovative solutions to reuse all scrap tyre components. The results of this study are instrumental to understand the fundamental behaviour of RuC and contribute to the development of high-value structural applications.

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2 Experimental Programme

A total of 40 rubberised concrete mixes and 180 standard cylinders (100×200mm) were produced. To optimise the mix proportions and achieve a RuC with satisfactory fresh properties and short-term compressive strength, the first part of the experimental study (Part 1) examined RuC produced using different water to binder ratios (w/b), binder materials, specimen preparation techniques, rubber treatments and admixture contents at a fixed rubber content of 40% of the fine aggregate volume. Based on the results of Part 1, an 'optimum mix' was selected for the second part of the study (Part 2) to investigate the effects of rubber contents and sizes on the concrete compressive strength. In Part 2, the rubber replaced a) volumes of either fine or coarse aggregates (0%, 10%, 20%, 40%, 60%, 80% and 100%), or b) volumes of both fine and coarse aggregates (20%, 40% and 60% total aggregate replacement).

2.1 Material Properties and Characterisation of Rubber Particles

High strength Portland Limestone Cement CEM II – 52.5 N (10-15% Limestone) conforming to BS EN 197-1 [63] was used as main binder to reduce the carbon footprint of the mixes. Alternative binder materials including Silica Fume (SF) (Microsilica – Grade 940) and Pulverised Fuel Ash (PFA) (BSEN 450 – 1, Class N Category B LOI) were also examined. Two commercially available high range water-reducing admixtures were used [64, 65]. Round river washed gravel was used as coarse aggregate (Sizes: 5-10 mm and 10-20 mm; Specific gravity: 2.65; Absorption: 1.24%), whereas medium grade river washed sand was used as fine aggregate (Sizes: 0-5 mm; Specific gravity: 2.65; Absorption: 0.5%, Fineness modulus: 2.64). Mineral aggregates were replaced with rubber particles of roughly similar size distribution to minimise the impact on the packing of the concrete mix constituents. The rubber particles were recovered through mechanical shredding at ambient temperature and assorted in two types: a) fine (0-5mm) and coarse rubber (5-10mm) from car tyres and b) large rubber chips (10-20mm) from truck tyres. Fine rubber particles (0-5mm) were sorted in five size groups and a linear gradation was used to calculate their proportions. The rubber surface, particularly the large rubber chips, was jagged and contaminated with steel fibres and fluff, as shown in Figure 2b-c. The relative density of tyre rubber reported in the literature ranges from 0.51 [7] and up to 1.30 [28], therefore, the mass of rubber replacing the mineral aggregates was calculated assuming a relative density of 0.80.

Figure 2: (a) Fine rubber (0-5mm), (b) coarse rubber (5-10mm), (c) coarse rubber (10-20mm) and (d) mineral aggregates used in the experimental programmes.

Table 1 summarises a typical composition of rubber crumbs as reported by the provider, whereas Figure 3 shows the particle size distribution of rubber and mineral aggregates obtained according to ASTM C136 [66].

Table 1. Chemical characterisation of rubber granulates and powder

COMPOSITION INFORMATION OF INGREDIENTS* (data provided by ADRIA [67])
Polymers: 40-55%
Include Natural Rubber (NR), Styrene-Butadiene Rubber (SBR), Isoprene Rubber (IIR), Isobutylene-Isoprene Rubber (IIR), Halogen Isobutylene-Isoprene Rubber (modified IIR), Polybutadiene Rubber (BR), and Acrilonitril-Butadiene Rubber (NBR)
Carbon black: 20-25%
Other (softener, filler): 20-40%

*Percentages of each constituent will vary according to mixture
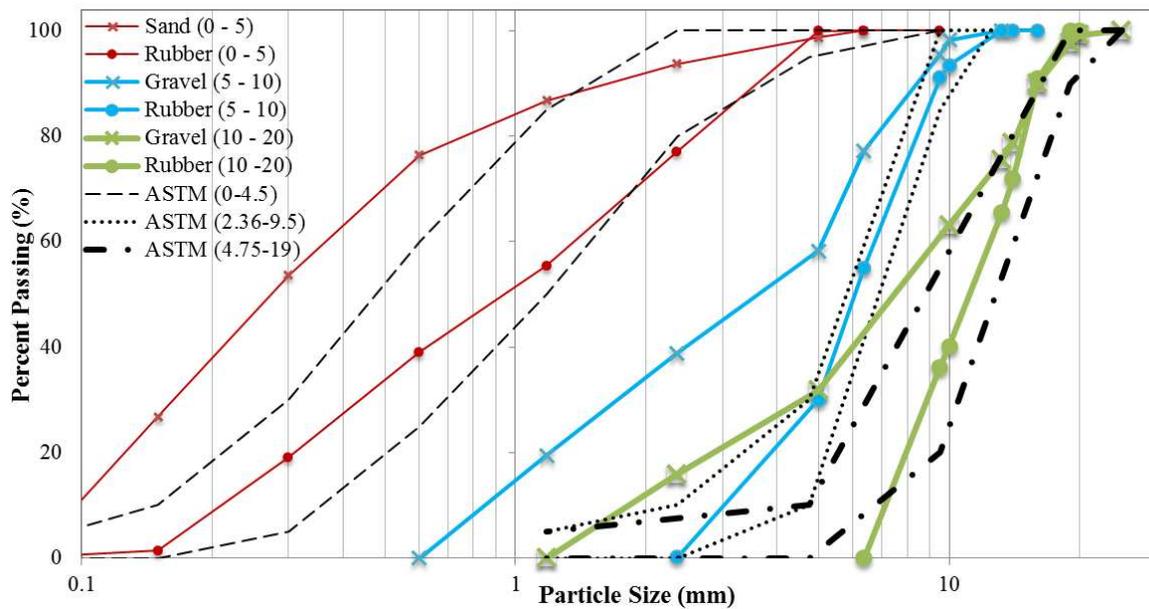
Figure 3: Particle size distribution of rubber and mineral aggregates and ASTM 33 boundaries for fine, medium and large coarse aggregates.

The rubber and mineral aggregate shape and physical properties were evaluated as follows: particle density and water absorption according to BS EN 1097-6-Annex C for lightweight aggregates [68]; bulk density according to BS EN 1097-3 [69]; and flakiness index according to BS EN 933-3 [70]. Particle density, water absorption and flakiness of fine rubber particles (0-5mm) were not evaluated as these particles float and agglomerate, thus giving misleading results. The aggregate properties are shown in Table 2.

Table 2. Physical and mechanical properties of rubber and mineral aggregates

Material (size in mm)	Apparent density (g/cm^3)	Oven dry density (g/cm^3)	SSD* density (g/cm^3)	Water absorption (%)	Specific gravity	Bulk density (g/cm^3)	Flakiness Index
Rubber (0-5)	-	-	-	-	-	0.4-0.46	N/A
Rubber (5-10)	1.1-1.2	1.0-1.1	1.1-1.2	5.3-8.9	1.1	0.45	6.6-8.3
Rubber (10-20)	1.1	1.1	1.1	0.8-1.3	1.1	0.48	10.4-17.5
Gravel (5-10)	2.69	2.60	2.63	1.24	2.65	1.51	7.1
Gravel (10-20)	2.69	2.60	2.63	1.24	2.65	1.58	9.7
Sand (0-5)	2.65	2.62	2.63	0.50	2.65	1.78	N/A

*Saturated Surface Dry

The results in Table 2 indicate that the water absorption of the rubber particles was unexpectedly high, e.g. up to 8.9% for the 5-10mm coarse rubber particles. This could be due to the difficulty of achieving

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surface dry conditions and to the presence of contaminants (steel, fluff and others), which was particularly high for the 5-10mm rubber particles. It is also observed that all rubber particles had a relatively low uncompacted bulk density (0.40 to 0.48 g/cm^3) in comparison to that of the mineral aggregates (1.51 to 1.78 g/cm^3). This can be attributed to the lower specific gravity of the rubber, but also to a lower packing of the rubber aggregates. The lower bulk density of ultra-fine rubber particles (compared to the larger particles) could be caused by surface inter-particular forces, which lead to flocculation and agglomeration among fine rubber [57]. Nevertheless, more accurate data on particle density and size of fine rubber are required before any conclusions can be drawn. The 10-20mm coarse rubber particles had higher flakiness compared to the replaced gravel (Table 2). This high flakiness indicates that the conventional sieve analysis is not suitable to measure rubber particle sizes. For instance, rubber particles measuring 40mm in one dimension and 20mm in the other orthogonal dimensions would still pass through a 20mm sieve, thus providing a misrepresentation of the actual particle sizes.

2.2 Mix Design and Parameters

A typical bridge pier mix design with a target 28-day compressive strength of 40 MPa was used as reference (mix O), according to the proportions shown in Table 3. This mix was selected because the RuC investigated in this study can potentially be used in applications where vibration damping and/or energy dissipation are needed (e.g. integral bridges, earthquake resistant structures, etc.). Mix O was designed to be highly flowable with relatively high cement content, water to binder ratio (w/b=0.423) and fine to coarse aggregate ratio. The concrete constituents were mixed as follows: 1) the aggregates (both mineral and rubber) were dry-mixed for 30 seconds. All mineral aggregates were Saturated Surface Dry (SSD), whereas the rubber particles were mixed dry and as-received (excluding the mixes with pre-treated rubber); 2) half of the mixing water was added and mixed for another minute; 3) the mix was allowed to rest for three minutes; 4) the binder materials (including cement and other pozzolanic materials) and the remaining mixing water were then added followed by a gradual addition of the admixtures and 5) the concrete was then mixed for another three minutes.

Table 3. Mix design for the original mix (O).

Material	Original mix (O) Quantity/m³
CEM II – 52.5 MPa	425 kg
Aggregates 0/5mm	820 kg
Aggregates 5/10mm	364 kg
Aggregates 10/20mm	637 kg
Fine aggregate : coarse aggregate	1 : 1.22
Water	180 l
Plasticiser (P)	2.5 l
Superplasticiser (SP)	5.1 l

2.3   Part 1: Mix Optimisation

The original mix O was very segregated, non-homogeneous and non-cohesive when rubber was incorporated and, consequently, various mixes were attempted to achieve improved fresh properties and short-term compressive concrete strength. Table 4 summarises data from representative mixes in Part 1 of the experimental programme while Table 5 shows the rubber quantities used. In Table 4, the trial mixes are identified according to the different parameters examined: water to binder content (w/b=0.3-0.38 – mixes A), admixture content (B), rubber pre-treatments (C) and binder material (D). The number in the ID represents the mix trial number. For comparison purposes, a fixed rubber content of 40% was used to replace the sand aggregates (by volume) in all trial mixes. In this initial study, the parameters leading to the best mix performance in terms of workability and compressive strength were selected as the "optimised mix" parameters. Additional rubber contents of 10% and 100% were used to replace aggregates in the original mix O and mix D. Note that the latter is the "optimum mix" selected based on the fresh and hardened concrete performance, as described in sections 3.1-3.7. Two rubber pre-treatments were examined. In mixes C.1 and C.2, the rubber was pre-washed with water to remove surface impurities, air dried and then stored in a closed container under standard laboratory conditions to maintain relatively constant moisture throughout the study. In mix C.3, the rubber was pre-coated with silica fume (SF) mixed with some water (10% of the cement weight). The pre-coated particles were then allowed to rest for 20 min before they were mixed with the aggregates and remaining concrete constituents, following the sequence described in section 2.2.

Table 4. Representative trial RuC mixes examined in Part 1 (Aggregate quantities shown in Table 5).

Mix I.D.	Cement (kg/m^3)	SF (kg/m^3)	PFA (kg/m^3)	Fine aggregate replacement (%)	w/b	Other varied parameters
O.1	425	-	-	0	0.423	-
O.2	425	-	-	10	0.423	-
O.3	425	-	-	40	0.423	-
O.4	425	-	-	100	0.423	-
A.1	425	-	-	40	0.38	-
A.2	425	-	-	40	0.35	-
A.3	425	-	-	40	0.32	-
A.4	425	-	-	40	0.30	-
B.1	425	-	-	40	0.423	Admixtures reduced by 20%
B.2	425	-	-	40	0.423	SP reduced by 40%
B.3	425	-	-	40	0.423	P reduced by 80%
C.1	425	-	-	40	0.38	Rubber pre-washed
C.2	425	-	-	40	0.35	Rubber pre-washed
C.3	340	42.5	42.5	40	0.35	SF as pre-treatment
D.1	340	42.5	42.5	0	0.35	-
D.2	340	42.5	42.5	10	0.35	-
D.3	340	42.5	42.5	40	0.35	-
D.4	340	42.5	42.5	100	0.35	-

2.4 Part 2: Variation in Rubber Contents

Based on the results from Part 1, the "optimum mix" D was selected to carry out an in-depth parametric study in Part 2 of the experimental programme. Rubber contents were varied from 0 to 100% of the fine aggregate (FA) or coarse aggregate (CA) volume. A combined replacement of both fine and coarse mineral aggregates (20%, 40% and 60% by volume) was also examined. Table 5 summarises the rubber and mineral aggregate proportions used for the RuC mixes examined in Part 2. All other mix parameters were fixed to the optimised mix proportions (mix D, Table 4). In Table 5, the mixes are identified with an ID that indicates the volume of rubber replacing aggregates in percentage (0%-100%) followed by the type of aggregate replacement, i.e. "FR" for rubber replacing fine aggregates (0-5mm) or "CR" for rubber replacing coarse aggregates (5-20mm). The IDs 20CR20FR, 40CR40FR and 60CR60FR identify mixes with 20%, 40% and 60% combined replacement of CR and FR, respectively.

Table 5. Proportions of rubber and mineral aggregate at different levels of replacement

Replacement Type	Mix I.D.	Rubber Mass (kg/m^3)		Mass of CA^a (kg/m^3)	Mass of FA^a (kg/m^3)
		CR	FR		
None	Plain	-	-	1001.0	820.0
Fine Rubber (FR)	10FR	-	24.8	1001.0	738.0
	20FR	-	49.5	1001.0	656.0
	40FR	-	99.0	1001.0	492.0
	60FR	-	148.5	1001.0	328.0
	80FR	-	198.0	1001.0	164.0
	100FR	-	247.6	1001.0	0.0
Coarse Rubber (CR)	10CR	30.2	-	900.9	820.0
	20CR	60.4	-	800.8	820.0
	40CR	120.9	-	600.6	820.0
	60CR	181.3	-	400.4	820.0
	80CR	241.8	-	200.2	820.0
	100CR	302.2	-	0.0	820.0
CR & FR	20CR20FR	60.4	50.0	800.8	656.0
	40CR40FR	120.9	99.0	600.6	492.0
	60CR60FR	181.3	148.5	400.4	328.0

^{a}CA = coarse aggregate, FA = fine aggregate

2.5 Specimen Preparation

A total of 180 standard concrete cylinders (100×200mm) and 30 cubes (100mm) were cast according to BS EN 12390-2 [71]. The cubes were cast to examine the development of axial compressive concrete strength for the highest rubber content (60CR60FR) at 3, 7, 14, 28 and 52 days. The specimens were cast in two layers and vibrated on a vibrating table (15-20s per layer). After casting, all readers to pinpoint the lack of consensus, insufficient understanding and technological issues associated with RuC behaviour specimens were covered with plastic sheets and kept under standard laboratory conditions for 48hrs until demoulding. The specimens were then stored in a mist room until 24hrs prior to testing. As the casting face of most RuC cylinders was uneven, two methods for cylinder surface preparation were examined: 1) cutting and grinding the cylinder surface according to BS EN 12390-3 [72], and 2) casting of gypsum caps (ASTM C617 [73]). Figure 4a-b show the concrete cylinders before and after casting the gypsum caps. The caps failed prematurely during the tests, leading to local crushing and failure at the top/bottom of cylinders. Conversely, surface cutting and grinding (Figure 4c-d) prevented local crushing and was used for testing the cylinders presented in this study.
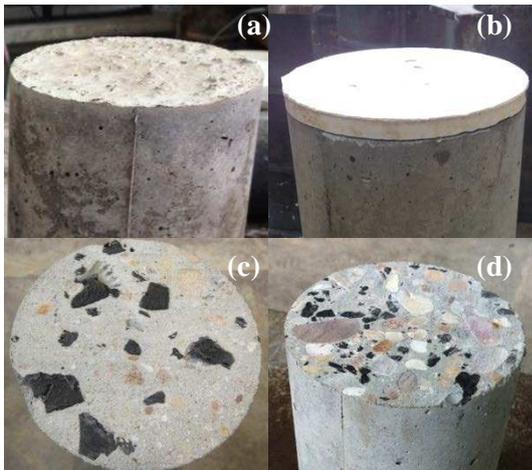
Figure 4: Cylinder without preparation (a), cylinder with gypsum cap (b) and cut cylinders from D mix with 10%CR (c) and 100% FR (d).

2.6 Test setup and instrumentation

As no standard method exists for measuring the fresh properties for RuC, these were evaluated using slump tests (BS EN 12350-2 [74]), flow table tests (BS EN 12350-5 [75]), or both depending on a visual assessment of the suitability of the test for each mix. Additionally, a visual stability index (VSI) was used to examine segregation and bleeding and to classify the mixes in descending order of stability (from 0 to 3) according to ASTM C1611 [76]. The hardened concrete density was obtained at the date of testing after air drying for 24hrs.

The cylinders and cubes were tested in uniaxial compression using a cube crusher of 3,000 kN capacity at a loading rate of 0.6 MPa/s according to BS EN 12390-2 [71]. However, the loading rate was reduced to 0.1 MPa/s to prevent premature failure of (weaker) cylinders with higher rubber contents (above 80% sand or gravel replacement). To speed up the experimental programme, 150 cylinders were tested after 7 days of casting, while the rest were tested after 28 days. Two cylinders were tested for each mix in Phase 1 of the experimental programme, whereas at least four cylinders were tested for each mix in Phase 2 to account for material variability.

High resolution scanning electron microscopy (SEM) and Energy dispersive X-ray Spectrostropy (EDS) analyses were used to observe the microstructure of selected RuC samples. The images were obtained in backscattered electron (BSE) imaging mode. Cylindrical cores (25 mm dia×10mm height) were extracted by cutting at the mid-height of the RuC cylinders and then coring at their centre. The face of each core was polished manually using commercial sanding paper to achieve a surface roughness of about 6 μm.

The samples were then polished gradually using diamond paste of 6, 3, 1 and 0.25 μm, and washed with isopropanol using an ultrasonic cleaner.

3 Results and analysis

Table 6 reports the following results from Part 1 of the experimental programme: a) slump of fresh mixes (when measurements were possible), b) average flow values, c) segregation, and d) 7-day compressive strength. Table 7 summarises the same results for Part 2 of the experimental programme, as well as the specific gravity and corresponding standard deviation (SD) of the 7-day compressive strength results. The following sections summarise the most significant observations of the testing programmes and discuss the results listed in Tables 6 and 7.

Table 6. Results - Part 1 of the experimental programme

Mix I.D.	Slump (mm)	Flow (mm)	Segregation (VSI)	Compressive strength at 7 days (MPa)
O.1	N/A[a]	700	0	46.8
O.2	N/A[a]	700	0	34.8
O.3	N/A[a]	685	2	14.1
O.4	N/A[a]	485	3	3.7
A.1	190	520	0	21.8
A.2	110	440	0	22.4
A.3	N/A[a]	N/A[a]	1	31.9
A.4	N/A[a]	N/A[a]	3	-[b]
B.1	N/A[a]	640	1	-[b]
B.2	N/A[a]	575	0	-[b]
B.3	N/A[a]	580	0	-[b]
C.1	N/A[a]	495	1	19.8
C.2	150	425	0	26.2
C.3	170	495	1	28.1
D.1	230	575	0	61.7
D.2	215	560	0	53.4
D.3	190	530	0	31.7
D.4	0	N/A[a]	2	9.6

[a] Mixes where flow or slump measurements were not possible
[b] No cylinders cast

Table 7. Results - Part 2 on optimum mix D with different rubber contents

Mix I.D.	Slump (mm)	Flow (mm)	Specific Gravity (SG)	Compressive strength 7day (MPa)	SD (%)
Plain	230	575	2.48	61.7	6.7
10FR	215	560	2.41	53.4	3.9
20FR	230	570	2.35	43.2	9.9
40FR	190	530	2.3	31.2	0.4
60FR	180	495	-	20.6	5.1
80FR	130	465	-	14.7	4.0
100FR	0	N/A[*]	2.13	9.6	7.4
10CR	N/A[*]	590	2.4	45.9	6.6
20CR	N/A[*]	535	2.34	32.7	18.5
40CR	45	N/A[*]	2.22	25.3	15.9
60CR	N/A[*]	510	2.2	15.8	27.1
80CR	40	N/A[*]	2.06	14.3	9.4
100CR	70	380	1.98	8.7	15.9
20CR20FR	210	490	2.22	32.0	3.2
40CR40FR	185	-	2.05	10.7	0.0
60CR60FR	40	410	1.94	7.1	16.8

[a] Flow or slump measurements were not possible

3.1 Fresh Properties

The fresh properties of RuC mixes changed significantly with the addition of rubber, and therefore the mix design was adjusted to achieve a good flow and no segregation. Figure 5 shows the flow table results of mixes O and D (with CR or FR replacement) as a function of the total volume of replaced aggregate. The results indicate that rubber contents of 0% to 10% FR did not change the flow of the original mix O. However, the flow reduced by 30% at a 100% FR replacement, which equals 45% of the total aggregate volume. The latter mix was very harsh, unworkable and segregated, as shown in Figure 6a. Compared to the plain mix O, the plain mix D had much lower flowability (575 mm at 0% rubber replacement). Flow reduced by 34% for mix D with the highest rubber content (100% CR, or 55% replacement of the total aggregate volume). Despite the high rubber content in mix D (55% total aggregate replacement compared to 45% in mix O.4), the former was more cohesive and homogeneous, as shown in Figure 6b.
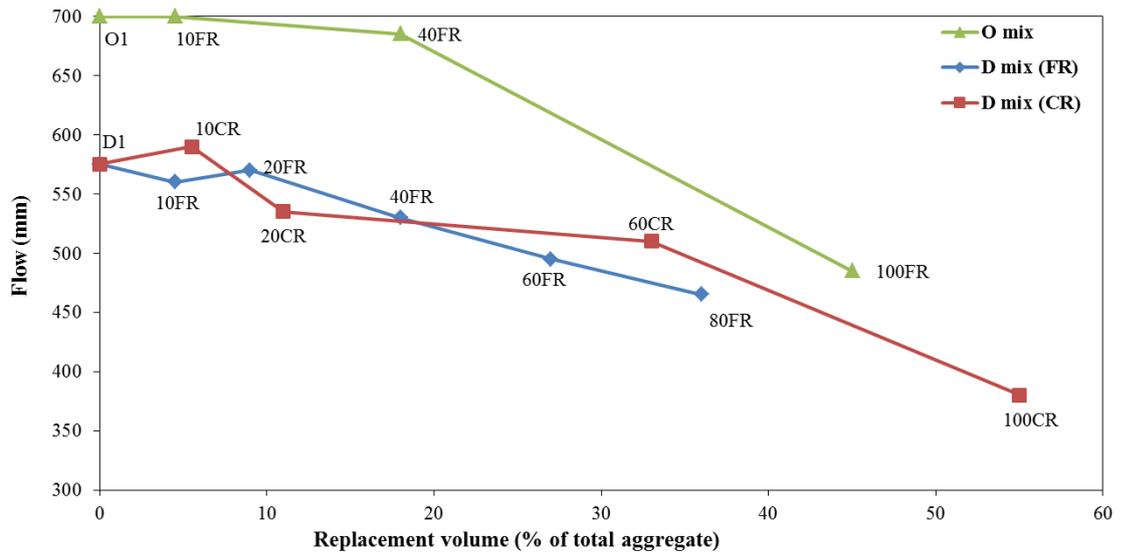
Figure 5: Flow table results for mixes O and D as function of the total aggregate volume replacement

Figure 6: View of flow table test of (a) mix O at 100% FR and (b) mix D at 100% CR.

The slump/flow values and levels of segregation in Table 6 show that most mixes achieved acceptable flowability for casting and compacting purposes (except mixes O.4, A.3 and A.4). However, segregation and bleeding were evident in RuC mixes, as confirmed by: 1) shear failure in slump test, particularly in dryer mixes (an indication to harshness and lack of cohesion, Figure 7a), 2) separation of coarse aggregates from finer particles in flow table tests (Figure 7b), and 3) the presence of a mortar halo. A gleam was also observed at the surface of RuC mixes with high water content (B.1 and O.3), indicating bleeding. This can be attributed to rubber hydrophobicity, poor particle grading and concrete porosity.

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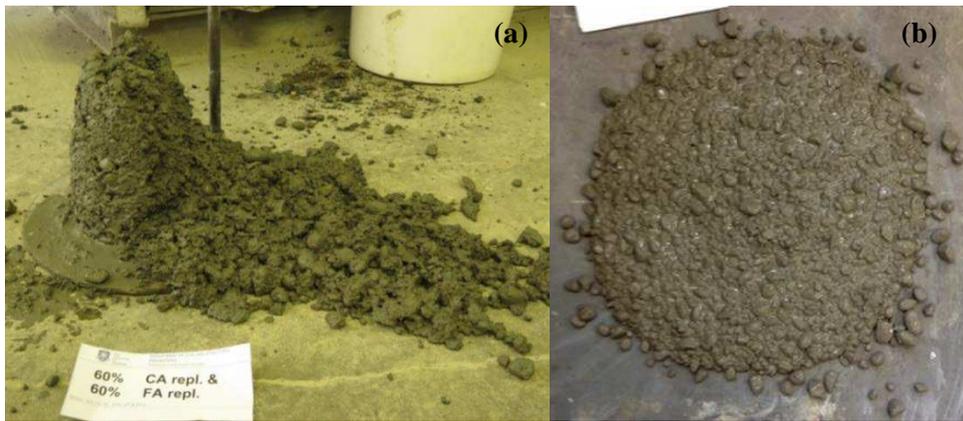
Figure 7: (a) Shear slump failure of 60C60F mix and (b) flowability test of mix C.3 with SF pre-treatment

The above results highlight the difficulty of achieving a highly flowable mix without compromising mix cohesion. To limit segregation and bleeding, sufficient water was added to hydrate the cement and superplasticisers were used to aid mix flowability and facilitate casting. The use of SF and PFA also limited segregation and bleeding and improved mix cohesion in mix D (Table 6). The effect of water/admixture content and binder materials on RuC performance is discussed in sections 3.3 and 3.4, respectively.

The optimisation of the mix proportions led to satisfactory fresh properties for RuC mixes at high rubber contents. Based on the results of this study, it is suggested to limit the w/b ratio to 0.35 and use SF and PFA to replace 20% of the cement mass (10% each).

3.2   Effect of rubber content

Figure 8 compares the average 7-day compressive strength of the original mix O and optimised mix D (normalised to the strength of corresponding mixes with no rubber) as a function of rubber replacing total aggregate volume. Figure 8 indicates that the strength of the RuC mixes reduced for all rubber contents up to a maximum of 92% for mix O (100% FR or 45% of the total aggregates). This is in line with previous studies that report a 90% reduction in compressive strength as a result of full replacement of sand with rubber [25].
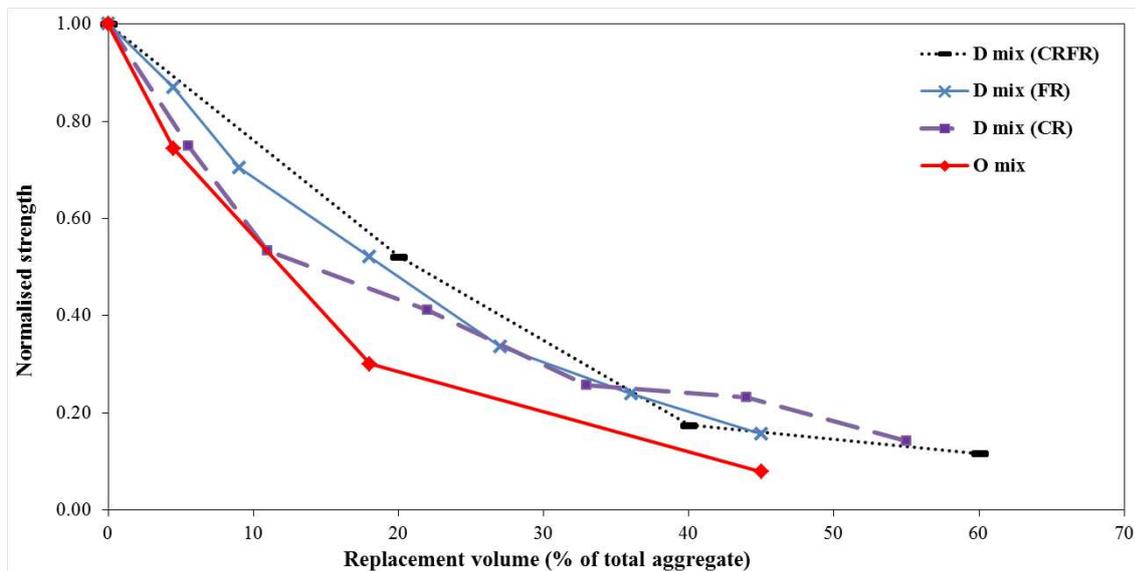
Figure 8: Variation in the normalised strength of O mix and D mix with FR replacement, CR and both

The plain mix D with no rubber had SF and PFA and lower w/b and, consequently, it had higher compressive strength than the plain mix O (see Table 6). Nonetheless, Figure 8 shows that the reduction in compressive strength due to increasing rubber content was less severe in the optimised mix D. For instance, at 40% FR content (equivalent to 18% total aggregate replacement), the strength of mix O reduced by 70%, whereas the drop was 49% in mix D. Similar results were observed in mix D with CR replacement, as well as in mixes with combined replacement of CR and FR.

The reduction in strength shown in Figure 8 is consistent with the drop in slump and flow discussed in section 3.1. Such properties are affected by the higher air content and lower workability of RuC, which can be attributed to rubber hydrophobicity, texture and shape. The reduction in compressive strength can also be due to a) lower content of strong mineral aggregates (as rubber replacement is increased), and b) rubber low stiffness and high Poisson's ratio, which tend to induce lateral tensile stresses in the concrete surrounding the rubber particles. Moreover, the reduction in strength of mixes O.3 and D.3 (at similar 40% FR) was 70% and 49%, respectively (Figure 8). This suggests that strength reduction in RuC does not only depend on rubber content but also on other mix parameters and proportions.

Figure 9 shows the reduction in density as a function of rubber content. In this figure, the density (obtained from standard cylinders) is normalised with reference to the density of concrete mixes with no rubber. To allow direct comparisons, the rubber content is expressed as a percentage of the total mix

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aggregate content. The results indicate that regardless of the mix constituents, the concrete density reduced with increasing rubber volume. This reduction is in line with the reduced compressive strength of RuC mixtures and can be mainly attributed to the lower specific gravity (SG) of the rubber particles, but also to an increase in air content. The SG of mix O was 2.54, 2.41, 2.33 and 1.92, respectively at 0%, 10%, 40% and 100% FR replacement. The SG results from mix D (Table 7) show that the plain mix D had a lower density of 2.48 (compared to mix O) due to the use of SF and PFA. The SG reduced by 24% as the sand was fully replaced with FR (100% FR replacement) in mix O. At 100% FR and 100% CR replacement, the SG of mixes D was reduced to 2.13 (14% reduction) and 1.98 (20% reduction), respectively, compared to mixes without rubber. The data in Figure 9 and Table 7 indicate that, compared to mix O, the density of mix D (with optimised proportions) reduced more gradually with increasing rubber content. This suggests a more moderate increase in air content relative to the rubber content in the latter mix. As a result, the mix optimisation was proven effective at minimising the amount of air introduced in the mix.
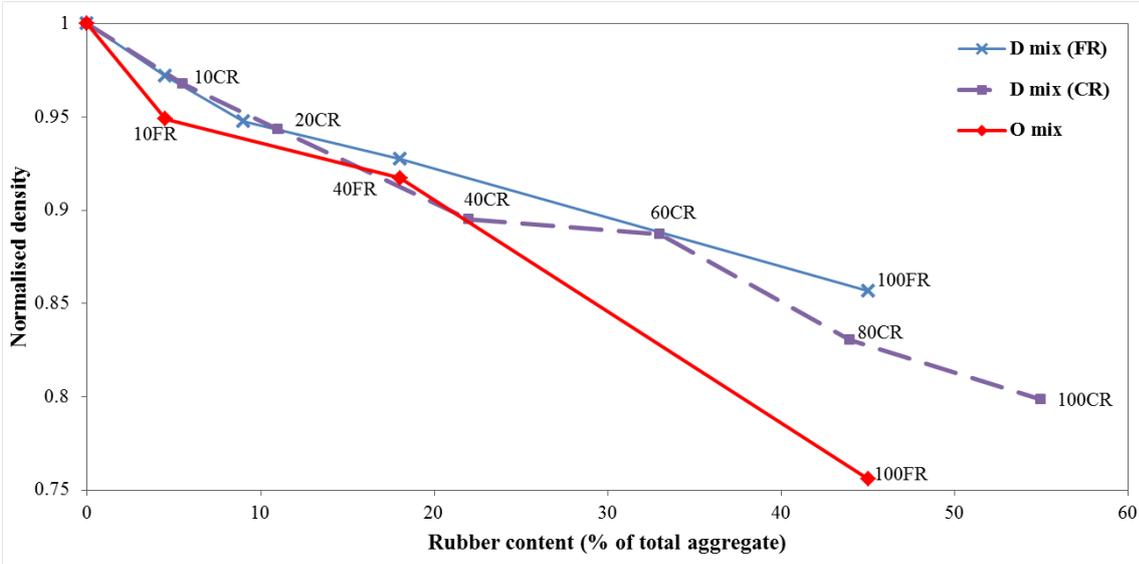
Figure 9: Variation of the density (normalised to the density of the corresponding plain mix) with rubber content for the original (O) and optimised (D) mixes.

To assess the effect of high rubber contents on the development of concrete compressive strength over time, cube compressive strengths at 3, 7, 14, 28 and 56 days were obtained for the optimised RuC mix with highest rubber content (60CR60FR). At least three cubes were tested per age and the maximum observed standard deviation was 1.6%. The results in Figure 10 indicate that the model proposed by

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Eurocode 2 (EC2) [77] estimates with reasonable accuracy the development of cube compressive strength of 60CR60FR RuC over time. However, further experimental results are necessary to fully confirm this conclusion.
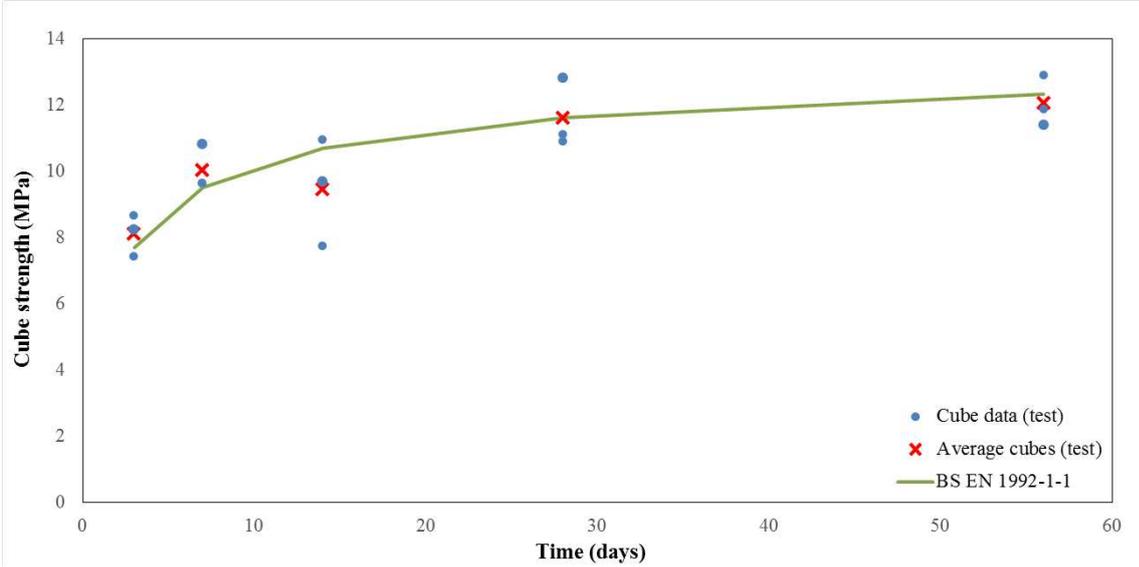

Figure 10: Development of cube strength at 3, 7, 14, 28 and 56 days (mix 60CR60FR)

3.3 Effect of water and admixture contents

The data in Table 6 show that at a rubber content of 40% FR, the fresh flow of mix O.3 (w/b=0.423) reduced from 685 mm to 520 mm, 420 mm and 'not flowable' for lower w/b contents (0.38 for mix A.1, 0.35 for A.2 and 0.32 for A.3, respectively). Figure 11 shows the effect of w/b on the fresh flow and compressive strength of RuC. Compared to mix O.3, the reduction in w/b also led to an increase in the 7-day compressive strength of mixes A.1, A.2 and A.3 by 55%, 59% and 126%, respectively. The reduction in w/b also resulted in significant improvements in mix cohesion and homogeneity. However, the use of w/b=0.30 (mix A.4) led to a very dry, unworkable, segregated and non-cohesive mix, and therefore no cylinders/cubes were cast. The extremely low flowability and the presence of segregation in mix A.3 (40% FR) indicated that such mix would behave inadequately at higher rubber contents. Therefore, a w/b of 0.35 was selected for Part 2 of the experimental programme.
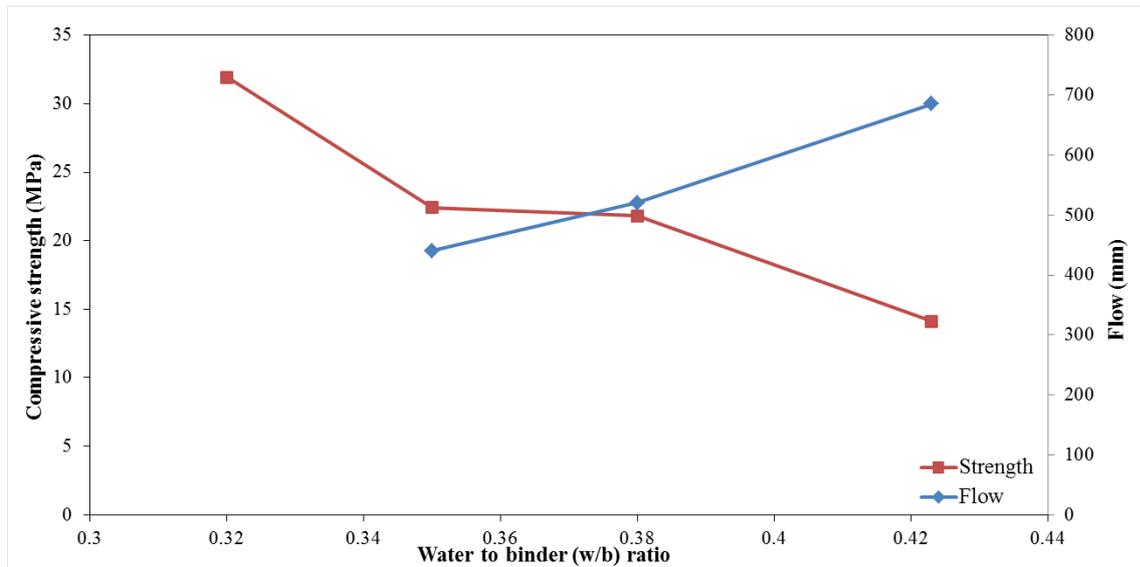
Figure 11: Variation of concrete flow and compressive strength with w/b at 40% FR content.

The mix water content also influences rubber distribution within the tested concrete cylinders. Figure 12a-b show the distribution of rubber in specimens of mixes O.3 and A.2 with 40% FR (w/b=0.423 and 0.35. respectively). It is shown that the rubber accumulated at the top of the cylinder cast in the original mix (O.3), whereas mix A.2 had a homogeneous distribution of rubber over the full cylinder height.

Figure 12: Rubber distribution in (a) Mix O.3 and (b) Mix A.2 with 40% fine rubber content.

The reduction of superplasticiser content by 40% (in mix B.2) reduced the flow (by up to 16%) but, more importantly, led to a reduction in mix segregation (segregation level reduced from 2 to 0) compared to mix O.3 with identical rubber content (see Table 6). However, due to the relatively high amount of lubricating water in the mixes and the rubber hydrophobicity, a thin gleam was observed at the surface of

all B mixes, thus indicating bleeding. Based on the above observations, it is proposed to limit the w/b to the minimum required for cement hydration and use water reducing admixtures to achieve the necessary flowability. To maintain mix stability and homogeneity, flowability should be kept to a minimum. The reduced w/b is also recommended to prevent significant strength reduction.

3.4   Effect of SF and PFA replacement

The partial replacement of cement with SF and PFA (10% each) improved significantly the concrete mix performance (Table 6). For instance, mix D (at w/b=0.35), had better fresh properties and compressive strength than the original mixes O and A (w/b=0.35) at all rubber contents. The improved mix performance due to binder material alone is evident by comparing mixes A.2 and D.3 at 40% FR, as the strength and flowability of the latter were 42% and 20% higher, respectively. The effectiveness of SF and PFA at enhancing RuC properties can be attributed to its filling effect (improved packing), as well as to its pozzolanic reaction with the cement hydration products. Previous research [78] indicates that fine SF particles also reduce bleeding, thus enhancing packing in the ITZ (which in turn increases the RuC strength). The effect of the fine fillers on the packing of the cementitious materials and RuC microstructure are discussed in sections 3.7 and 3.8, respectively.

3.5   Effect of rubber particle size

The results in Figure 5 (and Table 7) show that the fresh flowability of RuC is more affected by fine aggregate replacement than by coarse aggregate replacement, particularly at rubber contents above 20% of the total aggregates. At 100% FR replacement (45% of total aggregate volume), the mix was extremely dry and unworkable, whereas a flow of 380mm was achieved for the mix with 100% CR replacement (55% of total aggregate volume). This can be attributed to the filling effect of sand and its role in aiding mix flowability, as well as the excessive friction caused by the fine rubber particles, which have a rough surface and a larger surface area compared to coarse rubber particles with the same volume.

Figure 8 reveals a marginal difference in the compressive strength of the optimised mix D if coarse or fine rubber is used as aggregate replacement. Such difference varies with rubber content and seems to peak at around 10% total aggregate replacement, where CR led to compressive strength nearly 20% lower than the strength achieved using FR (strengths of 34 MPa vs. 42 MPa, respectively). This can be due to the load-bearing role of the larger coarse aggregates, which transfer loads directly within the specimens.

However, for rubber contents ranging between 20% and 40% of the total aggregate, a similar concrete compressive strength was achieved regardless of type of rubber replacement. This is not in line with previous studies reporting much lower compressive strength when replacing coarse aggregates as opposed to fine aggregates [24]. This deviation can be attributed to comparisons often being made between different amounts of rubber. Unfortunately, numerous previous studies express the rubber content as a fraction of the type of mineral aggregate replaced (coarse or fine), and therefore the actual amount of rubber in the mix could vary depending on the coarse to fine aggregate ratio, leading to comparisons among concretes with different overall rubber contents [61].

The standard deviation (SD) in compressive strength for mixes with FR replacement was 7.4% (mix D.4 with 100% FR, see Table 7). However, higher SD was observed for CR replacement, with a maximum of 27.1% at 60% CR. The higher variability associated with CR replacement may be attributed to the possible reduction in the amount of force that is transferred through direct contact in the stiffer mineral coarse aggregate, as well as to variations in stiffness across the cylinder. For instance, due to the larger size and lower quantity of CR particles (at an identical replacement level as FR), the particle distribution throughout the cylinder may vary significantly among test cylinders, leading to the observed higher variability.

The effects of combined replacement of coarse and fine aggregates (20CR20FR, 40CR40FR and 60CR60FR) on RuC fresh properties and compressive strength are shown in Table 7 and Figure 8, respectively. The compressive strength of mix 20CR20FR (20% total aggregate replacement - 32MPa) was higher than that achieved with 40FR or 40CR (18% and 22% total aggregate replacement, respectively) with strengths of 31.2 and 25.3 MPa, respectively, as shown in Figure 8. At 60% total aggregate replacement (60CR60FR), the strength reduction was similar to that observed in 100% FR or 100% CR replacement (45% and 55% of the total aggregates, respectively) despite having a higher overall rubber content. In terms of fresh properties, all mixes with combined CR and FR replacement had good workability with good cohesion and limited segregation. In particular, mix 60CR60FR had much better cohesion and homogeneity compared to mixes with total fine (100FR) or coarse aggregate (100CR) replacement.

The above observations indicate that, at low rubber contents, coarse aggregate replacement was more detrimental to the RuC compressive strength than fine aggregate replacement. This effect was not observed at higher rubber contents, where rubber properties seemed to control RuC behaviour regardless of the type of aggregate replaced. Whilst the compressive strength seems to be slightly more influenced by CR properties, the FR was slightly more detrimental to the concrete fresh properties, particularly at the higher rubber contents. The combined CR and FR replacement proved to be a suitable solution to maximise the rubber contents in RuC mixes without completely eliminating coarse or fine mineral aggregates, as well as to maintain satisfactory fresh properties and compressive strength. Mix 60CR60FR had a flow of 410mm, adequate cohesion and homogeneity and a compressive strength of around 7MPa. Such properties at high rubber contents were only achieved with the optimised mix and combining both CR and FR replacement. The 60CR60FR mix is instrumental for future studies by the authors in which high-rubber-content RuC cylinders are confined to achieve an environmentally friendly, high-ductility, high-deformability concrete.

3.6   Rubber pre-treatments

The results in Table 6 show that pre-washing rubber with water did not enhance the mix performance significantly. A minor reduction in flowability (3-5%) was observed in mixes C.1 and C.2 (with pre-washed rubber), respectively, compared to mixes A.1 and A.2 with the same amount of water directly added to the mix. Nonetheless, the strength and density of mixes C.1 and C.2 were comparable to mixes A.1 and A.2, respectively. These results confirm that pre-washing the rubber is not an effective solution to improve the rubber-cement paste bonding. Likewise, pre-coating the rubber with SF (mix C.3) rather than simply adding SF to the mix (mix D.3) did not improve mix performance. In fact, compared to mix C.3, mix D.3 had slightly higher slump, flow and strength (12%, 7% and 13%, respectively). Overall, the variation in performance and density of the mixes with pre-treated particles compared to mixes with as-received particles falls within the standard variation anticipated in normal concrete. Pre-treatments are also often costly, time-consuming and aggressive to the concrete and rubber and should be only used if significant benefits are foreseen.

3.7 Influence of water and binder in packing

The effect of optimising the water content and binder type on the packing of the concrete cementitious materials was examined using the wet packing method developed by Wong and Kwan [79]. Accordingly, the samples were prepared as follows: 1) Dry mix all binder materials for 2 min; 2) Place all the mixing water (based on selected w/b) in a bowl; 3) Add half the binder and admixtures to the bowl and mix at low speed for 3 min; 4) Divide the remaining binder and admixtures into four parts and add the portions (one after the other) to the bowl and mix for 3 minutes each; 5) Fill a (50mm dia × 100mm) cylindrical mould with the mixture to excess. The cylinder is either vibrated or left unconsolidated and the excess paste is removed; 6) Record the weight of the paste in the mould.

The binder mixes follow the proportions examined during the mix optimisation (see Table 3 and Table 4). Four representative mixes were selected to examine the influence of w/b and binder material on the packing of the cementitious mixes: a) O – with w/b=0.423; b) A - 0.38 – with a w/b=0.38; c) A - 0.35 – with w/b=0.35 and d) D – with w/b=0.35 and SF and PFA each replacing 20% of the cement mass (10% each). The void contents (ε) and solid concentration (ϕ) for mixes with/without vibration are shown in Table 8.

Table 8: Voids ratio, air ratio and solid concentration of cementitious mixtures with/without vibration

Mix I.D.	Unconsolidated		Vibrated	
	Voids content (ε)[a]	Solid Concentration (ϕ)[b]	Voids content (ε)	Solid Concentration (ϕ)
O	0.573	0.427	0.570	0.430
A–0.38	0.557	0.443	0.555	0.445
A–0.35	0.531	0.469	0.528	0.472
D	0.514	0.486	0.511	0.489

[a]Defined as the ratio of the volume of voids (voids content) to the bulk volume of the granular materials; [b]Ratio of the solid volume of the bulk granular material to its bulk volume [79]

Table 8 indicates that the solid concentration was slightly higher for all vibrated mixes compared to unconsolidated mixes. This increase in solid concentration is attributed to a decrease in voids and air content upon vibration (Table 8) and to the 'settling' of cementitious particles. As the w/b reduced from 0.423 (O) to 0.38 (A-0.38) and 0.35 (A-0.35), the solid concentration increased by 4% and 10%, respectively, indicating a better packing in the mix (vibrated and unconsolidated). The highest solid concentration (14% increase compared to O mix) was observed when SF and PFA were used to replace

portions of the cement (see mix D, Table 8). Moreover, compared to mix A-0.35 (with identical w/b), the packing density of mix D increased by 4% for both consolidation types. This can be mainly attributed to the filling effect of SF and PFA [79].

The data in Table 8 show that the increase in solid concentration coincides with a reduction in voids content, thus indicating a reduction in the mix water requirement (to fill the voids) and, in turn, a higher compressive strength in mixes with higher packing densities. At a fixed water content, the excess water (not filling the voids) can increase the mix workability [79] up to the point where segregation and bleeding occur. This is in line with the flow table test results summarised in Table 6. For example, the flow for mix D.3 (with SF and PFA binder) is 20% higher than that observed in mix A.2, despite having identical w/b (0.35) and rubber content (40% FR).

3.8 Microstructural observations

Figure 13a-d show 30x magnification images of RuC samples (at an age of 14 days) extracted from the following mixes: a) Mix D with alternative binder materials and w/b of 0.35, b) Mix O with w/b of 0.423, c) Mix A-0.35 with w/b of 0.35, and d) Mix A-0.38 with w/b of 0.38. The selected mixes had relatively small rubber content (combined 20% CR and FR replacement) so as to enable the manufacturing of samples including representative volumes of both mineral and rubber aggregates in all samples. Average 7-day cube strength (three cubes per mix) for mixes D, A-0.35, A-0.38 and O was 40 MPa, 39.2 MPa, 37.9 MPa and 37.5 MPa, respectively, with a maximum standard deviation of 5%. The darker features in Figure 13a-d represent voids or rubber, the mineral aggregates show an intermediate grey colour, whereas the hydrated cement phase is represented by a continuous light grey, as pinpointed in Figure 13c. Bright spots scattered across the images are either tyre steel fibres (Figure 13c) or unhydrated/partially hydrated cement particles as observed at higher magnifications (500x) for mixes O and D (see Figure 13e-f).
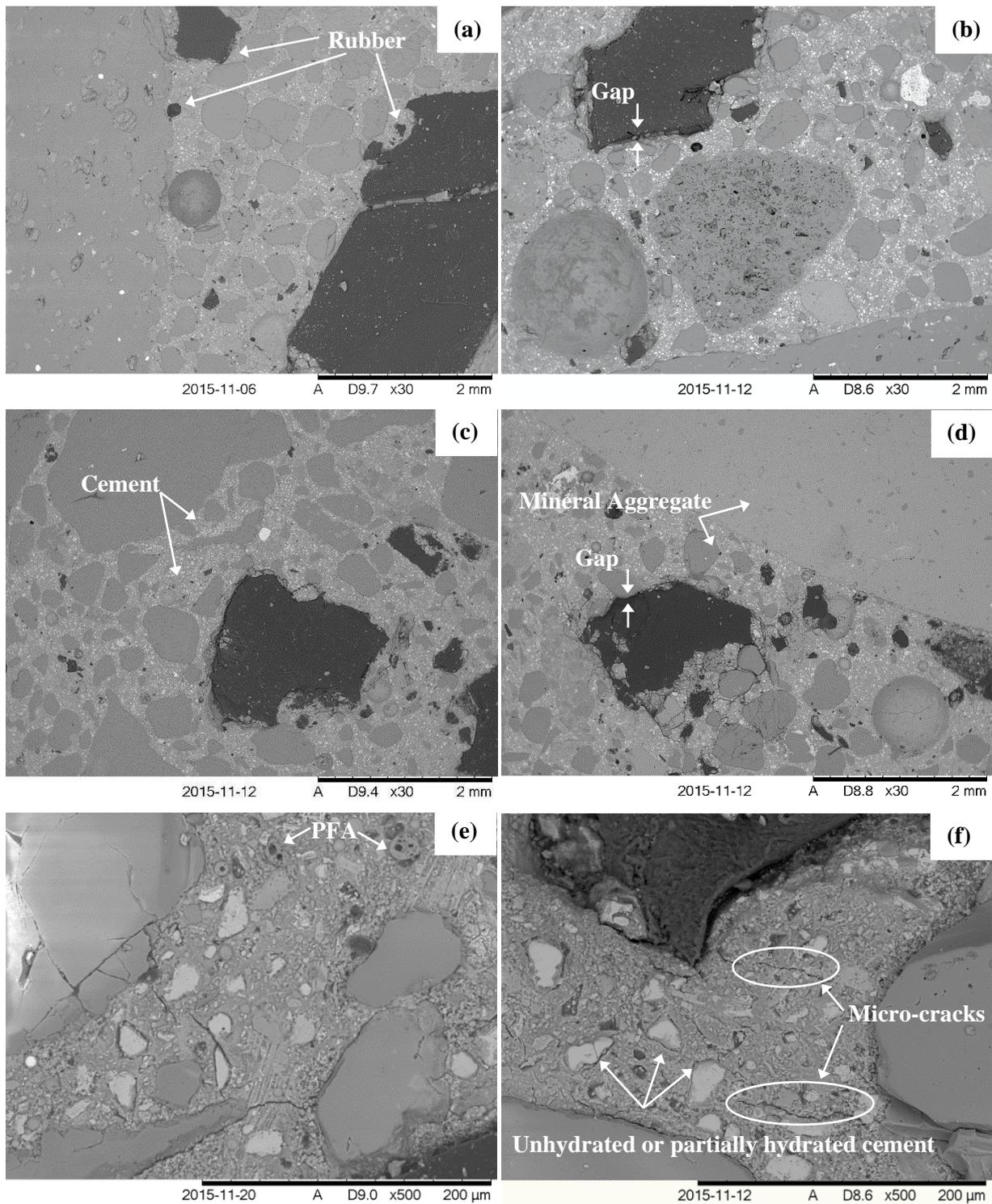
Figure 13: SEM images at 30x magnification: Mix D (a), mix O (b), mix A-0.35 (c) and mix A-0.38 (d) and at 500x magnification: Mix D (e) and mix O (f)

Whilst the rubber and mineral aggregates were randomly distributed over the concrete samples, large gaps

(e.g. see Figure 13b) were evident between the rubber particles and the surrounding cement paste (notably

in Mixes O and A-0.38). This effect could either be due to a) lack of bonding and limited cement

hydration in the rubber-cement paste ITZ, b) rubber detachment during specimen preparation, or c) a combination of the two. A highly porous ITZ layer surrounded the rubber, exhibiting a slightly darker halo around the rubber particle (due to its lower density) (Figure 13f). Overall, the gaps between the rubber and the cement phase are smaller in samples extracted from mix D (Figure 13a) than in those extracted from mixes O and A-0.38 (Figure 13 b and d, respectively). This is due to the higher water content in mixes O and A-0.38, which, along with the rubber hydrophobicity, creates a film of air around the rubber, leading to reduced cement hydration, weaker bond and weaker ITZ in the rubber vicinity. Moreover, mixes O and A were weaker than mix D, thus promoting rubber detachment. The improved integration of the rubber particles in mix D emphasises the beneficial effect of mix optimisation with lower water content, as well as the filling effect of SF and PFA.

The images also reveal cracks across the Interfacial Transition Zone (ITZ) between the mineral aggregates and the cement paste and in the cement paste (Figure 13f). This cracking can be attributed to shrinkage of the cement paste and differential restrain provided by the aggregates. The weak ITZ phase between the cement and the aggregates is much thinner in mineral aggregates than in rubber particles (Figure 13f). This is in line with conclusions of previous research that found a 'double porosity' in the ITZ surrounding the rubber aggregates [38].

PFA particles (spherical shape) were observed in SEM images of samples from mix D (Figure 13e). This was validated using chemical analysis of mix D in this locality (at 1000x magnification, Figure 14a), which shows the combined presence of Silicon (Si), Aluminium (Al) and Oxygen (O), the main constituent elements of PFA. Chemical analysis of mixes D and O (Figure 14a-b) shows that no Zinc (Zn) was present on the rubber surface (or in the entire concrete sample). Zinc is often suspected to be a cause of rubber hydrophobicity [41].
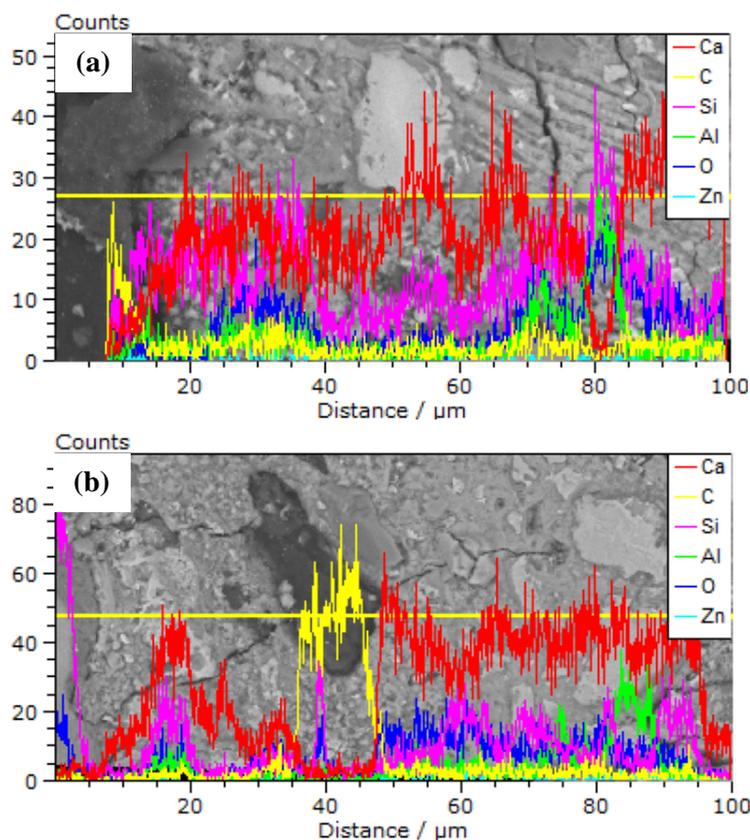
Figure 14: BSE elemental analysis for samples from mix D (a) and O (b) at 1000x magnification

The initial results from SEM observations indicate that the use of SF and PFA improved the bonding between the rubber and the cement phase in the optimised mixes and reduced the thickness of the gap or the weak ITZ. Nevertheless, the influence of sample preparation must be evaluated and more images are needed from different parts of the sample to account for variability.

4   Conclusions

Based on the results presented in this article, the following conclusions are drawn:

Effects of rubber:

1. Higher rubber contents reduce concrete workability, hardened concrete density and compressive strength.

2. The concrete strength appears to be more influenced by the overall rubber volume rather than the type of rubber replacement (coarse or fine aggregate replacement). Nevertheless, RuC fresh

properties were slightly inferior at high levels of fine aggregate replacement. The combined replacement of fine and coarse aggregates helps achieve high rubber contents with minimal influence on strength and workability.

Mix optimisation:

1. Mix optimisation minimises the adverse effects of rubber on the concrete fresh and hardened mechanical properties. For instance, the replacement of 40% fine aggregates with rubber reduced the strength of the original mix (O) by 70%, whereas such reduction was only 49% in the optimised mix (D).

2. Optimised RuC mixes (D) enabled the use of high rubber contents (up to 60% total aggregate volume replacement) whilst maintaining an acceptable workability and a compressive strength of 7 MPa at 7 days.

3. Using SF and PFA to replace 20% of the cement mass increased the concrete flowability by 20% and the strength by 42%. SF and PFA had a filling effect (thus improving packing) and a pozzolanic reaction with the cement hydration products.

4. Pre-washing rubber with water or pre-coating with SF did not improve RuC performance.

Microstructure:

1. SEM analysis revealed a gap between rubber and the rubber-cement paste ITZ, particularly for larger rubber particles. The use of SF and PFA reduced this gap.

Acknowledgments

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