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Influence of Mixing Procedures, Rubber Treatment, and Fibre Additives on Rubcrete Performance

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Abstract: This research extensively investigates how to enhance the mechanical performance of Rubcrete, aiming to move this type of concrete from the laboratory research level to a more practical use by the concrete industry. The effects of many different mixing procedures, chemical pre-treatments on the rubber particles, and the use of fibre additives, have been investigated for their impact upon Rubcrete workability, compressive strength, tensile strength, and flexural strength. The mixing procedure variables included mixing time and mixing order. The rubber pre-treatments utilized chemicals such as Sodium Hydroxide (NaOH), Hydrogen Peroxide (H_2O_2), Sulphuric acid (H_2SO_4), Calcium Chloride ($CaCl_2$), Potassium Permanganate ($KMnO_4$), Sodium Bisulphite ($NaHsO_3$), and Silane Coupling Agent. Soaking rubber particles in tap water, or running them through water before mixing, were also tried as a pre-treatment of rubber particles. In addition, the effects of fibre additives such as steel fibres, polypropylene fibres, and rubber fibres, were assessed. X-ray photoelectron spectroscopy (XPS) analysis was utilised to examine some of the pre-treated rubber particles. The results showed that doubling the net mixing time of all mix constituents together enhanced the Rubcrete slump by an average of 22%, and the compressive strength by up to 8%. Mixing rubber with dry cement before adding to the mix increased the compressive strength by up to 3%. Pre-treatment using water was more effective than other chemicals in enhancing the Rubcrete workability. Regardless of the treatment material type, the longer the time of the treatment, the more cleaning of rubber occurred. Significant Rubcrete flexural strength increase occurred when using 1.5% fibre content of both steel fibre and polypropylene fibre.

Keywords: rubberised concrete; rubcrete; rubber pre-treatment; rubcrete mixing procedures; fibre additives

1. Introduction

Every year, countless millions of new tyres are produced and at the same time, many of them reach the end of their practical life. For example the European Union produced 355 million tyres in 2013, which was about 24% of the estimated global production of approximately 1.5 billion tyres [1,2]. Worldwide, many approaches have been used to manage the problem of what to do with end of life tyres (ELT).

The organisation tasked with increasing the recycling of tyres in Australia is Tyre Stewardship Australia (TSA), which was commissioned by the national government, and is funded through a levy of 25c per Equivalent Passenger Unit added to the price of every new tyre sold by participating companies (which includes all of the major players in the industry). TSA reports that at present only ten per cent of tyres are recycled in Australia, with 30% exported for re-use, and the rest either stockpiled, placed in landfills or illegally dumped [3,4]. This is in stark contrast to the situation in Europe, where in 2010 only 4% of ELT ended up in landfills, with 96% being recovered (38% recovery of energy, 40% recycling of materials, 8% reconstruction, and 10% reuse/export) [5]. While there are many different ways of recycling tyres, in the structural materials area, a significant amount of research has recently focused on using crumb rubber from recycled tyres as partial replacement of aggregates (either sand or rock, depending on the rubber particle size). The research is also driven by the increasing scarcity and cost of obtaining or transporting natural materials, such as river sands that are used in concrete [6]. The resultant product has been termed as Rubcrete. Recycling of used rubber in concrete conserves valuable natural resources, and reduces the amount of rubber entering the landfill [7,8].

Research to date on Rubcrete has provided contradictory findings. There are some properties that are enhanced in Rubcrete compared with conventional concrete, including damping ratio, ductility, energy dissipation, impact resistance and toughness [9–12]. Conversely, the key mechanical properties of compressive strength, modulus of elasticity, and tensile strength are reduced, and this has raised concerns or limitations on its potential use in construction [13–15]. Several approaches have been examined to reduce or eliminate the rubber deficiencies in concrete, such as pre-treating the rubber particles before using them in concrete, and/or adding some external additives as general enhancers for concrete mechanical properties. However, the experimental findings regarding the effectiveness of these approaches have been quite inconsistent and in some cases conflicting in the research literature to date. Balaha et al. [16] showed that the Rubcrete properties improved with a cement content increase up to 500 kg/m^3. They also reported that using 15% silica fume (SF) and Sodium Hydroxide (NaOH) solution pre-treatment of rubber particles increased concrete slump by 77% and 7%, respectively, increased compressive strength by 18% and 15%, respectively, and increased tensile strength by 9% and 6%, respectively. Youssf et al. [17] found that the losses in Rubcrete compressive strength with higher cement content were less than when using lower cement content. In addition, when using pre-treated rubber, while the concrete slump and tensile strength decreased by 25% and 13%, the compressive strength and modulus of elasticity increased by 15% and 12% respectively, compared to non-treated rubber. No effect was observed in their results when using SF, except a slight increase in the compressive strength at rubber content of 20% by sand volume. Other researchers have also reported success in improving the concrete compressive strength of Rubcrete through a range of pre-treatment and additive methods including: Eldin and Senouci [18]; Pelisser et al. [19]; Güneyisi et al. [20]; Mohammadi et al. [21]; Youssf et al. [22], Su et al. [23]; Hamza and Ghedan [24] and Azevedo et al. [25].

There have also been a number of studies that have reported negligible improvement or even a lowering of compressive strength despite any pre-treatment or use of additives. For example, Raffoul et al. [26] tried two different rubber pre-treatments. The first one was pre-washing with water and then air drying, and the second one was pre-coating with SF paste for 20 min before mixing with other concrete constituents. Their results showed that not only did their pre-treatment methods marginally affect the Rubcrete strength, but they also resulted in a reduced flowability in concrete. Other researchers who reported negligible improvement in compressive strength, even though they used pre-treatments that were basically the same as those reported in the previous paragraphs, included: Deshpande et al. [27]; Tian et al. [28]; Li et al. [29]; Turatsinze et al. [30]; and Albano et al. [31].

Tian et al. [28] observed that rubber pre-treatment by inorganic salt Calcium Chloride ($CaCl_2$) improved the mechanical properties of Rubcrete; however organic, acidic, and alkaline solutions did not effectively enhance Rubcrete properties. Huang et al. [32] showed that rubber pre-treatment by

silane coupling agent followed by cement paste coating could increase the compressive strength by up to 110%. Dong et al. [33] used a similar method, but their results showed only a 10–20% strength enhancement in concrete incorporating coated rubber, compared to that with uncoated rubber. Abdulla and Ahmed [34] showed that rubber pre-treatment by Sulphuric acid (H_2SO_4) increased the rubberised mortar compressive strength by 2 times, but it negatively impacted on other properties of the cement mortar. Xiong et al. [35] observed a noticeable improvement in the microstructures of cement hydrates at the rubber/cement interfacial transition zone when using silane coupling agent solution (0.5–1.0% concentration) for pre-treatment. He et al. [36] showed that the oxidation and sulphonation of rubber particles significantly improved the compressive strength by 48.7%. Akinyele et al. [37] noted that rubber in concrete affects not only the mechanical, but also the chemical properties. They showed that increasing rubber in concrete decreased Ferrous iron, Oxygen, Calcium, Aluminium, and Silicon elements; however, it increased Carbon and Sulphur elements which act as impurities during the hydration process.

Limited research was carried out on the effect of fibre additives on Rubcrete performance. Carroll and Helminger [38] proved that the addition of fibre reinforcement increased the Rubcrete compressive strength, split tensile strength, and modulus of elasticity by 15%, 34%, and 9%, respectively. Hesami et al. [39] found that polypropylene fibres increased the compressive, tensile, modulus of elasticity, and flexural strength by 21%, 26%, 11%, and 34%, respectively. However, the presence of polypropylene fibres in concrete decreased its water absorption.

Another key point to note among the multitude of pre-treatment methods that have been researched to date, is that if Rubcrete is to become a practical and economical option within the pre-mix concrete supply industry, then any pre-treatment method must be able to be incorporated into the concrete production process in a practical and cost-effective manner, and many of the methods that have been researched seem unlikely to be able to achieve this. Mixing procedures and concrete workability are also areas that need investigation. In summary, further research is required to determine a practical and economic process for producing commercially viable Rubcrete that has suitable mechanical properties, and the necessary workability for use in practice. The research reported below examined both the mechanical and workability properties of a range of Rubcrete mixes incorporating 0%, 15%, 20%, and 30% of crumbed rubber as a partial volume replacement of sand. The effects of different Rubcrete mixing procedures, chemical pre-treatment of rubber particles, and fibre additives on Rubcrete slump, compressive strength, tensile strength, and flexural strength, were examined. X-ray photoelectron spectroscopy (XPS) measurement was carried out for selected pre-treated rubber particles. The results provide valuable additional data to further the development of Rubcrete for possible use in concrete structures.

2. Experimental Programme

2.1. Concrete Materials and Variables

A summary of the components of all concrete mixes in this study is provided in Table 1. The binder material used was General Purpose (GP) cement with a specific gravity of 3.15, in accordance with Australian Standard (AS) AS 3972 [40]. The coarse aggregate was dolomite stone (commonly used in Australia), with nominal maximum sizes of 10 mm, 14 mm, and 20 mm, while the fine aggregate was river sand with a maximum size of 5 mm. Crumb rubber, which was used as partial replacement of the river sand by volume, comprised two particle sizes of 2.36 mm and 4.75 mm. Figure 1 provides the sieve analysis for all of the aggregates used. The specific gravity, fineness modulus, and unit weight were 2.71, 7.89, and 1590 kg/m^3, respectively for dolomite; 2.61, 2.20, and 1420 kg/m^3, respectively for sand; and 0.97, 4.85, and 530 kg/m^3, respectively for rubber. Polycarboxylic ether type superplasticizer (SP), BASF masterglenium sky 8708, with a specific gravity of 1.085, was added to all concrete mixtures.

Table 1. Proportions of concrete mixes.

Mix Code	Rs (%)	Mixing Procedures*	Pre-treatment	Mix Proportions (kg/m^3)									
				Cement	Sand	Stone (mm)			Rubber	Water	SP	Fibre		
						10	14	20				SF	PF	RF
M1	0	P1	–	400	817	–	493	493	–	200	2.37	–	–	–
M2	15	P1	–	400	695	–	493	493	44.9	200	2.37	–	–	–
M3	30	P1	–	400	572	–	493	493	89.7	200	2.37	–	–	–
M4	15	P2	–	400	695	–	493	493	44.9	200	2.37	–	–	–
M5	30	P2	–	400	572	–	493	493	89.7	200	2.37	–	–	–
M6	15	P3	–	400	695	–	493	493	44.9	200	2.37	–	–	–
M7	30	P3	–	400	572	–	493	493	89.7	200	2.37	–	–	–
M8	15	P4	–	400	695	–	493	493	44.9	200	2.37	–	–	–
M9	30	P4	–	400	572	–	493	493	89.7	200	2.37	–	–	–
M10	15	P5	–	400	695	–	493	493	44.9	200	2.37	–	–	–
M11	15	P6	–	400	695	–	493	493	44.9	200	2.37	–	–	–
M12	15	P4	Water wash	400	695	–	493	493	44.9	200	2.37	–	–	–
M13	15	P4	Water soaking-A	400	695	–	493	493	44.9	200	2.37	–	–	–
M14	15	P4	Water soaking-O	400	695	–	493	493	44.9	200	2.37	–	–	–
M15	0	P1	–	340	826	458	–	550	–	204	1.36	–	–	–
M16	20	P1	–	340	661	458	–	550	62.0	204	1.36	–	–	–
M17	20	P1	NaOH	340	661	458	–	550	62.0	204	1.36	–	–	–
M18	20	P1	H_2O_2	340	661	458	–	550	62.0	204	1.36	–	–	–
M19	20	P1	$CaCl_2$	340	661	458	–	550	62.0	204	1.36	–	–	–
M20	20	P1	H_2SO_4	340	661	458	–	550	62.0	204	1.36	–	–	–
M21	20	P1	Silane	340	661	458	–	550	62.0	204	1.36	–	–	–
M22	20	P1	$KMnO_4$_$NaHSO_4$	340	661	458	–	550	62.0	204	1.36	–	–	–
M23	20	P1	–	338	658	456	–	547	61.7	203	1.35	39.6		
M24	20	P1	–	337	654	453	–	544	61.4	202	1.34	79.3		
M25	20	P1	–	335	651	451	–	542	61.1	201	1.33	118.9		
M26	20	P1	–	338	658	456	–	547	61.7	203	1.35	–	4.5	
M27	20	P1	–	337	654	453	–	544	61.4	202	1.34	–	9.1	
M28	20	P1	–	335	651	451	–	542	61.1	201	1.33	–	13.6	
M29	15	P1	–	396	688	–	488	488	44.4	198	2.35	–	–	9.7
M30	15	P1	–	392	681	–	483	483	43.9	196	2.32	–	–	19.4
M31	15	P1	–	388	674	–	478	478	43.5	194	2.30	–	–	29.1

Rs Per cent of sand volume replaced by rubber. SP Superplasticizer dosage. SF Steel fibre.
PF Polypropylene fibre. RF Rubber fibre.
* Mixing procedures description is show in Section 2.2

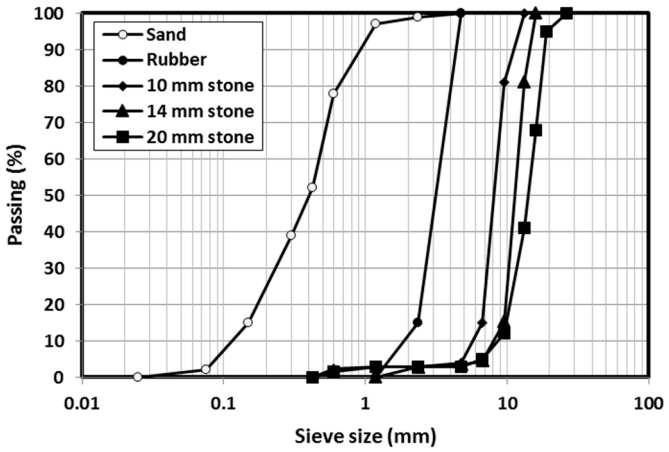
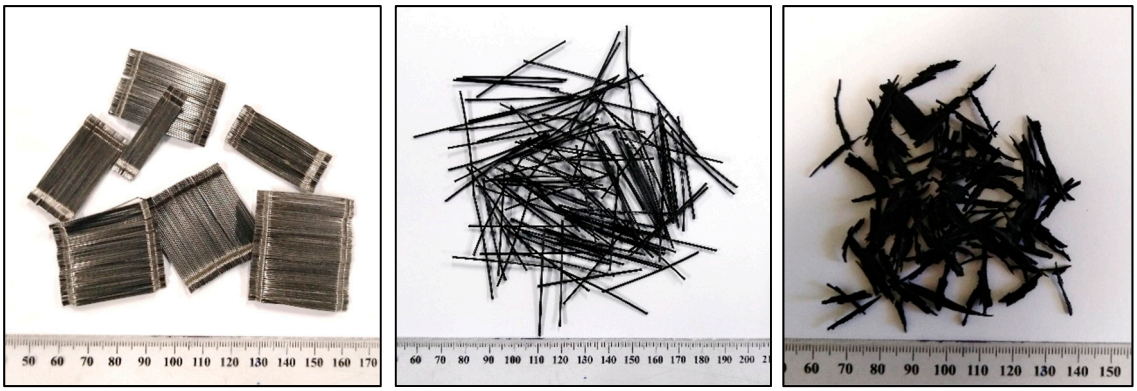


Figure 1. Sieve analysis of the aggregates used.

In order to compare the performance of fibre from waste material with commonly used fibre as concrete additives, rubber fibres (TyreCycle Rubber Buffings), steel fibres (TEXO ReoCo 65/35), and polypropylene fibres (TEXO ReoShore 45) were used in some concrete mixes, see Figure 2. Table 2 shows the properties of fibres used, as provided by the manufacturers.



Steel fibres Polypropylene fibres Rubber fibres

Figure 2. Different fibre additives used in this study.

Table 2. Proportions of fibres used.

Fibre Type	Diameter (mm)	Length (mm)	Specific Gravity	Tensile Strength (MPa)
Steel fibres	0.55	35	7.93	1300
polypropylene fibres	0.80	45	0.91	800
Rubber fibres	0.5–1.3	15–30	0.97	N/A

The variables in this study were: The mixing procedures including mixing time and mixing order; the rubber particle pre-treatment material including Sodium Hydroxide (NaOH), Hydrogen Peroxide (H_2O_2), Sulfuric acid (H_2SO_4), Calcium Chloride ($CaCl_2$), Potassium Permanganate ($KMnO_4$), Sodium Bisulphite ($NaHsO_3$), Silane Coupling Agent, water soaking and water washing; and fibre additives including fibre type (steel, polypropylene, and rubber) and fibre dosage (0.5%, 1.0%, 1.5%, 2.0%, and 3.0% by concrete volume). The effects of these variables were measured on concrete mixes containing 0%, 15%, 20%, and 30% rubber content. The rubber particle pre-treatment methods were selected according to the previous studies as options that could potentially improve the performance of Rubcrete.

2.2. Concrete mix Designs and Proposed Mixing Procedures

The concrete mixes in this study were all designed in accordance with AS 1012.2 [41]. There were two control mixes designated as M1 (50 MPa target compressive strength) and M15 (40 MPa target compressive strength). As indicated by the mix proportions in Table 1, the following properties were held constant for each mix set: Cement content (400 kg/m^3 for the 50 MPa mixes and 340 kg/m^3 for the 40 MPa mixes respectively); water to cement ratio (W/C) (0.5 for 50 MPa and 0.6 for 40 MPa); SP content as % of cement weight (0.6% for 50 MPa and 0.4% for 40 MPa); fine/coarse aggregate ratio by weight (1/1.2 for 50 MPa and 1/1.22 for 40 MPa) and the coarse aggregate ratios by weight (14 mm /20 mm was 1/1 for 50 MPa and 10 mm /20 mm coarse was 1/1.2 for 40 MPa mixes).

As rubber is still not commonly used as an aggregate in practical concrete applications, ready mix companies need to be provided with the mixing procedures of this relatively new type of concrete material. Previous research published in Rubcrete has not mentioned clearly the mixing steps and their effects on Rubcrete properties. Therefore, six mixing procedures were considered in this research to investigate the best way to mix rubber in concrete, through experimenting with the order of adding rubber to the mix, or the time required for mixing rubber with other concrete constituents. The rubber particles were mixed with dry cement or mixing water, before being added to the mix in some procedures aimed at enhancing the hydrophilicity of rubber. The mixing time of all Rubcrete constituents including rubber, was doubled in other procedures for better dispersion and conductivity of rubber particles within the concrete matrix. The Rubcrete mixing followed three mixing stages in addition to a rest stage in-between, as summarized in Table 3 and in words below:

- Procedure 1 (P1): Mix dry stone, sand, and rubber for 1 min; add 1/2 water and mix for 1 min; rest for 2 min; add cement, 1/2 water, and SP, and then mix for 2 min.
- Procedure 2 (P2): Mix dry stone, sand, rubber, and 1/2 water for 0.5 min; add cement, water, and SP and mix for 2 min; rest for 2 min; and then mix for 2 min.
- Procedure 3 (P3): Mix dry stone, sand, rubber, and cement for 1 min; add water and SP and then mix for 3 min.
- Procedure 4 (P4): Mix dry stone and sand for 1 min; add 1/2 water and mix for 1 min; rest for 2 min; add rubber, cement, 1/2 water, and SP, and then mix for 2 min. In P4, the rubber was mixed first with dry cement in a different container before being added to the concrete mix.
- Procedure 5 (P5): Mix stone, sand, rubber, cement, water, and SP for 2 min; rest for 2 min; and then mix for 2 min.
- Procedure 6 (P6): Mix dry stone and sand for 1 min; add 1/2 water and mix for 1 min; rest for 2 min; add rubber, cement, 1/2 water, and SP, and then mix for 2 min. In P6, the rubber was mixed with water and SP in a different container before being added to the concrete mix.

Table 3. Summary of the mixing procedures.

Mixing Procedure	Mixing Stage 1		Mixing Stage 2		Rest Time (min)	Mixing Stage 3		Overall Mixing Time (min)	Net Mixing Time of all Constituents, T_n (min)
	Constituents	Time (min)	Other Constituents	Time (min)		Other Constituents	Time (min)		
P1	Stone, sand, rubber	1.0	1/2 water	1.0	2.0	Cement, 1/2 water, SP	2.0	6.0	2.0
P2	Stone, sand, rubber, 1/2 water	0.5	Cement, water, SP	2.0	2.0	–	2.0	6.5	4.0
P3	Stone, sand, rubber, cement	1.0	Water, SP	3.0	–	–	–	4.0	3.0
P4	Stone, sand	1.0	1/2 water	1.0	2.0	(Rubber+cement), 1/2 water, SP	2.0	6.0	2.0
P5	Stone, sand, rubber, cement, water, SP	2.0	–	–	2.0	–	2.0	6.0	4.0
P6	Stone, sand	1.0	1/2 water	1.0	2.0	(Rubber+SP+1/2 water), cement	2.0	6.0	2.0

Mixing procedure P1 was followed in all mixes that included fibre additives, and the fibres were added with all dry materials from the mix at the start. In Table 3, T_n is the net mixing time in which all concrete constituents were added and mixed together.

2.3. Rubber Particle Pre-Treatment

Pre-treatment of rubber particles can play an important role in improving the adhesion at the rubber/cement interface in the concrete matrix, as it can remove impurities and change the surface morphology and topography. In this study, different pre-treatment approaches were used according to the previous studies [28,32–36] and their suggestions for better Rubcrete performance. The approaches used were: Water washing, water soaking-A, water soaking-O, NaOH, H_2O_2, $CaCl_2$, H_2SO_4, Silane, and $KMnO_4$_$NaHSO_4$. In the water washing method, the rubber was washed by tap water for 5 min in a container using a narrow outlet water hose with high water pressure, that was able to keep flipping the rubber in the water while washing. The rubber was then filtered from the water and left to air dry. In the water soaking-A method, the rubber particles were soaked in tap water for 24 h in a container, before being washed and filtered, and then left to air dry. The same procedures were followed in the water soaking-O method; however, the rubber was dried in a 100 °C oven for 6 h for quicker drying. In both NaOH and H_2O_2 methods, the rubber was submerged in a 10% solution of the chemical for 30 min. The rubber particles were then washed by stirring in tap water until their pH became 7, and then they were left to air dry. The same procedures were used in the $CaCl_2$ method, except that

the rubber particles were not washed in water and were directly left to air dry after being soaked in 10% $CaCl_2$ solution for 24 h. The H_2SO_4 method was the same as the NaOH one, but the rubber particles were soaked in 35% H_2SO_4 solution for 24 h. In the silane method, the procedure began with preparing a 1% concentration silane solution, stirred for 10 min, then the rubber particles were added to the solution and stirred for 20 min. This was followed by heating the solution, including rubber, at 80 °C for 30 min while stirring, and then the solution was cooled to room temperature. The rubber particles were then rinsed in alcohol by filtration and left to air dry. The $KMnO_4$_$NaHSO_4$ method was a double treatment method including oxidation and sulphonation of the rubber particles. In this method, the rubber particles were soaked in 5% $KMnO_4$ solution that was heated at 60 °C for 2 h, and then cooled to room temperature. The rubber particles were then filtered from the $KMnO_4$ solution, and directly soaked in a 5% $NaHSO_4$ solution that was heated at 60 °C for 1 h, and then cooled to room temperature before filtration, stirring in tap water until its pH became 7, and leaving to air dry.

2.4. Preparation and Testing of Specimens

The concrete workability for each mix was assessed using the standard slump test in accordance with AS 1012.3.1 [42]. For each mix, three 100 × 200 mm cylinders were prepared to test the 28-day compressive strength. Three additional cylinders were prepared for mixes M1-M14 and M29-M31 to evaluate the indirect tensile strength at 28 days, resulting in a total of 144 cylinders for these two test sets. 28 prisms of 100 × 100 × 470 mm were poured to test the flexural strength at 28 days, two prisms each from mixes M15-M28. A standard compaction rod and hammer were used to compact the concrete in all cases.

After 24 h, all specimens were de-moulded, labelled and placed in a water bath for curing at 23 ± 2 °C, in accordance with AS1012.8.1 [43]. The compression tests were carried out in accordance with AS 1012.9 [44], using a 1500 kN capacity testing machine with a constant loading rate of 20 ± 2 MPa/min. Indirect tensile tests were performed according to AS 1012.10 [45], using a 1500 kN capacity testing machine with constant loading rate of 1.5 ± 0.15 MPa/min. Flexural strength tests were performed according to AS 1012.11 [46], using a 100 kN capacity testing machine with a constant loading rate of 1.0 ± 0.1 MPa/min.

X-ray photoelectron spectroscopy (XPS) analysis was carried out for selected rubber pre-treatments to measure the elemental composition at the rubber surface before and after the different treatments. This was done on a Kratos Axis-Ultra spectrometer, using a monochromatic Al Kα source (1487 eV) operating at 15 kV and 14 mA, 10^{-8} Pa vacuum in the analyser chamber, and an analysis spot size of 300–700 μm. A spectrometer pass energy of 40 eV was used for all elemental spectral regions, while 160 eV pass energy was used for the survey spectra for element identification and surface atomic concentration calculations. The binding energy scale of the spectrometer was calibrated using the metallic Cu $2p_{3/2}$ and Cu $3p_{3/2}$ lines, and Au Fermi Edge of the respective reference metals. Core electron binding energies are given relative to C–C/C–H in hydrocarbon (sample or adventitious) at a C 1s binding energy of 284.8 eV.

3. Experimental Results and Discussion

The effects of Rubcrete mixing procedures, pre-treatment of rubber particles, and the addition of fibres are indicated in the experimental results summarised in Table 4. These results demonstrate that the addition of crumb rubber to the mix increased the concrete slump, but decreased the compressive strength, tensile strength, and flexural strength. This can be observed by comparing the results of mixes M1 to M3, and M15 to M16. Using 15%, 20%, and 30% rubber contents increased the concrete slump by 7%, 48%, and 40%, respectively. Concrete slump is an important indicator of the workability of concrete for construction, and is influenced by a range of factors, including particularly the water content and the ratios of cement to aggregates, and coarse to fine aggregates [47]. When sand is replaced by crumbed rubber, the free water content within the concrete matrix is increased, as are the

weight ratios of cement to aggregate and coarse to fine aggregates. This is because rubber particles have relatively lower water absorption, hydrophilicity (affinity to water) and unit weight compared with the sand they replace, which hence increases the slump. Using 15%, 20%, and 30% rubber contents decreased the compressive strength by 30%, 34%, and 43%, respectively. Using 15% and 30% rubber contents decreased the indirect tensile strength by 14.5% and 28.5%, respectively; and using 20% rubber content decreased the flexural strength by 17%. These strength decreases when crumb rubber partially replaces sand in concrete are generally attributed to the lower adhesion at the rubber/cement interface. This results in easier separation of rubber from the cement paste around it at small stresses, hence generating weak points at the interface. In addition, the significant difference in the stiffness and hence the relative deformations between rubber aggregates and concrete paste leads to early cracking, and hence strength reduction.

Table 4. Experimental results.

Mix Code	Rs (%)	Mixing Procedures	Pre-treatment	Slump (mm)	Compressive Strength (MPa)	Tensile Strength (MPa)	Flexural Strength (MPa)
M1	0	P1	–	145	53.3	4.62	–
M2	15	P1	–	155	37.5	3.95	–
M3	30	P1	–	203	30.5	3.30	–
M4	15	P2	–	215	41.2	4.10	–
M5	30	P2	–	230	31.5	3.21	–
M6	15	P3	–	210	40.3	3.98	–
M7	30	P3	–	220	29.8	3.27	–
M8	15	P4	–	195	38.5	3.91	–
M9	30	P4	–	220	31.2	3.44	–
M10	15	P5	–	225	39.3	3.91	–
M11	15	P6	–	190	35.8	3.85	–
M12	15	P4	Water wash	160	40.8	4.30	–
M13	15	P4	Water soaking-A	113	37.1	3.68	–
M14	15	P4	Water soaking-O	105	35.7	3.90	–
M15	0	P1	–	135	41.5	–	6.0
M16	20	P1	–	200	27.3	–	5.0
M17	20	P1	NaOH	180	29.2	–	5.0
M18	20	P1	H_2O_2	183	27.5	–	4.9
M19	20	P1	$CaCl_2$	162	29.1	–	5.4
M20	20	P1	H_2SO_4	150	27.4	–	5.5
M21	20	P1	Silane	185	28.9	–	5.2
M22	20	P1	$KMnO_4$_$NaHSO_4$	183	26.5	–	5.3
M23	20	P1	–	120	28.6	–	5.8
M24	20	P1	–	65	24.4	–	5.4
M25	20	P1	–	32	26.9	–	8.3
M26	20	P1	–	120	28.0	–	5.2
M27	20	P1	–	75	27.9	–	4.6
M28	20	P1	–	45	25.6	–	7.0
M29	15	P1	–	150	35.7	3.66	–
M30	15	P1	–	125	31.7	3.35	–
M31	15	P1	–	113	30.7	3.36	–

3.1. Effect of Rubcrete Mixing Procedures.

The effects of the proposed mixing procedures on Rubcrete slump, compressive strength, and tensile strength were determined through a comparison of the results of the mixes M2 to M11. Mixes M2, M4, M6, M8, M10 and M11 are comparable at 15% rubber content. Mixes M3, M5, M7, and M9 are comparable at 30% rubber content. It was generally observed that the net mixing time of all mix constituents together (T_n) was more effective than the change in any mixing order of the

concrete constituents. As shown in Figure 3a,b, increasing T_n enhanced the Rubcrete slump. P2 and P5 (T_n = 4 min) showed higher slump higher than P3 (T_n = 3 min) by an average of 5% and higher than P1, P4, and P6 (T_n = 2 min) by an average of 22%. A similar observation was recorded when using 30% rubber content but with less significance, as shown in Figure 3b. The relatively longer time that all concrete constituents were mixed together might allow the SP to work well in dispersing the cement particles and hence, provide more water in the matrix to enhance the Rubcrete workability.

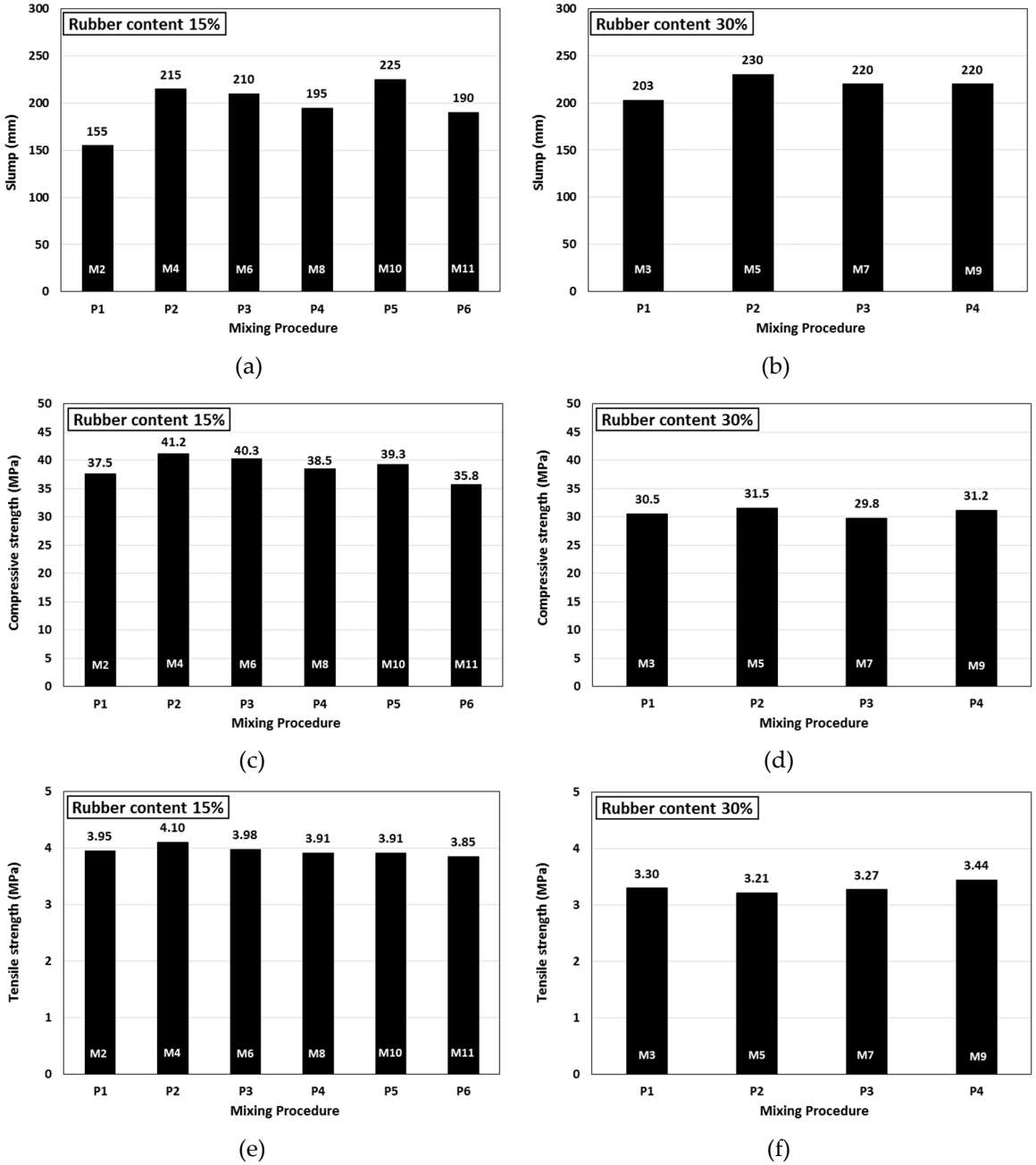


Figure 3. Effects of mixing procedures on Rubcrete: (**a**,**b**) slump, (**c**,**d**) compressive strength, and (**e** and **f**) tensile strength.

The Rubcrete mixing procedures had a lesser effect on the compressive strength than the slump, as shown in Figure 3c,d. At the same T_n (P1, P4, and P6), mixing 15% or 30% rubber content with dry cement before adding to the mix (P4) slightly increased the compressive strength by 3% or 2%, respectively. However, mixing 15% rubber content with water+SP before adding to the mix (P6)

deceased the compressive strength by 4.5%, compared to mixing procedures P1, which had rubber added with other aggregates from the start of mixing. This was attributed to the ability of cement fine particles to attach to the rubber surface when mixing with dry cement, which relatively enhanced the rubber/cement interface adhesion. However, when the rubber was first mixed with water+SP, this could decrease the chance of fine cement particles reaching the rubber surface due to the rubber hydrophobicity, resulting in strength reduction. Doubling T_n from 2 min to 4 min increased the compressive strength by an average of 8% at 15% rubber content, and by an average of 2% at 30% rubber content. This was attributed to the same reasons of enhancing the concrete workability where the cement particles can be better dispersed within the concrete matrix and hence, strength increases. No significant effect of Rubcrete mixing procedures on tensile strength was recorded, as shown in Figure 3e,f.

3.2. Effect of Rubber Pre-Treatment

The effects of various crumb rubber pre-treatment processes on the slump, compressive strength, tensile strength, and flexural strength of the Rubcrete were evaluated by comparing the results of mixes M8, M12 to M14, and M16 to M22. Mixes M8 and M12 to M14 are comparable at 15% rubber content, and investigated different methods of rubber pre-treatment using tap water. Mixes M16 to M22 are comparable at 20% rubber content, and investigated different chemical pre-treatment of rubber particles. As shown in Figure 4a,b, all the rubber pre-treatment methods resulted in Rubcrete slump decrease. This may be attributed to the ability of the pre-treatment material to clean the rubber surface of impurities that can be present as a result of manufacturing and usage, and which can increase the Rubcrete flowability when present. However, microstructural analysis of the pre-treated rubber is recommended for future studies to confirm this observation.

The effect of using water in pre-treating the rubber particles on Rubcrete slump was more pronounced than the effect of the other chemicals used, see Figure 4a. Using methods of water washing, water soaking-A, and water soaking-O decreased the slump by 18%, 42%, and 46%, respectively. Water soaking-A and water soaking-O methods showed lower slump than that of water washing, due to the relatively longer time of soaking rubber in water used in these two methods (24 h soaking). The oven drying of rubber used in water soaking-O led to a relatively higher rate of water evaporation from rubber particles' surfaces and hence, less slump. Using NaOH, H_2O_2, $CaCl_2$, H_2SO_4, silane, and $KMnO_4$_$NaHSO_4$ decreased the Rubcrete slump by 10%, 8.5%, 19%, 25%, 7.5%, and 8.5%, respectively. Both $CaCl_2$ and H_2SO_4 displayed slump values lower than those presented by other chemical pre-treatments. Noting that in these two methods, the rubber particles were treated in their solutions for 24 h, compared with only 0.5–3.0 h used in other methods, indicated again that regardless of the treatment material type, the longer the time of the treatment, the more rubber cleaning and hence, lower slump.

The effect of the rubber particle pre-treatment on the Rubcrete compressive strength, tensile strength and flexural strength, was insignificant, as shown in Figure 4c–f. The variations in the compressive strength, tensile strength, and flexural strength were about −2% to +10% which is actually not worth the effort and cost needed to complete each pre-treatment process. It is generally agreed that the poorer bond between rubber and cement, and the significantly lower stiffness of the rubber particles compared with other ingredients in concrete, result in less contribution to stress transfer, leading to a lower strength for Rubcrete. To improve the strength properties of Rubcrete, it is suggested that the future investigations on applying pre-treatment methods should be focused on using coating materials to improve the stiffness of the rubber particles.

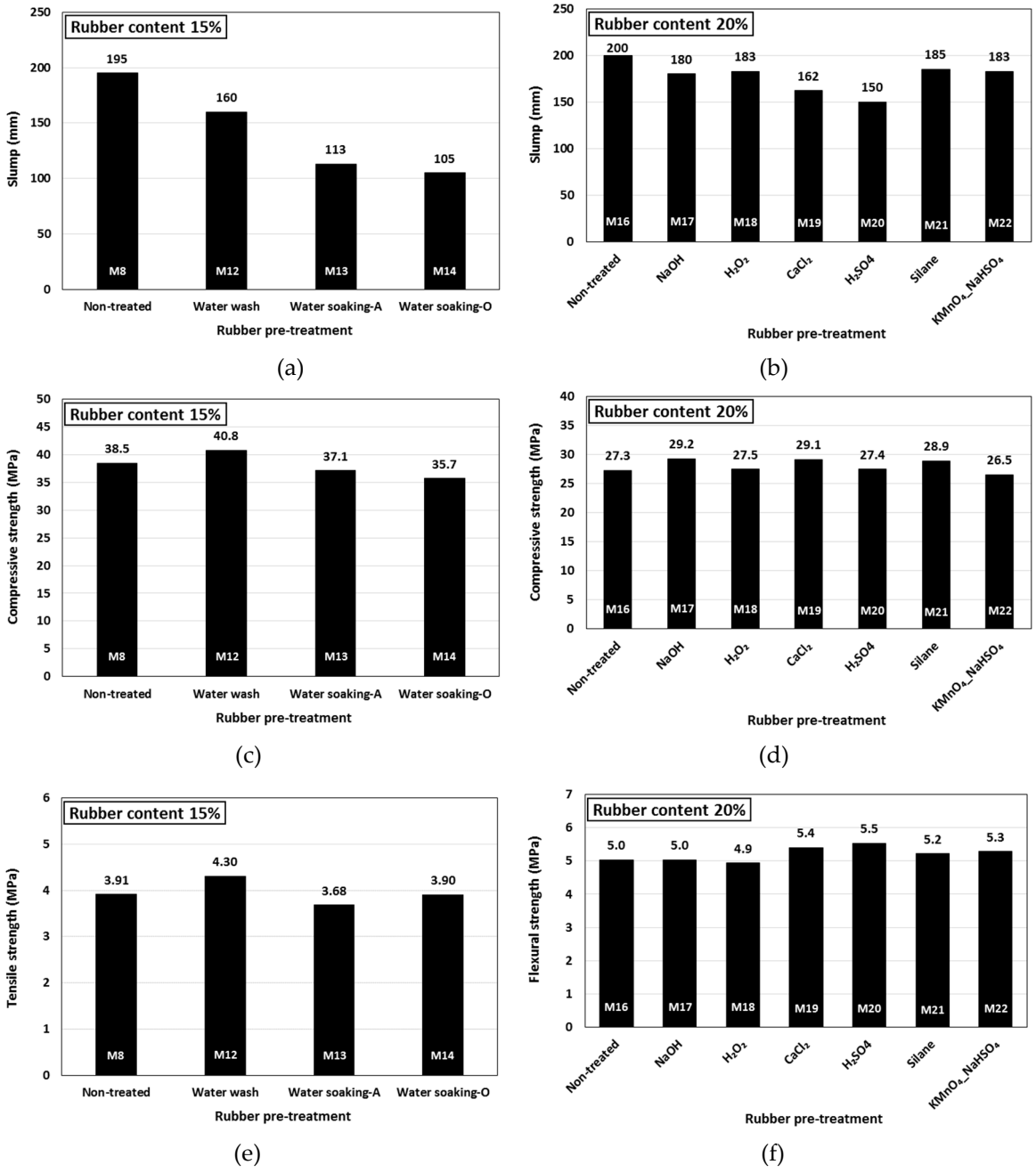


Figure 4. Effects of rubber pre-treatment on Rubcrete: (**a**,**b**) slump, (**c**,**d**) compressive strength, (**e**) tensile strength, and (**f**) flexural strength.

The major observations from the XPS analysis are manifested in the variation of the carbon and oxygen surface concentrations, which were the dominant constituents at the rubber surface. As shown in Figure 5a, all rubber pre-treatment methods resulted in a decrease in carbon (by an average of 11%) as a result of increased oxygen concentration (by an average of 4.7 times) in the surface. The increased oxygen in the rubber surface is correlated with an increase in carbon-oxygen bonding as a result of the various treatments. Figure 5b presents the C 1s and O 1s region comparison between non-treated and NaOH-treated rubber. Untreated rubber exhibits only a small contribution from oxidised carbon species, consistent with atmospheric exposure. Post-treatment surfaces show a dramatic increase in abundance and type of carbon-oxygen functionalities. New C 1s contributions are consistent with a range of species, including ether/–COH, carbonyl and carboxyl [48]. Oxygen functionalities at the rubber surface can increase its hydrophilicity (wettability) which enhances the rubber/cement

water transfer rate and hydration at the interface, thus improving rubber/cement adhesion. However, as discussed above, pre-treatment has only a small effect upon strength improvement in Rubcrete via improving the rubber to cement bond. However this mechanism is limited, as the main factor causing the strength reduction in Rubcrete is the considerable difference in stiffness of rubber particles and concrete paste.

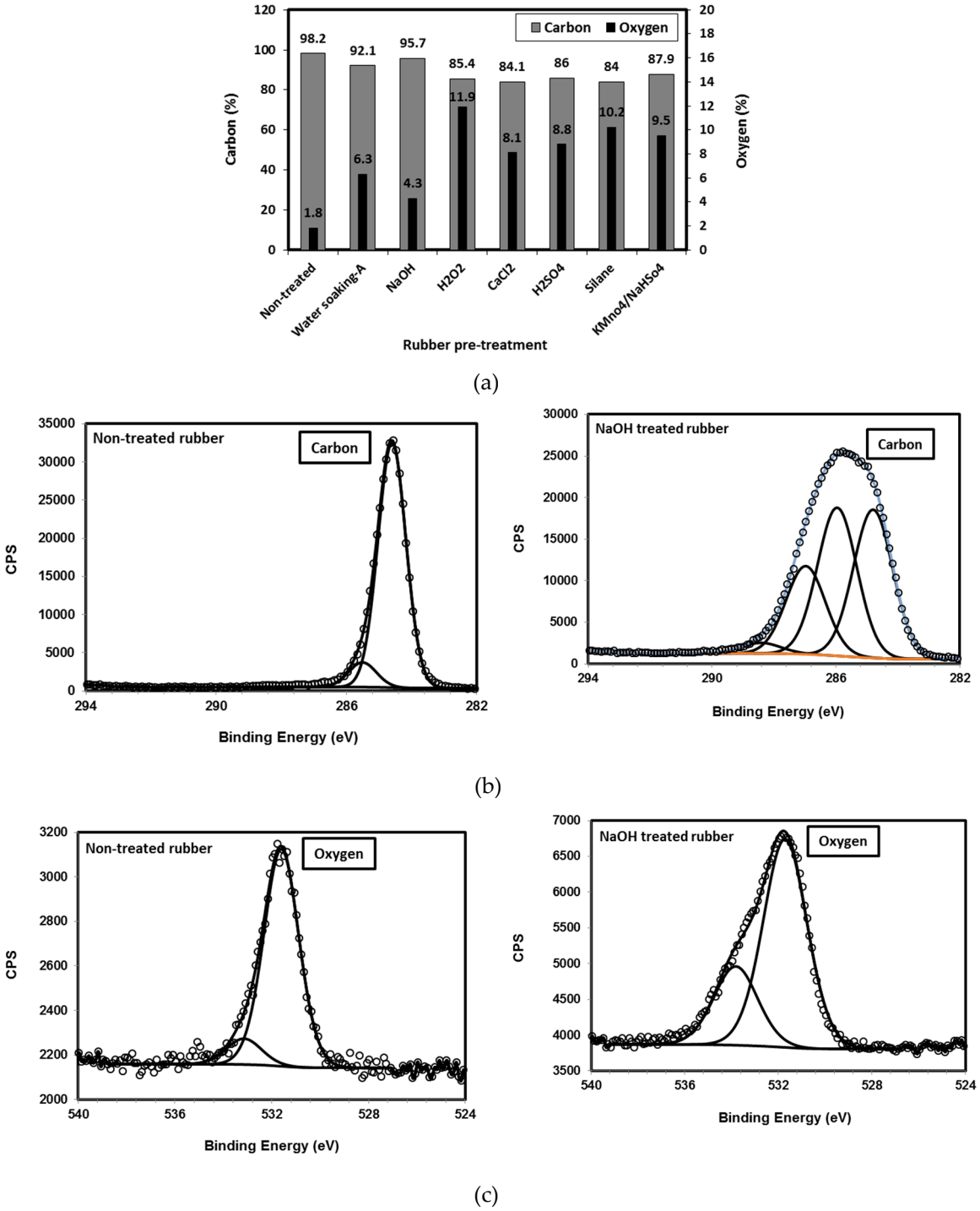


Figure 5. X-ray photoelectron spectroscopy (XPS) analysis results. (**a**) Results summary. (**b**) Carbon survey spectrum of non-treated and NaOH-treated rubber. (**c**) Oxygen survey spectrum of non-treated and NaOH-treated rubber.

3.3. Effect of Fibre Additives

The effects of different fibre additives on Rubcrete slump, compressive strength, tensile strength and flexural strength, were determined through comparison of the results of mixes M2, M16, and M23 to M31. Mixes M16 and M23 to M25 are comparable for the effect of steel fibre additive. Mixes M16 and M26 to M28 are comparable for the effect of polypropylene fibre additive. Mixes M2 and M29 to M31 are comparable for the effect of rubber fibre additive. As shown in Figure 6a,b, increasing the fibre volume fraction in Rubcrete decreased its slump due to the high aspect ratio of the fibre by nature, compared to any other constituent in the concrete matrix, which causes difficulties in the concrete mobility, and the mix becomes much stiffer. Using 0.5%, 1.0%, and 1.5% fibre content decreased the Rubcrete slump by 40%, 68%, and 84%, respectively for steel fibre; and by 40%, 63%, and 78%, respectively for polypropylene fibre (Figure 6a). The steel fibre caused higher slump losses due to the fibre rigidity that could slow the concrete mobility, compared with that of polypropylene fibre, especially at the high fibre content. Using 1.0%, 2.0%, and 3.0% fibre content decreased the Rubcrete slump by 3%, 19%, and 27%, respectively for rubber fibre (Figure 6b). The rubber fibre effect on Rubcrete slump was less significant than those of steel and polypropylene fibres due to its relatively smaller average length and aspect ratio, and the higher flexibility of the rubber fibre used, which can decrease such slump reduction.

Figure 6c,d shows the effect of fibre additive on Rubcrete compressive strength. Using steel or polypropylene fibres up to 1.5% fibre content showed insignificant effects on the Rubcrete compressive strength with maximum strength variation of −11% to +5%. Using up to 1% rubber fibre content slightly decreased the Rubcrete compressive strength by 5%; however, increasing the rubber fibre content to 2% and 3% decreased the Rubcrete compressive strength by 16% and 18%, respectively. The insignificant effect of fibre up to 1.5% fibre content is attributed to the inability of fibres to resist the shear stresses developed between the relatively soft cementitious matrix and the stiffer aggregates, which is the main reason for concrete failure under axial compression. However, it could resist the tensile stresses in the cementitious matrix, which may result in slight strength increase. The higher strength losses with the higher rubber fibre content were attributed to the lesser axial capacity of rubber fibre compared with the replaced volume of other concrete constituents.

Figure 6e shows the variation of the Rubcrete flexural strength with an increase in the fibre content. As shown in the figure, there was no significant effect up to 1% fibre content (+8 to +16% change with using steel fibre, and −8% to +4% change with using polypropylene fibre).

However, relatively significant Rubcrete flexural strength increase (70% when using steel fibre, and 40% when using polypropylene fibre) occurred when using 1.5% fibre content of both steel fibre and polypropylene fibre. The steel fibre was more effective than the polypropylene fibre, due to its relatively high tensile strength and rigidity, that delayed the initiation of the cracks it was bridging, compared to that of polypropylene fibre.

Similar to its effect on Rubcrete compressive strength, using 1%, 2%, and 3% rubber fibre in Rubcrete decreased its tensile strength by 7%, 15%, and 15%, respectively, as shown on Figure 6f. Due to the flexibility and high Poisson's ratio of rubber fibre, it is supposed to work well in enhancing the tensile strength of concrete through bridging cracks. However, this flexibility, in addition to the rubber surface nature, causes a poor bond between the rubber and the surrounding cementitious material. This results in non-completion of the bridging mechanism that needs good supports (bond) to take the reaction of each fibre under tension and hence leading to a similar trend to that of the corresponding compressive strength, with no effect on tensile strength.

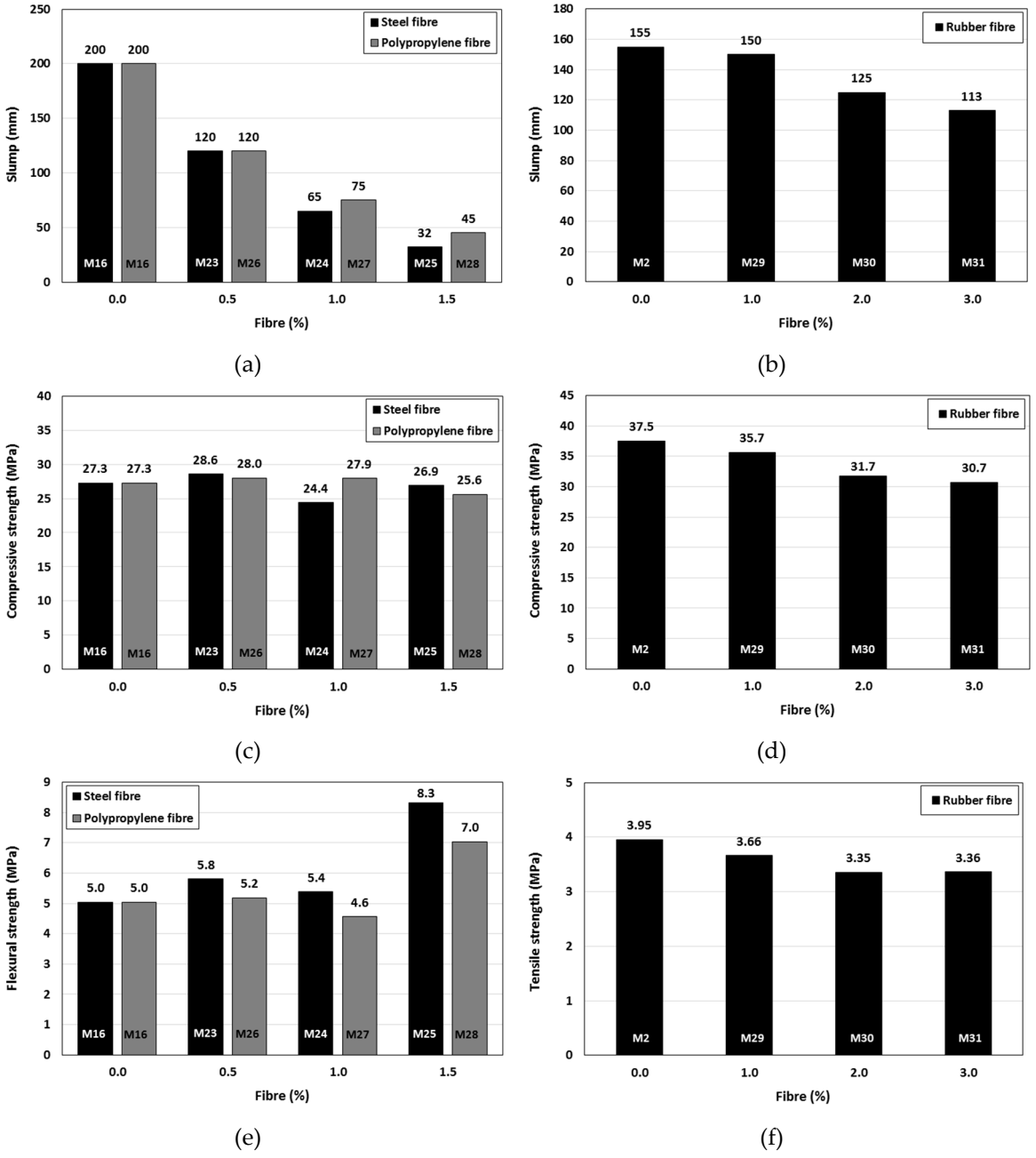


Figure 6. Effects of fibre additives on Rubcrete: (**a**,**b**) slump, (**c**,**d**) compressive strength, (**e**) flexural strength and (**f**) tensile strength.

4. Summary and Conclusions

This paper investigates the effect of different mixing procedures, rubber chemical pre-treatments, and fibre additives on Rubcrete workability, compressive strength, tensile strength, and flexural strength through the testing of 31 concrete mixes with a range of rubber contents. X-ray photoelectron spectroscopy (XPS) analysis of some of the pre-treated rubber were also carried out. The main findings of this investigation are summarised in the following points:

1. Partial replacement of concrete sand by crumb rubber increased the concrete workability, and decreased its compressive strength, tensile strength, and flexural strength.

2. Doubling the net mixing time, T_n, enhanced the Rubcrete slump by an average of 22% and the compressive strength by an average of 2–8%. Mixing rubber with dry cement before adding to the concrete mix was more effective and hence, it is suggested.

3. All rubber pre-treatment methods used resulted in a reduction in the workability of Rubcrete, but had no significant effect on Rubcrete compressive strength. Pre-treatment using water wash is recommended for practical use. It is suggested that future investigations on applying pre-treatment methods should be directed towards using coating materials to enhance the stiffness of the rubber particles, rather than just improving the rubber to cement bond.

4. XPS analysis showed that all rubber pre-treatment methods used resulted in decreasing the Carbon component by an average of 11%, and increasing the Oxygen component by an average of 4.7 times at the rubber surface, which enhances the rubber hydrophilicity.

5. Using fibre additives decreased the Rubcrete slump, regardless the fibre material type. Steel or polypropylene fibres showed insignificant effect on the Rubcrete compressive strength. However, rubber fibre decreased the Rubcrete compressive strength and tensile strength. Significant Rubcrete flexural strength increase occurred when using a 1.5% fibre content of both steel fibre and polypropylene fibre.

In summary, this research suggests that pre-treatment methods are probably not worth the time and cost involved. Simply washing the rubber with water to decrease surface impurities, mixing the rubber with dry cement at the start of the mix process, and using slightly longer mixing times, are able to produce some enhancement of slump and concrete strength, with no additional cost. Fibre reinforcement was also shown to provide no useful enhancement to strength and significant decrease to workability. It is recommended that the reduced strength of Rubcrete should be accepted for what it is, and investigations could be better focused on practical applications for Rubcrete, like residential footings and slabs, that suit or compensate for the reduced strength available from Rubcrete, compared with traditional concrete.

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