



# Effects of surface treatments of recycled tyre crumb on cement-rubber bonding in concrete composite foam



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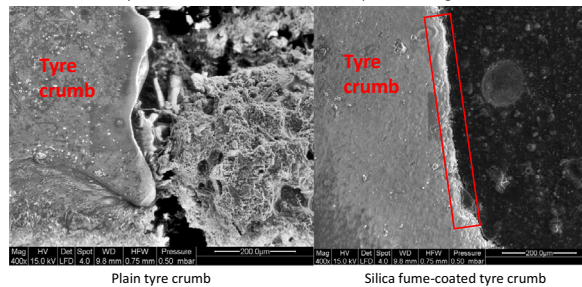
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## HIGHLIGHTS

- Five surface treatments of tyre crumbs yielded strength improvements of 27–56%.
- Silica fume coating improves the adhesion between tyre crumbs and concrete foam.
- A denser gel with lower Ca/Si was observed around crumbs for silica fume treatment.
- Chemical treatments reduced water contact angle on rubber (increased hydrophilicity).
- Sulfuric acid etched the crumbs and formed zinc sulphate crystals on the surface.

## GRAPHICAL ABSTRACT

Silica fume-coated tyre crumb created a denser ITZ with superior bonding with the cement matrix



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## ABSTRACT

The uptake of recycled tyre crumb (RTC) in concrete can result in a significant loss of strength because of weak adhesion between cement and rubber. Five RTC surface treatment methods (cement coating, silica fume coating, sodium hydroxide, potassium permanganate and sulphuric acid soaking) were used to mitigate strength loss in concrete composite foam with RTC. All five methods yielded strength improvements between 27% and 56%, with sulphuric acid and silica fume coating having the largest improvement. Silica fume coating, a treatment method used for the first time, improves strength because of the better adhesion between RTC and mortar. In addition, the formation of low Ca/Si gels was observed at the rubber-mortar interface based on SEM-EDS analysis. A reduced water contact angle on rubber showed that the three soaking methods were able to successfully reduce the hydrophobicity of RTC. Microscopic analysis of sulphuric acid treated RTC revealed etching of the surface and possible formation of polar zinc sulphate crystals. Silica fume coating provides an economically feasible solution with less safety and environmental risks associated compared to the chemical treatments.

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## 1. Introduction

Only 7.8% of 56 million end-of-life tyres in Australia were recycled and approximately 56% were disposed into landfills or illegally

dumped [37]. The non-biodegradable nature of vulcanised rubber in the tyre can lead to serious environmental and social impacts, such as toxic fire hazards and breeding habitats for insects and pests which can increase the spread of disease. With improvements in material technologies, the use of waste tyre in innovative engineering applications such as asphalt binders, non-structural concrete, playground surfaces and fuel derivation have been explored [30,29,4,25].

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The use of recycled tyre crumb (RTC) in concrete has shown improved elasticity, ductility, abrasion resistance and damping properties, as well as sound and heat insulation [27,16,6,33,15]. Thomas and Gupta [34] reviewed the recent applications and properties of waste tyre in concrete. The durability of concrete with waste tyre as a partial replacement of aggregates is excellent as it is extremely resistant to aggressive environments such as acids [35]. The lower specific gravity of tyre crumbs also results in the reduction of the unit weight of concrete. However, the uptake of RTC in concrete has been hindered by reduced compressive strength [5,36,17,13]. Also, the decomposition of the tyre at elevated temperature (above 150 °C) is shown to adversely affect residual properties of concrete with tyre crumb at above 10 wt% replacement of sand with tyre crumbs [10]. Loss of strength has primarily been attributed to poor adhesion between RTC and cement mortar due to factors such as the hydrophobic (water repelling) nature of RTC [3]. Furthermore, replacing rigid material i.e. aggregates with soft rubber crumbs significantly lowers stiffness in concrete and creates stress concentrations at the RTC-cement interface which results in the crack formation and strength reduction [5,18,23]. Previous work has adopted different techniques to treat the RTC surface in order to improve adhesion between rubber and concrete, which is briefly reviewed.

### 1.1. Literature review on RTC surface treatment

Treatment of tyre crumbs with sodium hydroxide (NaOH) solution has been widely used to increase adhesion between cement and rubber by etching the surface of the crumbs resulting in increased strength [26,1,19,8]. Additionally, it is argued that NaOH treatment converts zinc stearate compound (an admixture used in tyre manufacturing that takes part in hydrophobic characteristics of the tyre crumb surface) to a soluble form of sodium stearate that can be removed by washing with water [27,20].

Silane coupling agents have been explored to alter surface properties of tyre crumbs. Studies report that the formation of hydrogen bonds between rubber and cement leads to increased adhesion [19,8]. Compressive strength improvements up to 25% for samples comprising of 30% vol. of silane treated RTC have been reported [3]. Studies have also looked at combining treatment methods to obtain a better result. He et al. [12] used a three-stage process: initially soaking RTC in NaOH (24 h) to clean the surface; using potassium permanganate (KMnO<sub>4</sub>) and sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) for oxidation; and using sodium bi-sulphite to sulphonate the rubber. The combined three-stage treatment resulted in a 47% increase in compressive strength for concrete comprised of 4% rubber crumb per cement mass. The hydrophobic properties of the crumbs were partially alleviated through reduced water contact angle (WCA) measurements on the tyre crumbs compared to as-received rubber [12].

Solvents such as acetone can also cause surface alterations of RTC and acetone-treated rubber has been shown to improve compressive strength compared with untreated rubber [24]. Ossola and Wojcik [22] exposed RTC to ultraviolet (UV) light. Their results confirmed that UV exposure (20–60 h) of rubber improved the flexural strength of concrete samples with 15% RTC by weight of cement. Huang et al. [14], utilised a two-stage treatment: silane coupling agent to modify the tyre crumbs; and coating RTC with cement paste to develop a 'hard shell', which resulted in significant improvements in compressive strength.

Onuaguluchi [21] used limestone powder to pre-coat the surface of rubber crumbs and added silica fume at 10% volume as a supplementary cementitious material in the paste. Image analysis showed that the adhesion at the interfacial transition zone (ITZ) between rubber and cement was affected, and the voids were filled. Amongst existing research, there exists an abundant indica-

tion that silica fume as a supplementary cementitious material can improve the strength of concrete. This has been mainly attributed to increased pozzolanic activity giving rise to the increased formation of calcium silicate hydrate (C–S–H) gels. Additionally, the small size of silica fume particles results in filling the voids at the rubber-cement interface [7,28,9,38].

Previous research in the field on improving the mechanical properties (compressive strength) of rubber concrete through tyre crumb surface treatment has focussed mainly on traditional concrete. The effectiveness of these methods in lightweight concrete composite foam with tyre crumb has not been explored extensively. Concrete foam is formed in a process of aeration and has a high strength to weight ratio, as well as excellent thermal and sound insulation properties [11]. It can be used for various non-structural construction applications such as precast or cast-in-situ wall elements, slabs, blocks, facades, insulation screens, oil-well cementing or as the core of sandwich panels. Kashani et al. [15] showed that the application of RTC in the concrete foam can result in a sustainable material with improved durability, thermal and sound insulation. However, RTC resulted in a reduction of compressive strength of the concrete composite foam. Highly aerated concrete results in considerably less contact (adhesion) between cement and rubber. Therefore, the limited bonding between cement and rubber needs to be fortified in order to alleviate substantial strength reduction by the inclusion of RTC in concrete foam.

This study investigates five different methods used for surface treatment of tyre crumbs, namely sodium hydroxide, potassium permanganate and sulphuric acid soaking, as well as cement and silica fume coating (i.e. a new method which is investigated for the first time in this study). The coating of tyre crumbs with silica fume slurry would result in a cost-effective application of this expensive material because a considerably lower amount of silica fume is consumed compared to its use as a supplementary cementitious material in the paste. Furthermore, this method would alleviate strength reduction at the source of the issue, which is the interfacial transition zone (ITZ) between cement and rubber. In addition, a comprehensive analysis was performed to justify the strength improvement in RTC, namely scanning electron microscopy (SEM) together with an elemental analysis of energy-dispersive X-ray spectroscopy (EDS) at the ITZ, as well as measurement of the water contact angle on the treated RTC. The results of this study provide a fundamental understanding which compares different surface treatment methods of tyre crumbs, resulting in improved strength of concrete composite foam comprising recycled tyre crumb.

## 2. Research methodology

### 2.1. Mixture proportion and treatment methods

General purpose cement in compliance with Australian Standard (AS 3972) was sourced from a local supplier. The recycled tyre in the form of single-graded crumb rubber (2.36–4.75 mm) was sourced from Tyrecycle, Australia. Tyre crumbs had an estimated specific gravity of 1100 kg/m<sup>3</sup>. The rubber content in the samples was maintained at 10% by weight of cement. Fine sand is normally used as aggregate in concrete foam. In this study, tyre crumbs were used as lightweight aggregates for the potential replacement of sand. A water to cement ratio of 0.45 and a commercially available foaming agent (Isochem S/X) at 0.2% weight of cement was used. A control sample was prepared with as-received rubber (no treatment). Five different treatment methods were applied to the rubber crumbs as shown in Table 1. KMnO<sub>4</sub>, H<sub>2</sub>SO<sub>4</sub> and NaOH were sourced from Chem-supply, Australia with impurities below 1, 2

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