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Surface and bulk hydrophobic cement composites by tyre rubber addition

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HIGHLIGHTS

- Tyre-rubber addition in cement mortars abates penetration of water drops.
- Hydrophobic character is shown both on the surface and in the bulk of the mortar.
- Smaller rubber grains average size enhances mortar hydrophobicity.
- Rubber addition increases porosity, nevertheless it can hinder liquid water entrance.

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ABSTRACT

Penetration of water in cement composites, porous and hydrophilic materials, is cause of progressive deterioration and failure. Standard procedures for protecting building structures generally involve uniquely the modification of the surface by coating or impregnation procedures.

In this work, the addition of tyre rubber (TR) to the cement paste is demonstrated to be effective for developing mortars with a pronounced hydrophobic behavior in every part of their structure. Hydrophobic performances are better in the case of finer TR grains size and for larger TR volume addition. TR mortars show higher porosity than the conventional ones, nevertheless the effect of the low rubber surface energy prevails, and the absorption of water drops is almost completely abated. These lightweight materials result to be very competitive for non-structural applications and are in agreement with the environmentally sustainable policies finalized to convert a synthetic waste to an engineering resource.

1. Introduction

The increasing number of vehicles on the roads of industrialised and developing nations generates millions of end-of-life (ELT) tyres (about 1.4 billion tyres are sold worldwide every year) which are a large and problematic source of waste, due to the large volume and long durability. The limited space and their potential for reuse has led many countries to impose a ban on the practice of landfilling. The estimated EU annual cost for the management of ELTs is estimated at € 600 million [1,2].

Tyre rubber is resistant to mould, heat humidity, bacterial development, ultraviolet rays, some oils, many chemicals. These characteristics, which are beneficial during on-road life, are disadvantageous in post-consumer life and boost the transformation of this material from an environmental problem to engineering resource.

One of the recovery routes is the so called "granulate recovery" which involves tyre shredding and chipping, by which tyres are cut into small pieces of different sizes (shreds: 460–25 mm; chips: 76–13 mm; crumb rubber: 5–0.1 mm) [1]. After the removal of the steel and fabric components, the *recycled tyre rubber* (RTR) can be used for a variety of civil engineering applications such as, i.e., soft flooring for playgrounds and sports stadiums, modifier in asphalt paving mixtures or additive/aggregate to cement concrete. Among these, the addition (as crumb rubber) to asphalt mixtures is highly diffused due to the good chemical interaction, even leading to a partial dissolution [3,4].

The recovery of RTR as aggregate in cement structures has been proposed since the 90's but it is considered not convincing compared to applications in asphalt pavements [3,5,6]. An important reason is the not favorable interaction with the matrix. Indeed,

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the cement paste is mainly characterized by hydrated metal/semimetal oxides, which explains the hydrophilic nature (high surface energy) of this matrix and the good adhesion to the conventional aggregates generally based on quartz and/or limestone. Rubber, instead, made of organic polymers, is characterized by a low surface energy, and therefore by a hydrophobic character. The interaction hydrophilic-hydrophobic is very unfavorable resulting in a poor adhesion between rubber particles and the cement matrix. Various rubber chemical treatments have been lately tested with the purpose of improving adhesion. Among these, treatments with NaOH [7–9], HNO_3 and cellulosic derivatives [10] or silane coupling agents [11] have been reported.

More importantly, lower compression resistances are always observed in rubber-cement composites with respect to the conventional ones [5,12]. This is mainly due to the fact that rubber sites are significantly softer than their surrounding media acting like "holes" inside the concrete. For this reason only non-structural applications have been proposed (exterior wall materials [13], pedestrian blocks, highway sound walls, residential drive ways, and garage floors [3]) and no building practice seems to be diffused.

However, an enhancement of toughness and ability to absorb impact energy has been somewhere observed (somewhere also explained and modeled), also in addition to an increased flexural strength [3,12].

Further, the lightweight character of the rubberized materials (due to the low specific weight of rubber) should be considered an advantage for the use as construction material since nowadays the structural efficiency is more important than the absolute strength level. Specifically, a decreased density for the same strength reduce the dead load, foundation size, and construction costs; it also enhances sound and thermal insulation [14].

Our objective is to focus on a specific feature of the rubber-cement composites, i.e. the low surface energy of the rubber particles which, although responsible of a low adhesion to the cement paste, should inhibit the absorption of water in artifacts.

This is a relevant applicative feature since hydrophobic cement structures have i) longer durability upon freezing-thawing cycles, as opposite to conventional porous and hydrophilic composites which, after water absorption, tend to expand on freezing thus starting cracks within the matrix; ii) self-cleaning ability; iii) resistance to paints/graffiti [15,16]. Also it has been observed how hydrophobicity can be relevant to icephobicity [17,18]. These properties have not deeply investigated.

Standard procedures for protecting cement structures are mainly based on impregnation and coating methods, involving, therefore, only the modification of exterior layers and leaving a hydrophilic bulk [16]. Specifically, silane or siloxane are mostly used for these applications [19]. Recently, the addition of polymeric fibers to the paste mixture, combined to the use of a hydrophobic coating, has been reported to reduce water penetration and to turn to hydrophobic or over-hydrophobic this building material [17,20].

In this work, the effect of the TR grains addition to cement mortars has been investigated, with specific reference to wetting properties and, more specifically, to contact angle and absorption of water drops. Tyre rubber was added to the mixtures formulation as partial and/or total replacement of the conventional aggregate (sand). Aiming at affordable applications of this process (addition of TR) we have tailored an addition to the cement paste without any use of additive/chemical to improve adhesion. Since the material is modified in its whole mass, and no coating is present on the surface, both the side surface and the inner (fracture) surface of the mortars/specimens have been investigated. Wetting properties have been characterised and correlated to the micro-scale structure (SEM) and the porosity of the specimens. Moreover, flexural and compressive strengths of the composites have been measured.

2. Materials and methods

2.1. Mortar specimens preparation

CEM II A-LL 42.5 R, a limestone Portland cement [21] provided by Buzzi Unicem S.P.A. was used for the preparation of the cement composites. The main constituents are: 80–94% clinker, 6–20% limestone LL (<0.2% organic carbon), gypsum (0–5%), and minor additional constituents; it shows high early resistance (Rc (2 days)) > 25.0 MPa, Rc (28 days)) > 47.0 MPa) and Blaine specific surface area ranging 3100–4400 cm^2/g. Natural siliceous sand was provided by Societè Nouvelle du Littoral, Leucate, France with grains in the 0.08–2 mm size range [21,23].

Mortar specimens were overall prepared using this type of cement, sand, tap water (water/cement ratio kept constant at 0.5) and tyre rubber grains with particle size in the 0–2 mm range. The samples were molded in the form of prisms (40 × 40 × 160 mm) and 28 days water cured after demolding.

Tyre rubber was added to the mortars formulation as partial and/or total replacement of the conventional aggregate (sand). Tables 1 and 2 report the aggregate and mortars composition. Sand replacement was made on volume basis rather than on weight basis due to the low specific weight of the lightweight materials under investigation. In order to tailor TR added mortars without the addition of chemicals to improve adhesion, we previously evaluated the maximum TR volume which could be incorporated into the mixture to achieve a proper workability. Such a volume (500 cm^3) was set as constant total volume of the aggregate. A reference, named Sand, prepared by using 500 cm^3 of 0.5–2 mm sand, has been compared to the TR specimens. Total sand replacement was carried out with 100% TR grains in the size range <0.5 mm (TR-small), 100% TR grains in the 0.5–2 mm size range (TR-large) and the last one with 50% TR grains <0.5 mm and 50% in the range 0.5–2 mm (TR-mixed). Sand-TR sample was prepared by replacing 50% of the sand volume with TR grains in the size range <0.5 mm. A further conventional sand-based (normalized) mortar was prepared as control [22] and indicated as Normal.

2.2. SEM/EDX analysis and porosimetric measurements

Cement-based composites were characterized by scanning electron microscope (SEM) and energy dispersive X-ray (EDX) analysis. Specifically, in the case of SEM and EDX analysis, used to have magnified images and the elemental composition of the samples, an electron microscope FESEM-EDX Carl Zeiss Sigma 300 VP (Carl Zeiss Microscopy GmbH, Jena, Germany) was used. The samples were fixed on aluminum stubs and then sputtered with gold with a Sputter Quorum Q150 (Quorum Technologies Ltd, East Sussex, UK).

Measurements of porosity % (parameter dependent on the total volume of the pores) were carried-out by Ultrapyc 1200e Automatic Gas Pycnometer (Quantachrome Instruments, Boynton Beach, FL, US). The apparatus utilises helium as inert gas which penetrates the finest pores of the material thus overcoming the

Table 1
Aggregates composition of the mortars.

Sample	Type of aggregate	
Normal	Normalized sand	
Sand	Sieved sand (0.5–2 mm) 100%	
TR-small	Rubber Tyre (0–0.5 mm) 100%	
TR-large	Rubber Tyre (0.5–2 mm) 100%	
TR-mixed	TR (<0.5 mm) 50%	TR (0.5–2 mm) 50%
Sand-TR	Sieved sand (0.5–2 mm) 50%	TR (<0.5 mm) 50%