



Use of tire rubber particles as addition to cement paste

N. Segre, I. Joekes*

Instituto de Química, Universidade Estadual de Campinas - UNICAMP, Caixa Postal 6154, CEP 13083-970, Campinas, SP, Brazil

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Abstract

The surface modification of powdered tire rubber to increase its adhesion to cement paste was studied. The particles were surface-treated with NaOH saturated aqueous solutions for 20 min. Scanning electron microscopy (S.E.M.), water absorption, density, flexural strength, compressive strength, abrasion resistance, modulus of elasticity and fracture energy measurements were performed using test specimens (water/cement ratio = 0.36) containing 10% of as-received or 10% of NaOH-treated rubber. The results of fracture energy and flexural and compressive strength show that the addition of rubber particles improves the toughness and reduces the porosity of the specimens. Mass loss by abrasion ($-1.2 \pm 0.4\%$ for control specimens; $-1.7 \pm 0.4\%$, using treated rubber and $-4.0 \pm 1.2\%$ using as-received rubber, after 600 cycles) and electron microscopic examination show that the NaOH surface treatment enhances the rubber–matrix adhesion. The use of thus treated tire rubber particles, as addition, instead of a coarse aggregate, in cement-based materials is promising for applications such as driveways or in road construction. © 2000 Elsevier Science Ltd. All rights reserved.

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1. Introduction

The growing amount of waste rubber, produced from tires, has resulted in an environmental problem. According to the MEAM company (Medições Ambientais Consultoria, Belo Horizonte/MG, Brazil), specialized in environmental monitoring, about 32 million tires are produced annually in Brazil alone and it is estimated that 10 to 15 million units are discarded each year. This is not only a cause for environmental concern but also represents a waste of useful resources.

Since waste rubber is not easily biodegradable even after a long period of landfill treatment, material and energy recovery are alternatives to disposal [1–6]. On the other hand, a wide variety of waste materials have been suggested as additives to cement-based materials [7–11].

The literature [12–20] about the use of tire rubber particles in cement-based materials focuses on the use of tire rubber as an aggregate in concrete and evaluates only the mechanical properties. Most of the research has been

conducted with particles ranging from 2 to 16 mm. The overall results showed a remarkable decrease in the mechanical properties of concrete after addition of tire rubber particles as an aggregate. The use of coarse rubber particles affects the properties more negatively than do fine particles [13,14,17]. Some authors suggested that the loss in strength might be minimized by prior surface treatment of the tire rubber particles [12,19,20].

In this work, the surface of powdered tire rubber was modified to increase its adhesion to cement paste. Low cost procedures and reagents were used in the surface treatment to minimize the final cost of the material. Among the surface treatments tested to enhance the hydrophilicity of the rubber surface, a sodium hydroxide (NaOH) solution produced the best results. Initial results showed that the higher NaOH concentration, the better the adhesion. It is assumed that the NaOH hydrolyzes the acidic and/or carboxyl groups present on the tire rubber surface [21–24].

2. Methods

The following materials were used in this work: Ordinary Portland cement, Type I, supplied by Companhia de Ci-

* Corresponding author. Tel.: +55-19-788-3094; fax: +55-19-788-3094.

E-mail address: ines@iqm.unicamp.br (I. Joekes).

mento Portland Itaú (Itaú de Minas/MG, Brazil); slag-modified Portland cement, Type I(SM), trade name Tupi (Cimento Tupi S/A, Juiz de Fora/MG, Brazil); powdered tire rubber, 35 mesh maximum size ($500\text{ }\mu\text{m}$), supplied by Borcol Indústria de Borracha (Sorocaba/SP, Brazil); commercial sodium silicate type H300 NDL, from Gessy Lever do Brasil (São Paulo/SP, Brazil); analytical grade sodium hydroxide (NaOH).

The tire rubber particles were surface-treated with saturated NaOH aqueous solutions for 20 min, at room temperature, with stirring. The mixture was filtered and the rubber was washed with water and dried at ambient temperature.

Cement paste test specimens were prepared (water/cement ratio = 0.36) with the addition of 10% by mass of as-received or 10% by mass of NaOH-treated rubber. When sodium silicate was added, the water was substituted by a 10% volume aqueous solution of this reagent.

For water absorption by capillarity, flexural strength, modulus of elasticity, scanning electron microscopy (S.E.M.) and fracture energy experiments the specimens were prepared with Type I cement and cured for 28 days at 50°C and 100% relative humidity. For compressive strength experiments, the specimens were prepared with Type I(SM) cement and cured for 28 days at 20°C and 100% relative humidity. For abrasion resistance, the specimens were prepared with Type I(SM) cement and cured for 28 days in a controlled chamber at 20°C and 60% relative humidity.

Water absorption by capillarity was followed gravimetrically. Prismatic specimens measuring $50 \times 29 \times 9\text{ mm}$ were dried at 50°C in an oven until constant mass and placed on a support grating in a dish supplied with a water leveler at room temperature. The water level was sufficient to wet only the lower surface of the test specimens. The lower parts of the sides of the specimens adjoining the inflow face were sealed with an adhesive to prevent absorption into the surface pores. Mass data of each specimen was

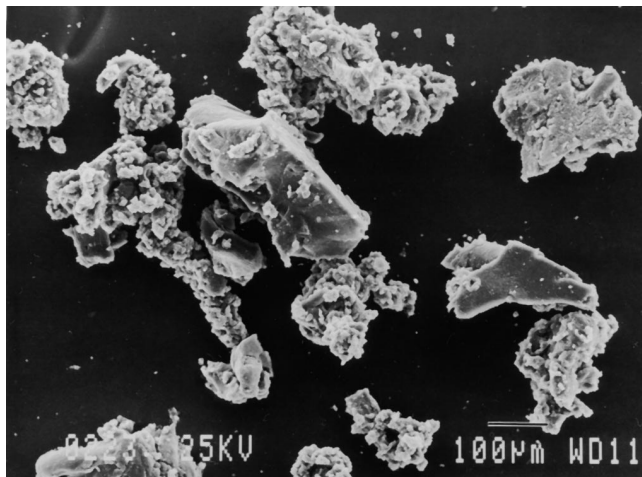


Fig. 1. SEI image of as-received tire rubber particles.

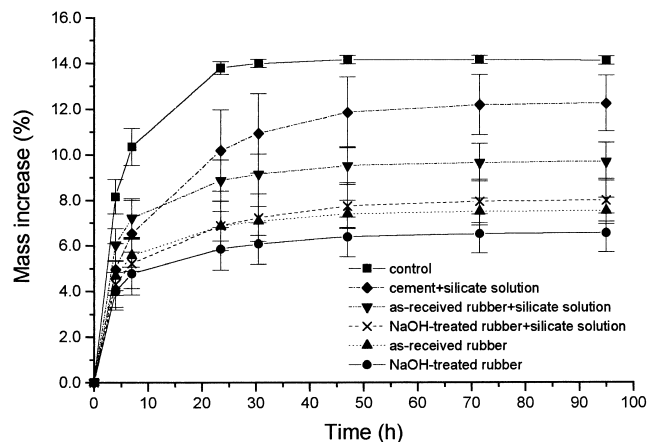


Fig. 2. Water absorption by capillarity, at room temperature, as a function of time for cement test specimens with 10% by mass of tire rubber. Control is cement paste. Results of triplicate specimens. Type I cement. w/c = 0.36.

obtained after several periods of time, until constant mass. Triplicate specimens were used.

For flexural strength, fracture energy and modulus of elasticity measurements, prismatic test specimens measuring $49 \times 10 \times 9\text{ mm}$ were used. A three-point bend test was performed. These measurements were performed in a MTS model 810 testing machine (MTS Systems, Eden Prairie/MN, USA). A loading rate of 10 mm/min was used in the flexural strength and modulus of elasticity experiments, performed with a set of four specimens. In fracture energy experiments, the loading rate was 0.2 mm/min for specimens with rubber and 0.01 mm/min for specimens without rubber; a set of seven specimens was used in each of these measurements.

Compressive strength measurements were performed according to the Brazilian Standard ABNT MB-1 [25], using cylindrical specimens measuring $100 \times 50\text{ mm}$, in a testing machine from Kratos Equipamentos Industriais

Table 1

Flexural strength, modulus of elasticity and fracture energy of cement test specimens with 10% by mass of tire rubber. Control is cement paste. Type I cement, w/c = 0.36. Results of quadruplicate specimens for flexural strength and modulus of elasticity. Results of a set of seven specimens for fracture energy

Specimen	Flexural strength (MPa)	Modulus of elasticity (GPa)	Fracture energy (J m^{-2})
Control	5.6 ± 3.1	5.9 ± 1.9	6.3 ± 0.9
With NaOH-treated rubber	9.9 ± 1.2	6.2 ± 0.3	22.0 ± 1.4
With as-received rubber	10.9 ± 2.0	7.3 ± 0.5	25.9 ± 3.5
With NaOH-treated rubber + silicate solution	4.6 ± 0.5	3.9 ± 0.7	25.4 ± 2.6
With as-received rubber + silicate solution	5.9 ± 1.1	5.2 ± 1.2	25.7 ± 3.1
Cement + silicate solution	7.2 ± 1.2	6.4 ± 0.8	

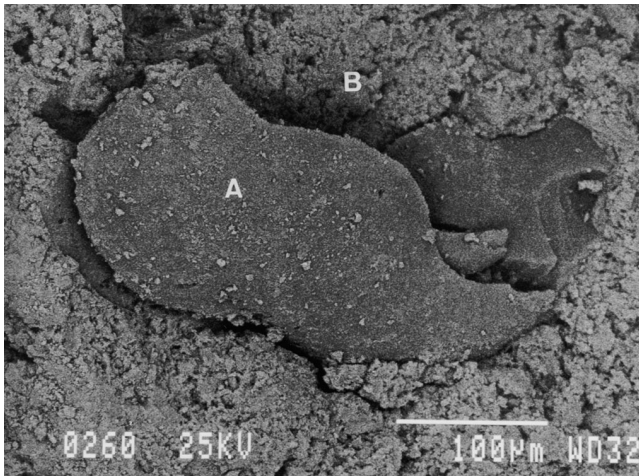


Fig. 3. S.E.M. image of fracture surface of cement test specimen with 10% by mass of as-received tire rubber. BEI image. (A) Rubber particle; (B) Cement paste.

(Taboão da Serra/SP, Brazil). The specimens were capped with quick-cure, high-hardness polyester resin. The loading rate was 0.25 ± 0.05 mPa/s. Triplicate specimens were used.

S.E.M. was used to characterize the rubber particles and to observe the interface between the rubber and the cement matrix. Fractures were obtained by compression from 20-mm fragments of the specimens, after immersion in liquid nitrogen for 10–15 min. Samples were sputtered with gold (~ 4 nm) in an Edwards S 150B Sputter. The S.E.M. micrographs were obtained in a Jeol JSM 840A microscope (Jeol, Akishima, Japan).

For the abrasion resistance experiments, a layer of cement paste with rubber was spread over an asbestos–cement support. After curing, the support was cut into specimens measuring $100 \times 100 \times 3$ mm. The wearing was followed gravimetrically after each 100-abrasion cycle,

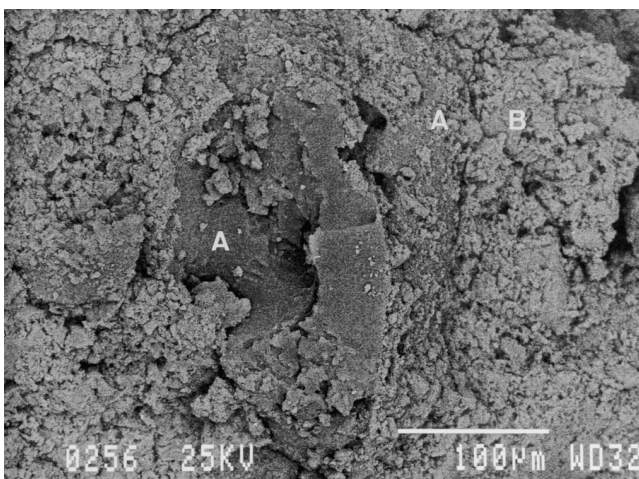


Fig. 5. S.E.M. image of fracture surface of cement test specimen with 10% by mass of NaOH-treated tire rubber. BEI image. (A) Rubber particle; (B) Cement paste.

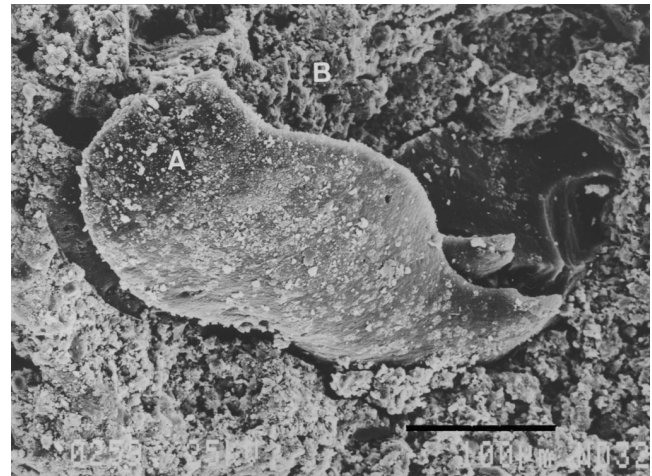


Fig. 4. S.E.M. image of fracture surface of cement test specimen with 10% by mass of as-received tire rubber. SEI image. (A) Rubber particle; (B) Cement paste. Bar: 100 μm.

up to 1000 cycles; a Taber abrader model 5130 (Taber Industries, North Tonawanda, NY, USA) was used. Duplicate specimens were tested.

3. Results and discussion

The rubber particles were observed by S.E.M. as shown in Fig. 1. The micrograph shows that the particles have a rough surface, with irregular shape and different sizes. No differences were observed in the particles after the NaOH treatment.

The amount of water absorbed is related to the porosity of the test specimens and gives a picture of the internal microstructure. As expected, Fig. 2 shows that the addition of rubber particles lowers the amount of water absorbed, since the rubber particles do not absorb water. Neverthe-

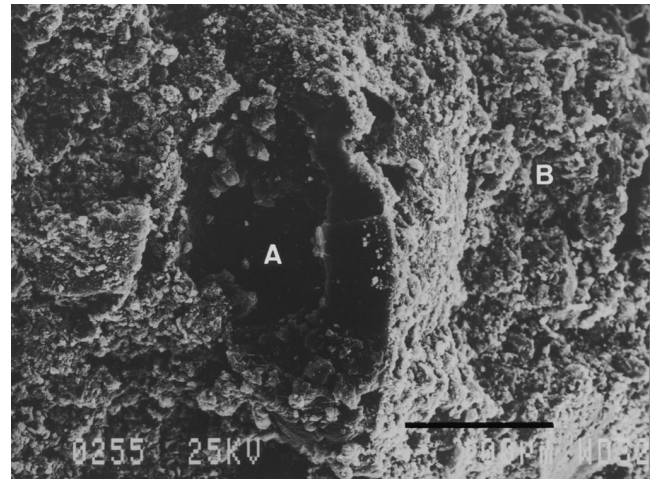


Fig. 6. S.E.M. image of fracture surface of cement test specimen with 10% by mass of NaOH-treated tire rubber. SEI image. (A) Rubber particle; (B) Cement paste. Bar: 100 μm.

less, the specimens containing NaOH-treated rubber absorb less water, indicating the better adhesion of these particles to the matrix.

Table 1 shows the results of flexural strength, modulus of elasticity and fracture energy. For flexural strength, the specimens with NaOH-treated rubber and with as-received rubber, without silicate solution, showed higher values than the control specimens. The inclusion of tire rubber increased the fracture energy by a factor of four, indicating that specimens containing rubber exhibit higher toughness, as compared to the control. No differences were observed for the modulus of elasticity. The density of all specimens decreased with the inclusion of the rubber particles, which can be explained by the difference of the individual densities (cement: $3.21 \pm 0.04 \text{ g cm}^{-3}$ and tire rubber particles: $1.152 \pm 0.001 \text{ g cm}^{-3}$).

The rubber–cement matrix interface was observed by S.E.M. The micrographs were obtained using two different detectors: one for backscattered electrons (BEI), which are capable of distinguishing the cement (inorganic material) from the rubber (organic material) by electron contrast differences, and one for secondary electrons (SEI), which show better surface detail. Detailed investigation on at least 15 specimens was performed. Micrographs of a typical (liquid nitrogen) fracture surface of a cement test specimen with 10% by mass of as-received rubber are shown in Figs. 3 and 4. The fracture surface shows a bulk region and the rubber particle seems to have been pulled-out. Discontinuity is observed in the rubber–matrix interface indicating that as-received rubber adhesion to cement paste is poor. On the other hand, an adhesive joint is observed between NaOH-treated rubber particles and the matrix, as shown in Figs. 5 and 6. Although the thermal expansion coefficient of both materials is quite different, the rubber particle shown in this micrograph did not separate from the cement matrix after contact with liquid nitrogen (195.8°C), indicating strong adhesion.

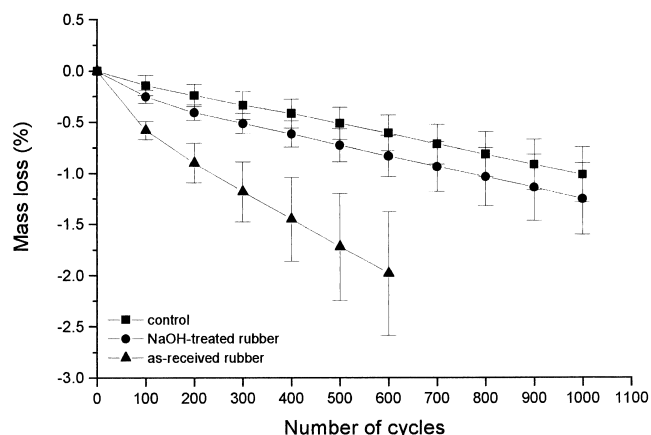


Fig. 7. Mass loss as a function of number of abrasive cycles, for cement test specimens with 10% by mass of tire rubber. Control is cement paste. Results of duplicate specimens. Type I(SM) cement. $w/c = 0.36$.

Table 2

Compressive strengths for cement test specimens with 10% by mass of tire rubber. Control is cement paste. Results of triplicate specimens. Type I(SM) cement. $w/c = 0.36$

Specimen	Compressive strength (MPa)
Control	43.1 ± 3.2
With as-received rubber	29.4 ± 3.1
With NaOH-treated rubber	28.1 ± 3.5

Since the NaOH-treated rubber showed good adhesion to the matrix, specimens were rubbed with an emery wheel and observed by S.E.M. For specimens containing NaOH-treated rubber, the particles were not pulled out from the matrix, as observed for the as-received particles.

Abrasion resistance experiments were performed with test specimens containing as-received or NaOH-treated rubber. Fig. 7 shows the mass loss as a function of number of abrasive cycles. The mass loss of specimens containing NaOH-treated rubber was significantly lower than that of the specimens containing as-received rubber. These results are fundamental to show the increased adhesion obtained by treatment of the rubber particles, since the pull-out force expresses the magnitude of the adhesion strength between the rubber and the cement matrix.

Finally, the effect of tire rubber as addition to cement paste on compressive strength was measured. Table 2 shows the results. As expected, the addition of rubber particles reduces the compressive strength of the specimens. However, a reduction of only 33% in compressive strength is observed, a value significantly lower than the 60% expected from Ref. [12].

4. Conclusions

The NaOH treatment enhances the adhesion of tire rubber particles to cement paste as observed in the MEV micrographs, abrasion resistance and water absorption experiments. Mechanical properties such as flexural strength and fracture energy were improved with the use of tire rubber particles as addition instead of aggregate. The decrease in the compressive strength was significantly lower than that described in the literature.

The overall results in this work show that the use of NaOH-treated tire rubber particles, as an addition to cement-based materials, may be promising to obtain a material suitable for engineering purposes where high strength is not necessary such as in driveways or in road constructions.

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