

EFFECT OF THE ADDITION OF SILICON CARBIDE PARTICLES ON THE MECHANICAL RESPONSE OF CONVENTIONAL PORTLAND CEMENT PAVERS

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Summary:

The mechanical behavior of conventional Portland cement paver mixes with and without silicon carbide (SiC) particle reinforcement was studied. The SiC used in this work was obtained from sequential thermal processes of calcination of wood residues and infiltration of silicon metal in carbon powder. For a first batch of paver mixes, controlled amounts of SiC particles were added to Portland cement paver mixes, the amounts added were 0, 2, 4, 6, 6, 8 and 10 % by volume, which replaced equivalent volumes of fine sand. In a second batch of paver mixes, volumes of SiC particles partially replaced equivalent amounts of Portland cement, keeping constant the volume of fine sand added to the mixes. From the study, it was confirmed that (i) the average maximum compressive strength increased progressively with increasing amount of SiC added to the mixes, changing from 28 to 34 MPa when 0 and 10% by volume of SiC particles were added to replace fine sand, respectively; and with a curing time of 14 days and (ii) the average maximum mechanical resistance to compression decreased progressively when increasing the amount of SiC added to the mixtures, changing from 48 to 35 MPa when 0 and 10% by volume of SiC particles were added to replace Portland cement, respectively; and with a curing time of 28 days. On the other hand, the microstructural characteristics of the paver mixes studied were evaluated, in all of them the presence of two phases was evidenced, a continuous and interconnected one of Portland cement that enveloped another one of sand and silicon carbide particles. The stiffness of the materials studied was also evaluated, finding (i) a slight increase in stiffness in those materials with a higher concentration of silicon carbide particles added (when SiC partially replaced fine sand) and a slight reduction in stiffness in those materials with a higher concentration of silicon carbide particles added (when SiC partially replaced Portland cement).

Palabras clave: silicon carbide, mechanical, Portland cement, pavers

1. INTRODUCTION

Currently, the manufacture of concrete, which accounts for a significant share in the global consumption of building materials, poses considerable environmental challenges due to the high CO2 emissions associated with the production of cement, its main component [1,2]. In response to this problem, the aggregation of alternative materials that can strengthen the properties of concrete, thus reducing the dependence on conventional cement in its composition, is currently being actively pursued. Moreover, advances in silicon carbide (SiC) ceramics offer new perspectives in various industrial applications [3,4]. In order to address environmental concerns, the potential of reusing natural resources such as wood to develop biomorphic SiC is being explored in recent years [5,6]. Wood, with its hierarchical structure composed of cellulose, hemicellulose and lignin, has proven to be a promising material for the fabrication of highly porous SiC by pyrolysis and silicon infiltration techniques. Methods such as molten silicon and silicon vapor impregnation have been successfully employed to transform wood-derived charcoal into functional SiC [7,8]. In industry, silicon carbide stands out as an exceptionally robust and durable ceramic material, offering high mechanical strength and toughness. [9,10] In both its crystalline and amorphous forms, SiC exhibits unique semiconducting properties that make it ideal for high temperature environments, high frequencies, intense fields and high power applications [11,12]. However, to date, there is a notable lack of specific studies on the use of SiC residues in the production of cementitiousbased materials. Therefore, this work focuses on investigating the influence of silicon carbide as a reinforcing



agent in Portland cement paver mixes [13,14]. The SiC used was obtained through wood waste calcination processes followed by infiltration of silicon in coal dust. Through the incorporation of different controlled proportions of SiC, with the purpose of replacing equivalent volumes of fine sand in Portland cement formulations, we will proceed to analyze how this addition positively influences the mechanical and microstructural properties of the pavers [15,16]. This study indicates that the introduction of SiC has the potential to significantly improve the structural characteristics of concrete, positioning it as a promising reinforcement material for various applications. In addition to potentially optimizing production costs, SiC represents an environmentally friendly alternative due to its renewable origin and environmentally beneficial properties [17,18].

2. MATERIALS AND METHODS

2.1. Raw material:

To obtain the reinforcing agent (SiC) we started by collecting wood residues from local sawmills in the city of Arequipa (Peru), residues of some specific type of wood were not considered, instead a mixture of various types of wood was chosen, residues in the form of fine powder were preferred. All material other than wood residue was manually separated, then the residues were washed with water to free them from dust and then went through a drying process in an oven at 80 °C for 24 hours.

Other starting materials used in this research were (i) Edgetech brand silica powder (99.99% purity), fine sand, Portland cement (99.99% purity), and (ii) silicon powder (99.99% purity).

2.2. Manufacture of the reinforcing agent

The reinforcing agent was SiC and was obtained from wood sawdust powder, previously washed and dried. The manufacture of the SiC powder followed several stages detailed below:

- (i) Cold pressing of sawdust powder to obtain briquettes.
- (ii) Calcination of sawdust briquettes at a temperature of 800 °C for 1 hour, at heating and cooling rates of 10°C/min and in argon atmosphere, to obtain charcoal briquettes.
- (iii) Grinding of charcoal briquettes to powder.
- (iv) Mixing of coal powder and silicon metal powder, with an excess of 10% by volume of coal, with respect to the stoichiometric quantity for the formation of the SiC molecule.
- (v) Thermodiffusion of silicon in carbon at temperatures of 1500 °C, for 1 hour, in a vacuum of 7 Pa and with heating and cooling rates of 10 °C/min.
- (vi) Removal of unreacted carbon by calcination in air at 500 °C for 1 hour at heating and cooling rates of 10 °C/min.
- (vii) Grinding of silicon carbide powder and sieving through ASTM 140 mesh.

2.3. Production of pavers

Mixtures of pavers reinforced with SiC particles and without reinforcement were manufactured in two consecutive batches. A first batch followed the volumetric proportions in Table 1, where the volume of Portland cement remained constant (25 vol.%) for all mixes and volume fractions of SiC particles partially replaced, and in equivalent volumes, fine sand particles. For a second batch of paver mixes, the volume proportions in Table 2 were followed, where the volume of fine sand remained constant (75 vol.%) for all mixes and volume fractions of SiC particles partially replaced, and in equivalent volumes, Portland cement particles. As can be seen, for the first batch the cement: sand+SiC ratio was 1:3 and for the second batch it was variable from 1:3 to 1:5.67. The water: cement ratio used for all mixes was 0.6.

Table 1. Mix matrix for the first batch of reinforced and unreinforced pavers.

mixture	fine sand	Portland cement	SiC particles
	(Vol.%)	(Vol.%)	(Vol.%)
M-1	75	25	0
M-2	73	25	2
M-3	71	25	4
M-4	69	25	6
M-5	67	25	8
M-6	65	25	10



After the evaluation of the effect on the mechanical behavior of the controlled addition of SiC particles replacing identical volumes of fine sand in mixtures of fine sand and Portland cement (first batch, Table 1), a new mix matrix was designed (second batch) in which the volume of Portland cement added to the mixes is gradually reduced from 25% to 15% by volume. Table 2 shows the mix matrix of the second batch of reinforced and non-reinforced paving block mixes, with gradual reduction of Portland cement.

Table 2. Mixing matrix for the second batch of reinforced and unreinforced pavers.

mixture	fine sand (Vol.%)	Portland cement (Vol.%)	SiC particles (Vol.%)
M-1	75	25	0
M-2	75	23	2
M-3	75	21	4
M-4	75	19	6
M-5	75	17	8
M-6	75	15	10

In all cases, the manufacturing of paver mixes followed the following steps:

- (i) Dry mixing of the solid components for 5 minutes.
- (ii) Addition of water, following the water: cement ratio of 0.6 used.
- (iii) Mixing of liquid component (water) and solids for 5 minutes.
- (iv) Pressing of cylindrical samples of 10 mm diameter and 20 mm height.
- (v) Curing in water of manufactured cylinders: 14 days in water for the first batch and 28 days in water for the second batch.
- (vi) Uniaxial mechanical compression tests after the curing process.

2.4. Physical, structural, microstructural and mechanical characterization

The physical characterization of the fabricated materials was carried out by means of tests to determine the apparent and real density, the apparent density was determined by simple measurement of masses and dimensions and the real density was carried out by helium pycnometry using a Micromeritics equipment, model AccuPyc II 1345. On the other hand, the microstructural characterization consisted of observations of polished surfaces of the samples studied, this characterization was carried out using a Zeiss (Germany) model Primotech tl/rl pol polarization binocular optical microscope. The mechanical characterization was carried out under uniaxial compression conditions at room temperature and at a constant compression speed of 0.5 mm/minute. A MICROTEST (Spain) model EM1 universal testing machine was used for the mechanical studies.

3. RESULTS AND ANALYSIS

Figure 1 shows stress vs. strain curves, with three repetitions per type of material studied. A gradual increase in the average maximum mechanical strength was observed when more silicon carbide particles were added to the paver composition. The values found were 27.87 MPa for conventional pavers without reinforcement (M1) and 33.56 MPa for pavers with 10% by volume of silicon carbide particles (M6).

From the mechanical results found, it was possible to deduce the better mechanical characteristics of the SiC particles, with respect to conventional fine aggregate particles (fine sand), this deduction is supported by the fact that as the SiC particles replaced the fine sand particles, the overall strength of the paver improved. On the other hand, considering that the mechanism of mechanical response in compression of this type of binder-aggregate materials (mortars, concretes, paving stones,) consists mainly in the transfer of mechanical stress from the continuous binder phase (Portland cement) to the discontinuous, dispersed and more resistant aggregate phase (fine sand), through the binder-aggregate interface, it can be suggested that the increase in the overall strength of the paver mixes was due to two factors (i) a better (higher) bond between binder and SiC particles, and therefore, better mechanical stress transfer capacity from the binder to the aggregate and (ii) higher mechanical strength and stiffness of SiC particles, with respect to conventional fine aggregate.

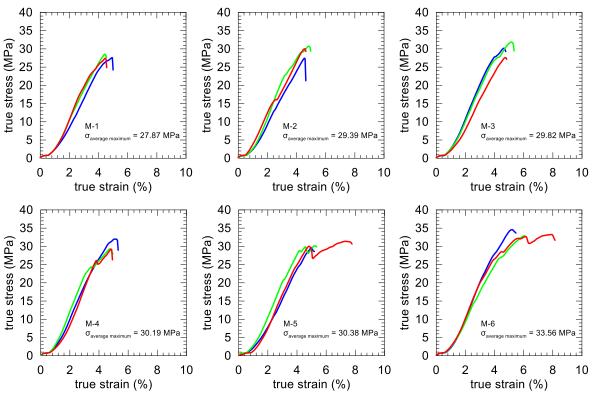


Figure 1. Stress vs. strain curves for conventional (M1) and reinforced (M2 to M6) pavers with partial replacement of fine sand particles by SiC and 14 days of curing (Table 1).

The Young's modulus (stiffness) of the materials studied was also determined from the measurement of the slope in the elastic range for all the stress vs. strain curves obtained (Figure 1). For the first batch of manufactured paver mixes (Figure 2), where SiC particles partially replaced fine sand particles, a gradual increase of the average stiffness was found when the amount of reinforcement increased (Figure 2). The range of Young's modulus values found was 8.07 and 9.42 GPa, for conventional (M1) and reinforced (M6) paver mixes, respectively.

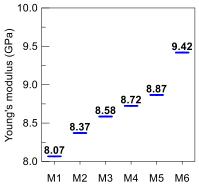


Figure 2. Young's modulus (stiffness) as a function of the type of pavers studied, conventional (M1) and reinforced (M2 to M6) with partial replacement of fine sand particles by SiC and 14 days of curing (Table 1).

Figures 3 and 4 show the mechanical results for the second batch of paver mixes studied. As mentioned above, in the second batch studied, we sought to evaluate the effect of gradually reducing Portland cement volumes by adding equivalent volumes of SiC particles (Table 2). In this regard, Figure 3 presents stress vs. strain curves for conventional (M1) and reinforced (M2 to M6) paver mixes. It should be noted that, for the second batch of mixes, the mechanical strengths are in a higher range of values than for the first batch, because they were



evaluated mechanically at a longer curing time (28 days), compared to the 14 days of curing of the mixes of the first batch.

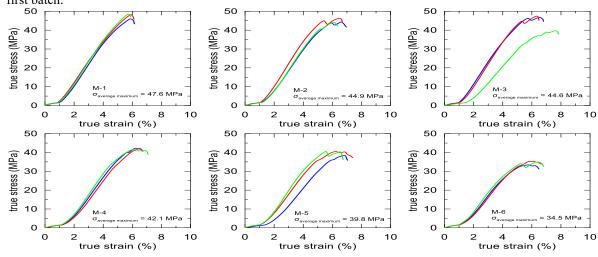


Figure 3. Stress vs. strain curves for conventional (M1) and reinforced (M2 to M6) pavers with partial replacement of Portland cement by SiC and 28 days of curing (Table 2).

Figure 3 shows a systematic reduction of the mechanical resistance in compression, from 47.6 MPa to 34.5 MPa, when the replacement of Portland cement by SiC particles was 0 and 10% by volume, respectively. It can be suggested that this result was mainly due to a reduction in the overall agglomeration capacity of the cement (due to its lower existence within the mix) which, however, was not dramatic since it was possible to maintain average mechanical strength values of up to 34.5 MPa (mix of 10% SiC + 15% cement + 75 % fine sand). This result suggests a saving of Portland cement for paver mixes that can be used in applications that are less demanding in terms of mechanical strength, such as pavers for pedestrian traffic (depending on the technical standards applied in each country).

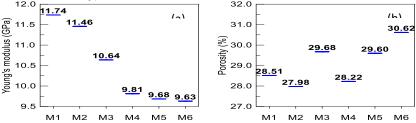


Figure 4. (a) Young's modulus (stiffness) as a function of the type of pavers studied, conventional (M1) and reinforced (M2 to M6) with partial replacement of Portland cement by SiC and 28 days of curing (Table 1) and (b) porosity of the paver mixes studied.

Figure 4(a) shows the Young's modulus values for paver mixes of the second batch studied in this work. In this figure we can appreciate the systematic reduction of the stiffness of the studied materials from 11.74 to 9.63 GPa for conventional (0% by volume of SiC added) and reinforced (10 % by volume of SiC added) paver mixes. Similar to the suggestion given for the mechanical behavior in uniaxial compression in Figure 3, in this case, the reduction in stiffness of the materials is attributed to the lower amount of Portland cement used in the reinforced paver mixes, which, however, does not lead to a dramatic reduction in stiffness. The average minimum value found was 9.63 GPa, which corresponds to a mixture of 10% SiC + 15% cement + 75% fine sand by volume. This mixture of reinforced pavers (M6 in Figure 4(a)) has a stiffness suitable for use in pavers for pedestrian traffic; however, its application will depend on the technical standards of each country. Figure 4(b) shows the relationship between the type of paver mix studied (Table 2) and the percent porosity found in the materials. The percent porosity values found were in the range of 28.5 and 30.6 % for mixes with 0 % and 10 % by volume added SiC, respectively. It was not possible to recognize a clear correlation, the



porosity percentage values did not follow an order and we could only suggest that, in general, the reinforced paver mixes have a slightly higher porosity compared to the conventional paver mix

Figure 5 shows micrographs obtained by optical microscopy applied on polished surfaces of samples of conventional (M1) and reinforced (M2 to M6) paver mixes. In general, for all the mixtures, two distinct phases were identified: on the one hand, a continuous Portland cement binder phase (uniform gray contrast) and, on the other hand, fine sand and SiC particles that were dispersed throughout the volume of the Portland cement binder phase. The fine sand and SiC particles were differentiated by their shape and size. The fine sand particles presented smaller sizes and their average shape was rounded, while the SiC particles were identified as larger and their shape was elongated and not very spherical. It was also possible to slightly identify the higher concentration of SiC particles in mixture M6 with respect to mixture M2, this result was expected and is in good agreement with the initial composition of the mixtures (Table 2).

M-3

M-6

M-1

M-7

M-6

Figure 5. Optical microscopy micrographs of samples of unreinforced (M1) and reinforced (M2 to M6) pavers from the second batch of paver mixes.

4. CONCLUSIONS

- Mixtures of conventional Portland cement pavers and pavers reinforced with silicon carbide particles were successfully fabricated and mechanically studied.
- A systematic increase in the average maximum strength was evidenced when more SiC particles were added to the paver composition (when SiC partially replaced fine sand). The values found were 27.87 MPa for conventional pavers and 33.56 MPa for pavers with 10% by volume of SiC particles.
- From the study, it was suggested that the increase in the overall strength of the paver mixes (when SiC partially replaced fine sand) was due to (i) better (higher) bonding between binder and SiC particles, and therefore, better mechanical stress transfer capacity from the binder to the aggregate and (ii) higher mechanical strength and stiffness of SiC particles, with respect to fine sand.
- When SiC particles partially replaced fine sand particles, a gradual increase in the average stiffness of the paver mixes was found with increasing SiC particle volume.
- A systematic reduction of the mechanical resistance in compression was evidenced, going from values of 47.6 MPa to 34.5 MPa, when the replacement of Portland cement by SiC particles was 0 and 10% in volume, respectively. From this part of the study, it was suggested that this behavior was due to the reduction in the overall agglomeration capacity of the cement (due to its lower existence in the mix).



- The systematic reduction of the stiffness of the materials studied was verified, going from values of 11.74 to 9.63 GPa for conventional (0% by volume of SiC added) and reinforced (10% by volume of SiC added) paver mixes (where SiC partially replaced Portland cement).
- It was not possible to recognize a clear correlation between the composition of the paver mixes and their degree of porosity, we could only suggest that, in general, the reinforced paver mixes have a slightly higher porosity compared to the conventional paver mix.
- In the morphological aspect, two well differentiated phases were found for all the mixtures, on the one hand, a continuous Portland cement binder phase and, on the other hand, fine sand and SiC particles that were dispersed within the volume of the binder phase.

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