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Evaluation of Engineered Cementitious Composites (ECC) With Rice Husk Ash Submitted to Different Milling Times

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Abstract

Engineered cementitious composites (ECC) are a special class of advanced cement composites, developed to produce more durable and with better performance materials. Thus, this work investigated the use of rice husk ash (RHA) as supplementary cementitious material. RHA was milled for 4 and 8 hours, to improve pozzolanic activity. Mechanical properties in terms of bending and tensile strength were evaluated. Resistance to reflective cracking and bond strength by slant shear were also assessed. The results showed that mixtures with RHA presented similar performance to the reference mixtures, presenting a well-defined strain hardening curve and expected multiple cracking behavior. Reflective cracking and bond strength tests indicated that RHA-ECC has suitable potential for pavement rehabilitation, since it bonds to the base concrete in a way that both materials act monolithically. RHA-ECC also presented ability to resist the strains generated at the tip of induced cracks, in order that micro-cracks did not cross the entire section of the material (for 50 mm layer). Also, no significant differences were noticed between the mixtures with RHA milled for 4 and 8 hours. Thus, it is recommended the use of RHA milled for 4 hours, since that it is used less energy during the milling process.

Keywords: Engineered Cementitious Composites; Rice Husk Ash; Pavement Rehabilitation.

1. Introduction

ECC is a term used to designate a high performance fiber reinforced cementitious composite (HPFRCC), composed by a blend of cement, supplementary cementitious material, fine sand, water and admixtures, with high deformation capacity and multiple crack behaviour. ECC is known for its high capacity to absorb energy after the elastic peak load, when subjected to tensile, leading to a strain hardening curve [1-8]. One of the proposed applications is for rehabilitation of rigid pavements, due to the potential capacity of avoiding reflective cracking, a serious problem usually encountered when using traditional unbound concrete overlays [9-11]. Other ECC characteristics, such as control of shrinkage cracking, high strength, durability, and the possibility of applying thin layers are also attractive. Another interesting fact of this material is that joints can be reduced or even suppressed, as suggested by [9].

ECC has been mainly evaluated with the use of polyvinyl alcohol (PVA) fibers [1,4,5,10,12], although some studies were based on the use of other fibers [3,7,13] such as polyethylene, polypropylene, polyester, acrylic, nylon and steel. Other authors have also considered the use of a blend of fibers, to improve impact resistance [2,6,14,15]. In this work, polypropylene fibers were chosen to reinforce the ECC, due to the lower cost and higher availability of this type of fiber, in the brazilian market, as well as due to its good results obtained in previous studies [16].

The improved strain capacity of ECC is only viable because of intense load transfer among fibers, which is controlled by the bond between the fibers and the matrix [3,5]. The high content of cement, the addition of supplementary cementitious material and the type of fiber are the factors affecting the fiber interaction to the matrix [3,13]. The use of supplementary cementitious materials has been widely studied in the past years, including mainly the use of fly ash [1-3,5-9,12,13,17,18], even though other studies have been carried out considering the use of other cement replacement materials, such as palm tree oil, silica fume and slag [17,19,20].

In Brazil, very promising research works have been developed using RHA as an addition or cement replacement material [21,22]. It has good pozzolanic activity, better chloride entrapment

capacity than local fly ash and good rheological behavior. It also fits well with the local economy, since it offers the possibility to add value and to give destination to a large volume of residue, generated by the rice industry. Many rice companies use the rice husk as fuel to energy and/or vapor generation. Usually, they correspond to small size companies, which do not have processes to reuse or discard correctly the ash generated by the combustion process [23].

The RHA presents in its composition silicone dioxide, commonly named as silica (SiO_2), as well as carbon (C), potassium oxide (K_2O), phosphoric oxide (P_2O_5), calcium oxide (CaO) and in smaller quantities, compounds of magnesium (Mg), iron (Fe) and sodium (Na) [24].

The combustion of rice husk leads to ashes with high amount of silica, with varying structural forms (amorphous and/or crystalline). The amount of silica and the structural form depends on, among other things, of the type of equipment used to burn the rice husk, the time interval at which the husk stays inside the furnace, the burning temperature and the cooling rate of the ashes [25].

To guarantee a controlled production of RHA, so that the whole process occurs at a specific temperature, the burning process is usually performed by fluidized bed systems [26,27]. The burning process carried out using these reactors, in general, presents advantages for the production of amorphous RHA when compared to the grill combustion system, due to the inexistence of temperature gradients or stagnated regions. However, due to the high costs associated to the use of processes based on fluidized bed systems, the majority of companies that use the rice husk as biomass to generate energy and/or vapour in Brazil adopt the grill combustion system, where burning does not occur homogeneously [28]. In this process, the burning occurs by the deposition of biomass into a grill system, where the drying, volatilization and combustion processes occur. However, although the installation costs are lower, the grill burning system presents some usage disadvantages, mainly related to the low efficiency, high emissions of atmospheric pollutants, and some common reactor problems, such as corrosion and deposition. These issues lead to the fact that the ashes generated from this system present high variation in quality, high carbon content and partially crystalline silica [28,29].

According to [30], this residual ash is classified as low reactive pozzolan. Hence, it is suggested that the material should be milled so the finer powder can achieve pozzolanic activity. The milling degree, in association with the burning conditions, are some of the factors that influence the RHA characteristics. The milling process has the potential to modify some of the physical properties of the RHA, such as the specific surface and fineness [31]. The milling process may be performed under dry or humid conditions, depending on the material. For RHA, the dry condition is normally used.

Aiming to reduce the environmental impact caused by the high cement content, and also to control the chemical bond between the fibers and the matrix, this research was developed to consider the use of non-processed RHA, as partial cement replacement, in proportion of 30% by volume. The results in terms of tensile and flexural behaviour and the performance in terms of resistance to reflective cracking and bond strength by slant shear are reported in the following sections.

2. Experimental Program

2.1. Mixture Proportions

Three ECCs mixtures were examined in this research, and their proportions are shown in Table 1. One mixture was considered as reference (REF), since no RHA was added. Mixtures RHA4 and RHA8 are similar to REF, except for the fact that they contain 30% by volume of non-processed RHA, milled for 4 and 8 hours, respectively, and used as partial replacement to the Portland cement. High early strength Portland cement was used in all mixtures.

Table 1 – ECCs mixtures and proportions

Mixture	Cement (kg/m³)	Rice husk ash (kg/m³)	Sand (kg/m³)	Water (kg/m³)	Fiber (kg/m³)	Superplasticizer (kg/m³)	w/c
REF	1009.16	-	757.53	348.55	17.90	7.05	0.34
RHA4	706.41	197.61	757.53	348.55	17.90	7.05	0.34
RHA8	706.41	204.43	757.53	348.55	17.90	7.03	0.34

Alkali-resistant polypropylene fiber with 10 mm of length, 12 μm of diameter, specific weight of 0.91 g/m³, tensile strength of 850 MPa, elastic modulus of 6 GPa and 21% of elongation at failure was used. The granulometric parameters of the fine sand are presented in Table 2, and they were obtained following the procedures of [32] and [33].

Table 2 – Fine sand granulometric parameters

Sieve (mm)	% retained	Cumulative % retained
4.8	0.40	0.40
2.4	0.30	0.70
1.2	0.40	1.10
0.6	0.40	1.40
0.3	3.30	4.70
0.15	53.90	58.60
<0.15	37.60	100
Total	100	-
Fineness modulus		0.69
Nominal maximum aggregate size (mm)		0.30
Specific mass (g/cm³)		2.52

Superplasticizer admixture polycarboxylic-ether based Tec-Flow® 8000 was applied in the mixtures. The RHA was donated from a rice cooperative located in the south region of Brazil. The non-processed ash was a residue of the parboiling process of rice grains, at which it was used as source to generate energy. The dark ash was obtained with no controlled burning temperature and non-uniform production process. The ash was milled in a ball mill containing spheres with diameter of 75 and 38 mm. Apart from the milling process carried out in laboratory, it is important to mention that no other treatment was performed in the ashes after burning. The chemical composition and physical properties of the RHA are presented in Table 3. As it can be seen, the pozzolanic activity index indicated good reactivity for both milling times, since the values obtained were above 75% [34].

Table 3 – RHA chemical and physical properties

Chemical composition (%)	RHA4	RHA8
SiO_2	63.26	65.83
K_2O	1.46	1.07
CaO	0.73	0.44
P_2O_5	0.52	0.33
Fe_2O_3	0.32	0.34
MnO	0.30	0.27
SO_3	0.18	0.05
Al_2O_3	0.13	0.06
MgO	0.12	0.08
CO_2	32.71	31.33
Specific mass (g/cm³)	2.04	2.06
Average grain size (μm)	7.29	3.59
Loss on ignition (%)	19.44	13.99
Pozzolanic activity index (%)	174.37	162.48

The materials were mixed in a 2 liter mechanical planetary mortar mixer. Initially, the dry materials (cement, sand and RHA) were mixed, until the visual aspect was considered uniform. Water and superplasticizer admixture were then added to the mixture. The paste obtained was sufficiently fluid to allow good fiber incorporation and dispersion. The fibers were then added and the material was mixed for 3 minutes. The resulting mixture was inserted inside of appropriated molds, and compacted using a shaking table.

2.2. Test Methods

As discussed before, some of the hardened specimens were used to evaluate flexural and tensile strength. Other specimens were specially created to assess resistance to reflective cracking and bond strength by slant shear, important characteristics for the planned used of the material in pavement rehabilitation. All specimens were conditioned in a room with controlled relative humidity (> 95%) and temperature (23ºC ± 2ºC)

Flexural tests were carried out using a four-point bending setup (Figure 1). Figure 1a indicates the configuration of the test applied, similar to the one used by [35], which was based largely on [36]. Already Figure 1b shows one of the 300 x 7 x 15 mm specimens during testing. Vertical and horizontal displacements were measured by two LVDTs (with maximum length of 10 mm), one placed vertically and the other below the specimen, longitudinally. The test was performed with a load speed of 1.2 mm/min. Three specimens for each mixture proportion were tested at 28 days. Stress-strain curves were registered for each specimen, to gather post-peak behavior data.

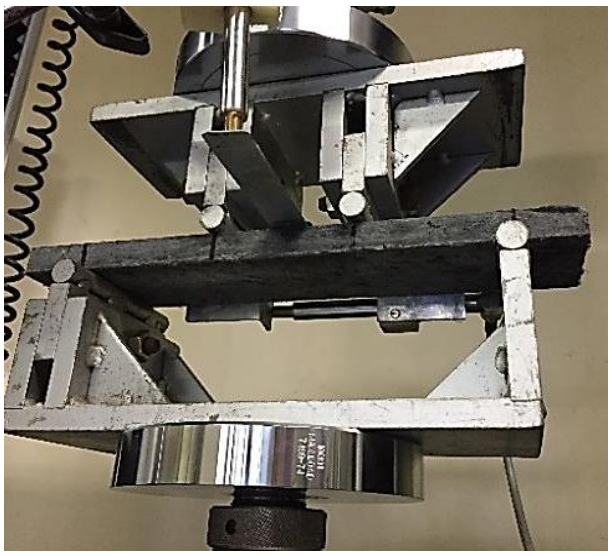


Figure 1 – Four point test - View of bending specimen during test

Tensile tests were based on the experimental setup used by [37], with a 0.30 mm/mim load speed. Figure 2 shows the tensile specimen. Its geometry is indicated in Figure 2a, while a specimen during testing can be seen in Figure 2b. Three specimens for each mixture proportion were tested at 28 days. Stress-strain curves were registered, to check the post-peak behaviour of the materials.

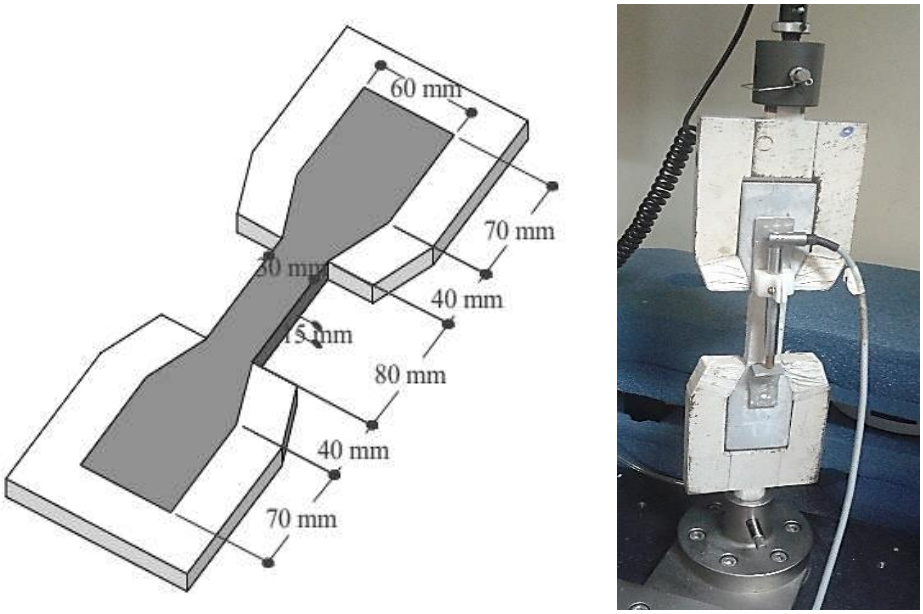


Figure 2 – Tensile specimen a) Geometry (attached to anchoring support) b) View during test

The resistance to reflective cracking was evaluated following the method of [38], based on a static bending test. The test was carried out to determine the potential performance of the material when used as overlay for rehabilitation of rigid pavements. The idea was to simulate a situation where the ECC layer is applied over an existing crack. Therefore, test specimens were created with two layers: a base layer of concrete made using a mixture typically employed in conventional concrete pavements, while the top layer was formed by ECC with different thicknesses.

The mixture proportion used for the base concrete was of 422 kg/m³ of high early strength cement, 712 kg/m³ of fine sand aggregate, 305 kg/m³ of medium sand aggregate, 958 kg/m³ of coarse basalt aggregate, of 2.80 kg/m³ of polycarboxylic-ether based admixture and a w/c ratio of 0.39. To simulate the crack, the base layer was created using two 177 x 75 x 50 mm concrete prisms

positioned 1 mm apart. After 14 days of curing, these two concrete segments were inserted inside of a mold and the ECC was placed over them, creating continuous top layers with three different thicknesses: 20, 30 and 50 mm.

After 28 days of ECC curing, the specimens were subjected to a flexural test, using a load speed of 1.20 mm/min. The test setup can be seen in Figure 3. The results were obtained in terms of stress x vertical deflection curves. Three specimens for each mixture proportion and each ECC thickness were tested. Two LVDTs (with maximum length of 5 mm) were used to measure the vertical displacements, one placed at each side of the specimen.

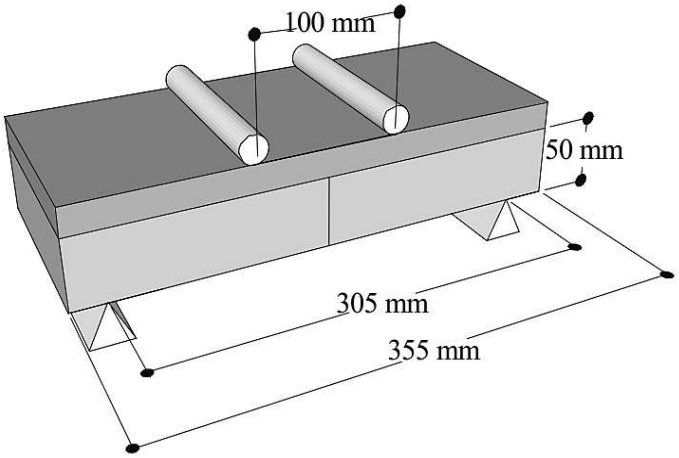


Figure 3 – Flexural test setup used to assess resistance to reflective cracking

The bond strength by slant shear test was performed to obtain an indication of the bond strength between an existing concrete pavement and the ECC overlay, and it was performed according to [39]. The test specimens were configured with 72 mm of diameter and 144 mm of height, and were composed by traditional concrete pavement and ECC. Initially a full specimen was molded with the same concrete used as the base for the resistance to reflective cracking test. For this, no superficial treatment aiming to create an adhesion bridge was applied between the materials. The concrete substrate was used considering the sawed face.

After 14 days of curing, the specimen was cut following a 30º angle, as shown in Figure 4. The cut specimens were placed back on the molds and an ECC layer was applied over them. After 28 days of ECC curing, the specimens were subjected to axial compressive load, with a speed of 0.45 MPa/s. The results were obtained in terms of maximum bond strength. Triplicate specimens for each mixture proportion were tested.

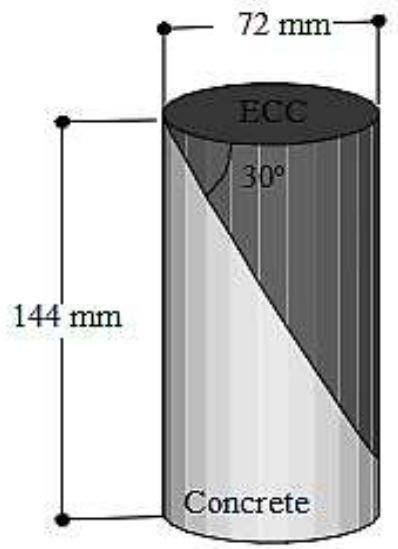
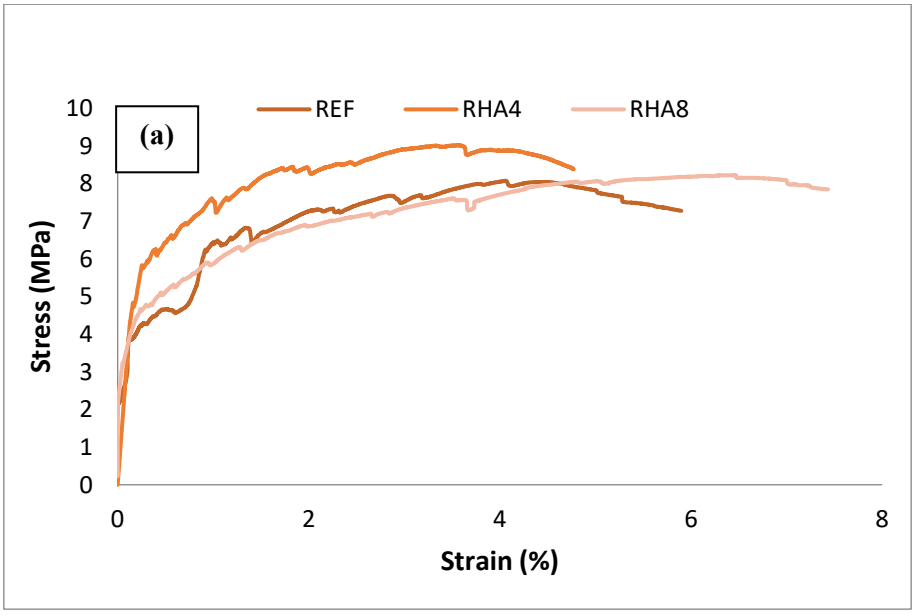


Figure 4 – Specimen used for bond strength by slant shear test

3. Results and Discussion

3.1. Bending and Tensile Tests

Figures 5 shows the stress-strain curves for the bending (Figure 5a) and tensile (Figure 5b) tests, for REF, RHA4 and RHA8 mixtures. Each curve represents an average from 3 samples. It is important to mention that x and y axle scales are different in both tests, since the levels of load and displacements were not comparable between both.



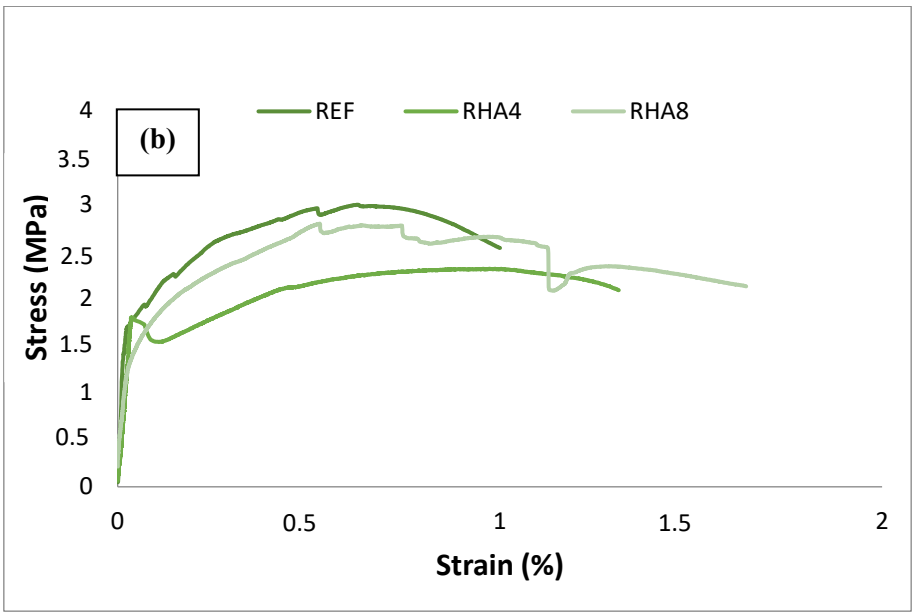


Figure 5 – Stress-strain curves for REF, RHA4 and RHA8 mixtures (a) Bending test (b) Tensile test

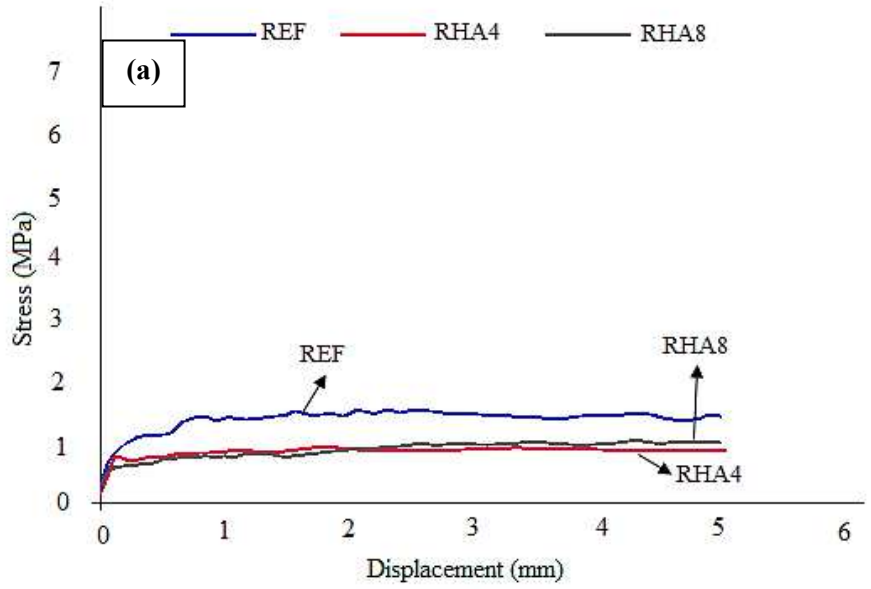
Regarding the bending test results, it can be observed that all mixtures presented similar behavior, in terms of maximum stress and load-bearing capacity, after the elastic peak, with a clear strain hardening characteristic. In tensile test, the mixture RHA8 performed slightly better than RHA4. All mixtures presented the multiple cracking behavior, characteristic of ECC (as seen in Figure 6). It is an indication that the inclusion of RHA resulted in a suitable matrix, with good interaction with the polypropylene fiber, and achieved a performance similar to the reference mixture.



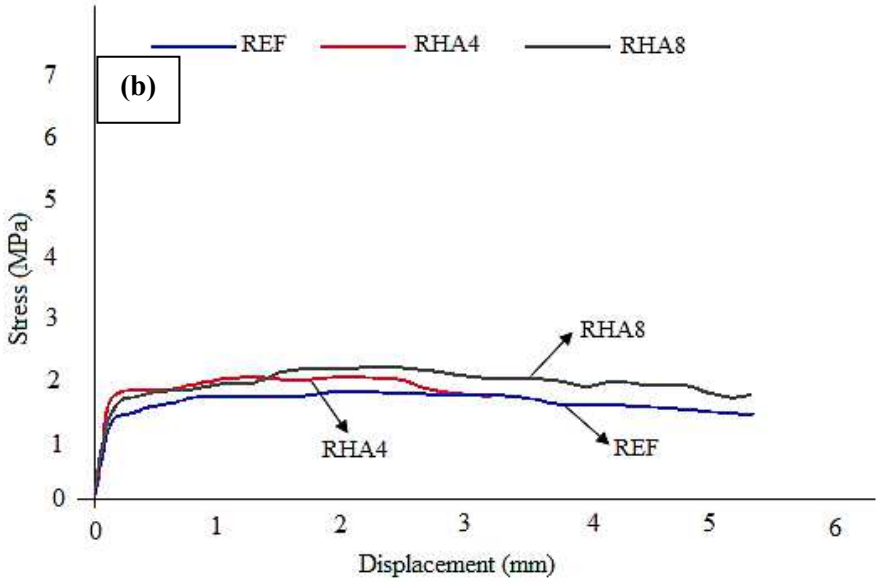
Figure 6 – Multiple cracking observed in the specimens after bending test

3.2. Resistance to Reflective Cracking

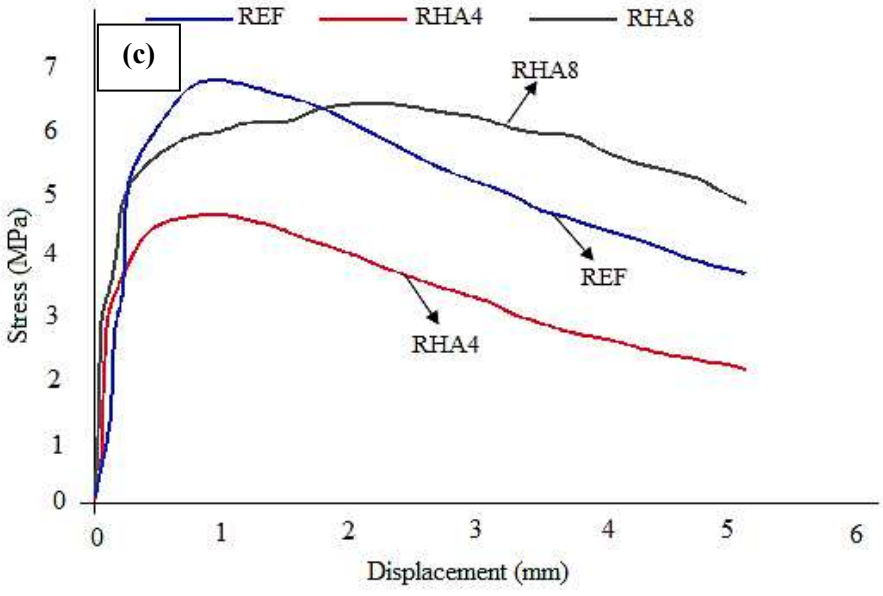
The results in terms of load-vertical displacement obtained from the resistance to reflective cracking tests are presented in Figure 7, for the three ECC thicknesses studied (20 mm (Figure 7a), 30 mm (Figure 7b) and 50 mm (Figure 7c)). The curves showed in the graphs are an average of three samples. For 20 mm of thickness, after reaching the elastic peak, the load capacity stabilizes, while the vertical displacement increases. REF mixture performed slightly better than RHA mixture, especially in terms of strength capacity after cracking. The test was interrupted when the deflection threshold of 5 mm was reached, due to a limitation of the equipment.



243
244



245
246



247

Figure 7 – Load-vertical displacement curves from resistance to reflective cracking (a) 20 mm (b) 30 mm (c) 50 mm

Figure 8 shows the typical cracking patterns obtained on the test, for thicknesses of 20 mm (Figure 8a), 30 mm (Figure 8b) and 50 mm (Figure 8c). It is possible to note that cracks are concentrated close to the induced base concrete crack. Multiple cracking can be observed, although the cracks cross the entire section of the overlay, which may lead to durability issues of the system if the cracks are not watertight [40,41].

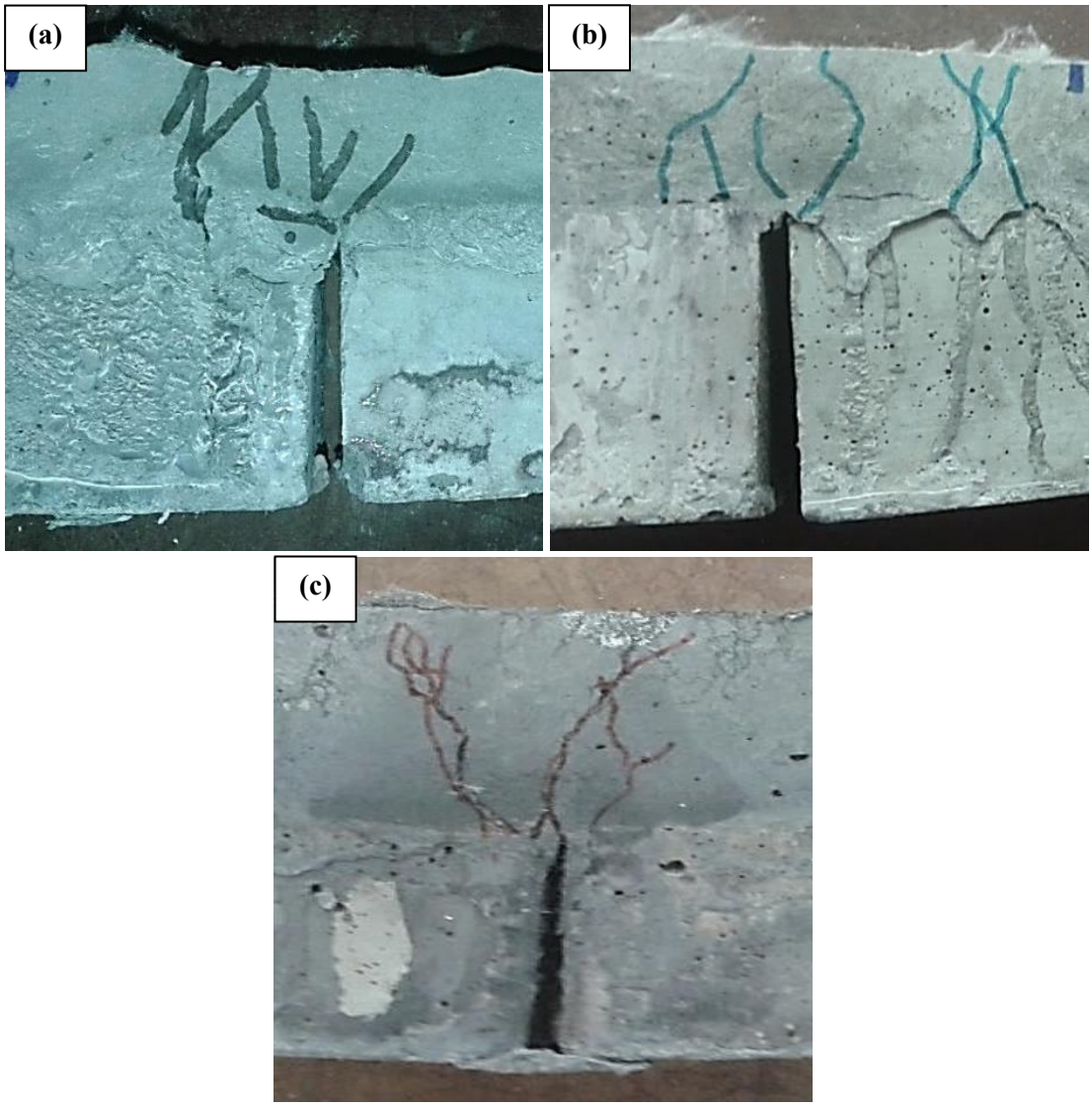


Figure 8 – Typical cracking patterns observed during resistance to reflective cracking test (a) 20 mm (b) 30 mm (c) 50 mm

In comparison to the 20 mm thickness results, there is a slight increase in the strength for the 30 mm thickness specimens, as expected. There is also an increase in the load capacity after reaching the elastic peak, indicating strain hardening behaviour. A similar cracking pattern as obtained for the 20 mm specimens was observed. For the 50 mm of thickness specimens, there was a considerable increase in the strength capacity, followed by a clear strain hardening behaviour. Similar cracking pattern is observed as for all the other thicknesses. However, the cracks do not cross the entire section, propagating to a point below the ECC surface. This is a very positive result in terms of potential performance, in the eventual use of the material as whitetopping in real life applications. The RHA8 mix had the best behaviour in terms of post-cracking behaviour.

The resistance to reflective cracking test showed that ECC can bond effectively to the base concrete, in such a way that the cracks originated from the bending test were concentrated close to the tip of the induced crack in the base concrete.

The test was not designed to provide data regarding thickness specification for concrete pavement rehabilitation, since this value needs to be properly established considering actual use and all other specific and appropriate variables. The target of the test was to identify if and how an existing concrete pavement crack would propagate towards the ECC overlay. The results obtained pointed out that all ECC mixtured tested (REF, RHA4 and RHA8) performed well, responding monolithically with the base concrete under tension. It also indicates that the strains can be suitable accommodated by the overlay, due to the high ductility and multiple cracking behaviour. The RHA mixtures performed in a satisfactory way, and may therefore be an interesting option given their greener matrix.

In terms of overall performance of crack propagation, the results were much better for all ECC mixtures when a 50 mm thickness layer was used, in comparison to the 30 mm thickness layer specimens. For the greater thickness, all tests indicated clear strain hardening and marked multiple cracking behavior, with cracks not propagating to the surface.

3.3. Bond Strength by Slant Shear

Table 4 shows the results of the bond strength by slant shear and compressive strength for REF, RHA4 and RHA8 mixtures. The average compressive strength of the base concrete was of 68.49 MPa, measured from 3 specimens with 100 mm of diameter and 200 mm of height.

Table 4 – Bond strength by slant shear and compressive strength of ECC mixes

Mix	Bond strength by slant shear (MPa)	Compressive strength (MPa)
REF	54.91 + 1.09	46.65 + 0.33
RHA4	59.21 + 1.80	60.41 + 0.73
RHA8	60.27 + 0.9	57.30 + 1.11

As expected, the values of strength were in-between the compressive strength of the base concrete and the ECC mixtures. The bond strength results were similar for both mixtures with RHA, slightly lower that the ones obtained for the reference mixture. The cracking maps obtained after the test may help explain this result.

Figure 9 shows the cracking maps after bond strength test, for REF (Figure 9a), RHA4 (Figure 9b) and RHA8 (Figure 9c) mixtures. For the REF mixture, the cracks did not propagate from one material to the other, stopping at the interface between both materials. On the other hand, the cracks in the specimens of RHA4 and RHA8 clearly propagated through both materials, showing that the bond between both materials was probably higher than the one with the reference mixture. This may be an indication that mixtures with supplementary cementitious materials bond more effectively to the base concrete. Probably, it happens due to the longer hydration period generated by the pozzolanic effect, associated with the use of the RHA, which enable better anchorage on the interface, as seen elsewhere when supplementary cementitious materials were used [42].

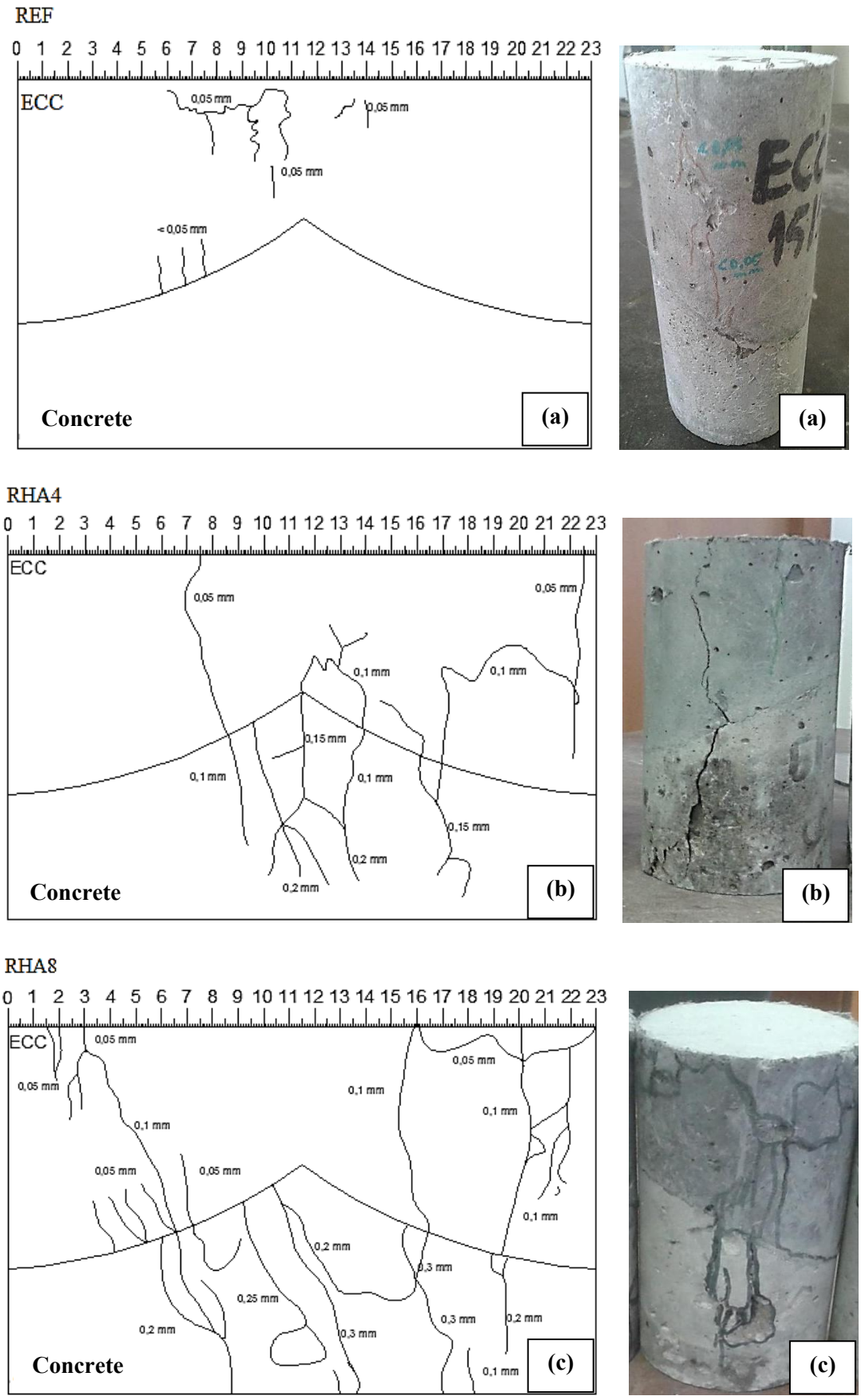


Figure 9 – Cracking map after bond strength test (a) REF (b) RHA4 (c) RHA8

4. Conclusions

The use of RHA as supplementary cementitious material in ECC mixtures was investigated in this work. The main findings of the study were:

- Mixtures with the inclusion of non-processed RHA as supplementary cementitious material showed tensile and bending behaviour similar to the reference mixture, with a suitable performance in terms of post-cracking behaviour (including the strain-hardening characteristic and the multiple cracking).
- There was no considerable difference among the results with RHA milled for 4 hours and for 8 hours, being then 4 hours of milling sufficient to process the material studied. Also, the smaller time is considered a more economic approach, being advantageous since that less process energy is consumed.
- Results from the resistance to reflective cracking test indicated that the thickness of the ECC overlay is very important for durability purposes, since that specimens with ECC layers of 20 and 30 mm of thickness did not prevent micro-cracks from propagating in the entire section. In the 50 mm of thickness, the ECC cracks stopped propagating before reaching the top surface of the section. The test also showed that the RHA-ECC mixtures tested had the ability to absorb the stresses originated from the simulated crack on the base concrete.
- ECC bonds effectively to the base concrete, in such a way that cracks propagated from one material to the other, when subjected to the bond strength by slant shear test. This behaviour was more noticeable in the RHA-ECC, when compared to the reference mixture.
- Based on the obtained data, the use of non-processed RHA milled for at least 4 hours and added at the amount of 30%, was considered a viable alternative to create greener ECC mixtures for pavement rehabilitation.

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