

Mortar modified with sulfonated polystyrene produced from waste plastic cups

Argamassa modificada com poliestireno sulfonado produzido a partir de copos plásticos descartados

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Abstract

In this work, we studied the addition of sulfonated polystyrene produced from waste plastic cups as an admixture for mortars. Mortars were analyzed with polystyrene content of 0.0; 0.2; 0.6; 1.0 and 1.4% in relation to the cement mass. The influence of polystyrene on the mortars' properties was evaluated by the consistency index, water retention, water absorption, porosity, elasticity modulus, compressive strength, flexural strength, bond tensile strength and microscopy. The increase in the sulfonated polystyrene content decreased the elasticity modulus of the mortar and, despite higher porosity, there was a reduction of water absorption by capillarity. In relation to mortar without admixture, the modified mortar showed an increase in water retention and consistency index, and a large increase in flexural strength and bond tensile strength. The significant increase of bond tensile strength (214% with admixture 1%) highlights the potential of the produced material as an adhesive mortar.

Keywords: modified mortar, polymeric admixture, sulfonated polystyrene, recycling.

Resumo

Neste trabalho estudou-se a adição do poliestireno sulfonado produzido a partir de copos plásticos descartados como aditivo para argamassas. Foram analisadas argamassas com os teores de poliestireno de 0,0; 0,2; 0,6; 1,0 e 1,4% em relação à massa de cimento. A influência da adição do poliestireno sulfonado nas propriedades das argamassas foi avaliada através dos ensaios de índice de consistência, retenção de água, absorção de água, índice de vazios, módulo de elasticidade, resistência à compressão, resistência à tração na flexão, resistência potencial de aderência à tração e microscopia. O aumento no teor de poliestireno sulfonado reduziu o módulo de elasticidade da argamassa e, apesar da elevação da porosidade, houve uma redução da absorção de água por capilaridade. Em relação à argamassa sem aditivo, a argamassa modificada apresentou aumento na retenção de água e no índice de consistência, além de um grande aumento na resistência à tração na flexão e na resistência potencial de aderência à tração. O aumento significativo da resistência potencial de aderência à tração (214% com 1% de aditivo) destaca o potencial do material produzido como argamassa adesiva.

Palavras-chave: argamassa modificada, aditivo polimérico, poliestireno sulfonado, reciclagem.

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1. Introduction

Recycling of materials has become a worthy field of research, development and application of resources, since there is an environmental and economic interest in the removal of discarded materials from the environment, avoiding the consumption of non-renewable raw material. In this sense, the search for viable routes for the use of materials, either in their original chemical form or by their chemical transformation, has been studied aiming to find a market for their application [1].

Recycling is seen as an alternative in trying to remove from the environment the waste produced daily by our society. In Brazil, about 54.38 million tons per year of municipal solid waste are produced [2]. However, a survey by the IPEA (Institute of Applied Economic Research) in the 2012 showed that, despite the waste collection being carried out in almost 90% of Brazilian municipalities, selective collection - collecting material to be recycled - takes place in barely 15% of the municipalities [2]. This solid waste largely consists of disposable plastic packaging, which is mainly fabricated of polyethylene terephthalate - PET; polyethylene - PE; polyvinyl chloride - PVC; polypropylene - PP and polystyrene - PS [3,4]. The consumption of PS plastic cups represents a considerable portion of municipal solid waste generated by humanity: its use and disposal must be rethought, aiming at sustainable consumption [5].

The construction industry is a sector of society that is currently growing rapidly. In Brazil, the rise is directly linked to economic growth and also to incentive programs offered by the current government to reduce the housing deficit. The construction processes related to this sector are responsible for a large proportion of urban pollution that affects the whole environment; however, this is also one of the sectors where part of the waste generated by social activities can be reused or incorporated in the construction process [6]. For example, research shows that sugarcane bagasse can be added in the production of cementitious composites, both in the form of fibers [7,8], ashes [9–11], and cellulose derivatives, such as cellulose sulfoacetate [12] and methylcellulose [13–15]. These additions are intended to improve both the physical and chemical properties of the composites, as well as reducing environmental pollution generated by waste. Therefore, to contribute to sustainability, it is essential to increase the practice of research, in order to minimize the impact generated by urban waste from human activities.

The mortars have curing and adhesion properties obtained by mixing binder, fine aggregate and water. Admixtures could be employed to improve some of the characteristics both in the fresh and hardened states. In preparing a quality mortar, one should think of producing it to obtain the best performance and durability, taking into account properties such as plasticity, cohesion in the fresh state, adhesion in the hardened state, cracking resistance, mechanical strength and resilience, among others.

The mortar must have adequate adhesion to the material to be applied. Adhesion is a property that is directly related to the mechanical performance influenced by the surface condition of the substrate on which it is applied, correct dosage and quality of materials, water retention capacity, thickness of the coating etc. Admixtures, which are largely soluble polymers or redispersible in water, allow the mortar to have better water retention capacity and greater plasticity in the fresh state, as well as improved mechanical properties [16,17].

A very common pathology regarding the use of mortars is the detachment of ceramic plates used in the cladding of buildings, where there is a large volume of waste generation along with an increase in the cost and consumption of new materials, where mortars with better performance would reduce many of the environmental impacts. Polystyrene (PS) has, in general, good mechanical, thermal and electrical resistance and low density. Due to these properties, it is used to produce disposable materials such as cups, food trays and plastic bags [18]. Some commercial plasticizer admixtures or superplasticizers incorporated into concrete and mortar have the presence of the sulfonate group, chemically modified group, responsible for the strong interactions with cement particles. Furthermore, the sulfonate group increases the solubility of the polymer in water, resulting in better dispersion and homogeneity of the cement paste [19–21].

Polystyrene can be recycled for the production of new materials. In a previous work, the PS obtained from discarded cups and food trays was used for the production of ion-exchange membranes [5], in the water treatment as an auxiliary agent of coagulation, flocculation and flotation for water and wastewater treatment [1,18], and as an admixture to concrete with excellent results, such as increased workability and water reduction, being classified as a superplasticizer admixture [19–21].

The objective of this study was to examine the application of sulfonated polystyrene, obtained from discarded plastic cups, as an admixture in mortars. Initially, the application for studied mortars was not defined, which could subsequently be targeted on the basis of the results obtained. To evaluate the influence of the admixture on the mortar properties, we determined the consistency index and water retention of mortars in the fresh state. In the hardened state, we evaluated the water absorption, porosity, elasticity modulus, compressive strength, flexural strength, bond tensile strength and microscopy.

2. Experimental

For the mortar composition, we used Initial High Strength Portland cement (CPV-ARI), which is a cement-free mineral addition, sand, and water in the ratio 1:4:0.84 by mass. The amount of water was chosen to achieve a 260 ± 5 mm consistency index, according to ABNT NBR 13276: 2005 [22], allowing higher workability of mortar and ease of application, for the various possible uses of the mortar. We studied 05 mortars with 0, 0.2, 0.6, 1.0 and 1.4% Sulfonated Polystyrene (SPS) on the cement mass, called A0, A0.2, A0.6, A1.0 and A1.4, respectively.

2.1 Production of sulfonated polystyrene (SPS)

The sulfonated polystyrene (SPS) was produced from the sulfonation of polystyrene plastic cups discarded post-consumer, as per already defined procedures in previous research carried out by our group [19–21]. The sulfonation was carried out with concentrated sulfuric acid (H_2SO_4 98%) and silver sulfate (Ag_2SO_4) as a catalyst. After sulfonation, the material was precipitated with water at $5^\circ C$ in an ice bath. A material with a rubbery aspect and high water solubility was produced.

An aqueous solution of the SPS treated with sodium hydroxide was used as an admixture to the mortars. The solution was produced at pH 13 at a concentration of 17% (w/w).

2.2 Preparation of the mortars

For the preparation of the mixture, we used a mechanical mixer in which water, cement, sand and the admixture were added in this order, by mixing at low speed for 30 s; this was followed by a rest period of 90 s, then blending at high speed for 60 s (ABNT NBR 13276: 2005) [22].

2.3 Testing of mortars in the fresh state

In the Consistency Index (CI) essay, fresh mortar is placed in a cone-shaped receptacle on the consistency table. After removing the mold, the table underwent 30 strokes in approximately 30 s, which caused the spreading of the mortar. Two orthogonal diameters were measured, and the value of the consistency index is the arithmetic mean of the measurements (ABNT NBR 13276:2005) [22].

For the Water Retention test, we applied a vacuum of 51 mm Hg in the fresh mortar for 15 min. From the mass difference of the mortar before and after the suction, it was possible to measure the water retention content (ABNT NBR 13277: 2005) [23].

2.4 Testing of mortar in the hardened state

To evaluate the properties of the mortar in the hardened state, we molded cylindrical specimens (5 cm x 10 cm), prismatic specimens (4 cm x 4 cm x 16 cm), and prepared molds on standard substrates for determining the bond tensile strength. The number of specimens for each test followed the recommendations of the respective standards adopted. The specimens were cured immersed in water for 28 days, and the molds for the adhesion test (Figure 1) were cured in air.

For determination of the flexural and the compressive strength, the cylindrical specimens were previously capped with sulfur and tested for compression, at 28 days of age in the EMIC universal testing machine, model DL 60000, with load cell of 10 kN. The loading rate was 0.25 ± 0.05 MPa/s (ABNT NBR 7215:1996) [24]. The number of specimens tested was 04 for each mortar.

The prismatic specimens were tested at three points bending, at 28 days of age in the INSTRON universal testing machine, model 5982, with load cell of 5 kN. The load was applied at a rate of 50 ± 10 N/s (ABNT NBR 13279:2005) [25]. Six specimens of each mortar were tested.

The elasticity modulus was determined in 03 cylindrical specimens by mortar, at the age of 28 days in an EMIC universal testing machine, model DL 60000, with load cell of 10 kN. The deformation was measured with strain gauges (ABNT NBR 8522:2008) [26].

The properties of water absorption by immersion were determined for 02 cylindrical specimens. The samples were placed in an oven at temperature of 105 ± 5 °C for 72 h and then weighed. Subsequently, they were immersed in water at 23 ± 2 °C and maintained at this condition for 72 h. After this saturation step, the samples were placed in a container with water and brought to boiling for a period of 5 h, then were cooled at 23 ± 2 °C, and the mass of the saturated samples was determined (ABNT NBR 9778:2005) [27]. The water absorption was calculated as a percentage relative to the dry mass of the specimen.

The porosity and the pore size distribution were determined by the mercury intrusion porosimetry (MIP) test. Two samples for each mortar of approximately 1 g were previously oven dried at a temperature of 100 ± 5 °C up to constant mass. The tests were realized in a Micromeritics AutoPore III high-pressure mercury intrusion porosimeter, with a maximum pressure of 476 MPa. Each sample was tested twice following the test parameters listed in Table 1.

To determine the water absorption by capillarity, we tested 03 cylindrical specimens at 28 days of age. The masses of the specimens were determined, and these were then dried in an oven at 105 ± 5 °C to constant mass. Then, they were cooled to temperature 23 ± 2 °C and weighed. The specimens were immersed in water; their masses were determined after 3, 6, 24, 48 and 72 h, counted from their placement in contact with water. The water absorption by capillarity is expressed in g/cm² and is calculated by dividing the mass increase by the cross-sectional area of the specimen surface in contact with the water (ABNT NBR 9779:1995) [28].

The mortars were applied to standard concrete rectangular substrate (45 cm width, 120 cm length and 5 cm thickness) for adhesion determination (ABNT NBR 14082: 2005) [29]. The application of the mortar on the substrate was standardized with the launch of approximately 20 cm height, and the thickness was fixed at 1 cm (Figure 1).

The bond tensile strength test was carried out according to the Brazilian norm ABNT NBR 15258: 2005 [30], which specifies that the mortar must be placed onto a substrate, and that after normal curing (i.e. 28 days), the test should be carried out by measuring the strength applied to take the mortar off the substrate.

The mortars were also characterized by Scanning Electron Micros-

Table 1 – Parameters of the mercury intrusion porosimetry test

| Parameters | Test conditions | |
|--------------------------|-----------------|---------------|
| | Low pressure | High pressure |
| Evacuation pressure | 6.67 Pa | - |
| Evacuation time | 5 min | - |
| Mercury filling pressure | 4.48 kPa | - |
| Equilibration time | 30 s | 30 s |
| Maximum intrusion volume | 100 mL/g | 100 mL/g |
| Contact angle | 130° | 130° |

Figure 1 – (a) Standard concrete substrate and mold for mortar application, (b) cure of the mortars applied for standard substrate and (c) mortar after adhesion test



copy (SEM). The samples were taken from fractured section of the specimens tested to bending, and were oven dried at 100 °C. Then, the samples were coated in gold. Images were obtained by secondary electrons in a Carl Zeiss EVO MA10 scanning electron microscope.

3. Results and discussion

3.1 Production of sulfonated polystyrene (SPS)

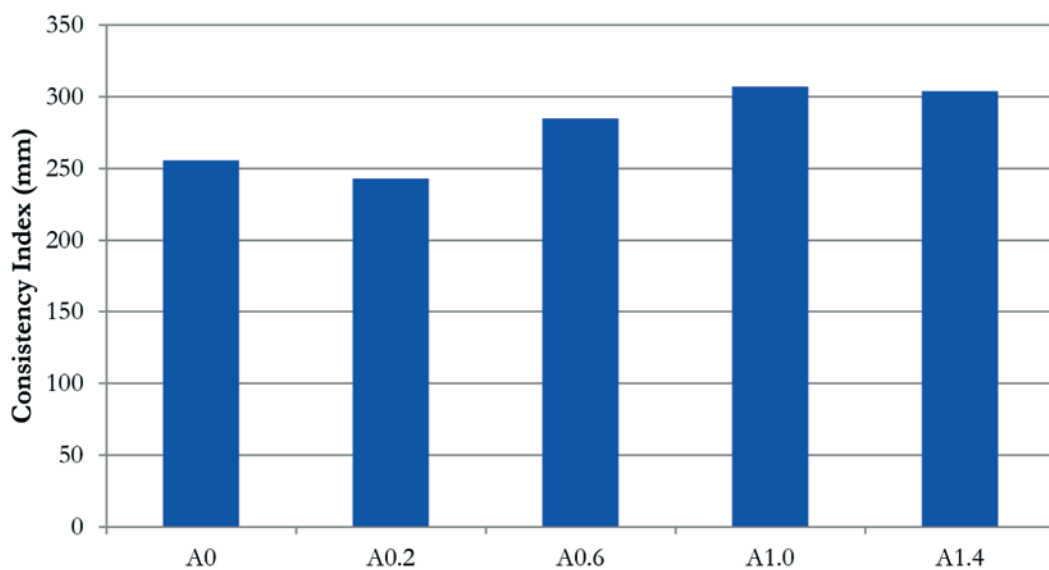
The degree of sulfonation of Sulfonated Polystyrene obtained was

58–63%. The solution of prepared sulfonated polystyrene presented a density of 1.16 g mL⁻¹ at 27 °C, determined by pycnometry.

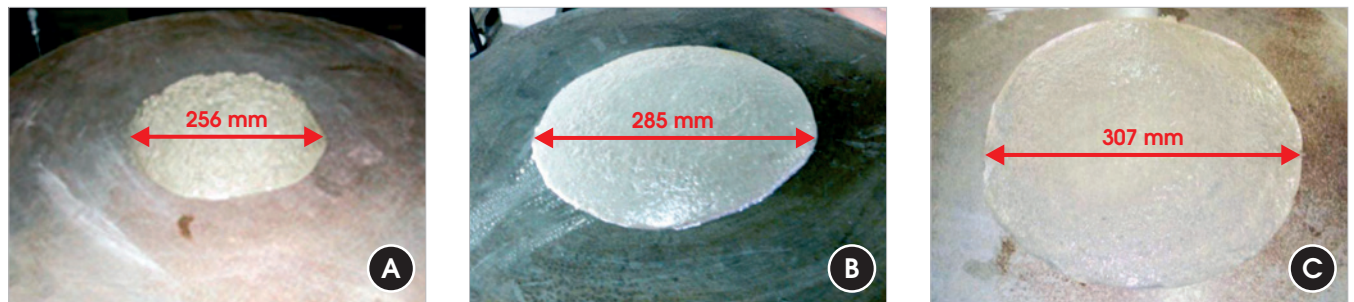
3.2 Evaluation of fresh mortars

The SPS admixture was dissolved in sodium hydroxide solution in order to neutralize it, where the volume of water used in the solution is only required for the polymer dispersion, the absence of free water that can act on the dispersion of the cement particles. Therefore, we chose not to consider the volume of admixture to the water/cement ratio of mixtures. From Figure 2, there was consistent index growth with the increase of admixture SPS quantity,

Figure 2 – Consistency index of the mortars



**Figure 3 - Mortar spreading after the test of consistency index:
(a) reference mortar, (b) mortar with 0.6% SPS and (c) mortar with 1.0% SPS**



reaching approximately 20% of increase to levels above 1%. Even when there was an increase in admixture content of 1–1.4%, there was no increase in fluidity, showing that the admixture had already reached the optimum content, and that the increase fluidity of the mixture is purely attributed to admixture action, and not the water present in its solution.

Note that the water/cement ratio has not changed; the increase in the consistency index is because the anionic long-chain molecules of the sulfonated polystyrene admixture became adsorbed on the surface of the cement particles that are effectively dispersed in water, as reported by Assunção et al. [19] and Royer et al. [20]. It was also observed that the mortar with SPS addition provided a cohesive aspect and more fluid than the reference mortar, as can be seen in Figure 3.

It can be seen from Figure 4 that the additions of 0.2%, 0.6% and 1.0% of polymeric admixture resulted in increased of water retention relative to the reference mortar, with an optimum level of 0.6%.

The improved dispersion of the cement particles by the action of the admixture also promotes greater water adsorption to the surface of the cement grains, so that the water is retained. However, for higher polymer contents, the fluidity of the water-cement system increases leaving part of free water, causing a decrease in water retention, as can be observed for the A1.4 mortar.

3.3 Evaluation of mortars in the hardened state

From Figure 5, it can be seen that there was a progressive increase in the porosity of the mortars with admixture content relative to the reference, reaching a 19% increase for A1.4. This increase in void content was accompanied by an increase in absorption above 50%, regardless of admixture content. It is known that one of the effects of the application of surfactant admixtures in cement mixtures is air void entrainment [31]. As a result of the increase in void content in the modified mortars, the bulk density decreased,

Figure 4 - Water retention values of produced mortars

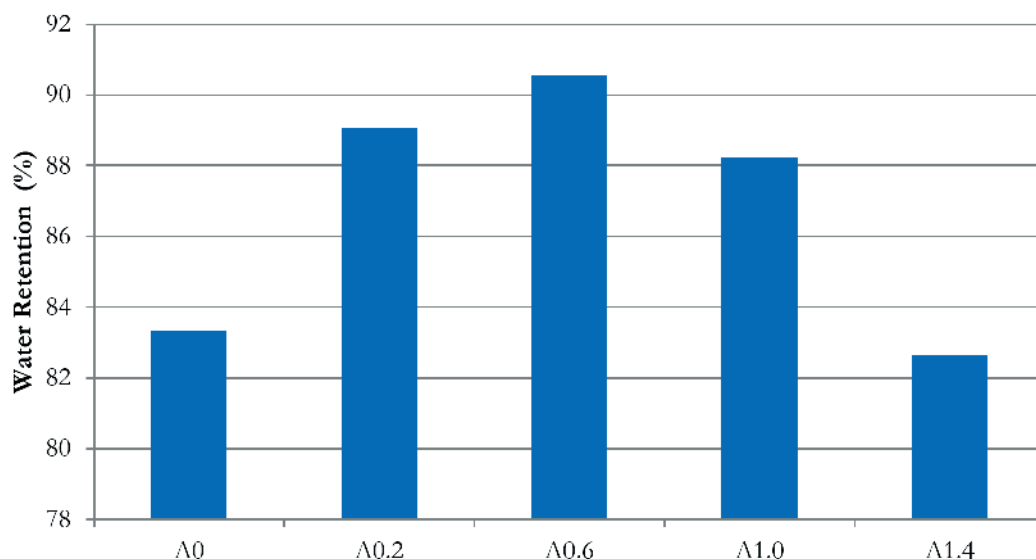
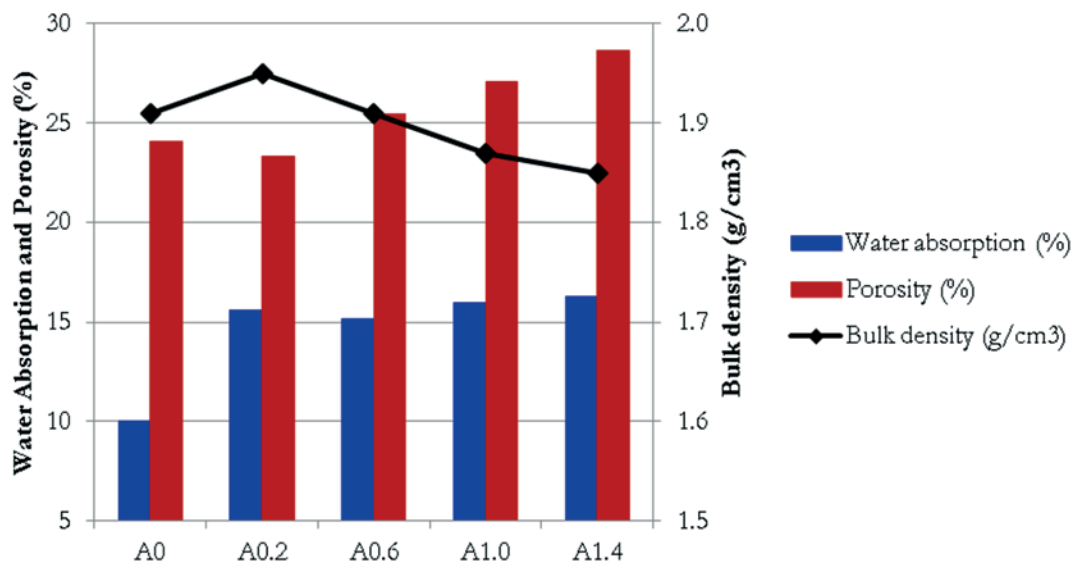


Figure 5 - Results of water absorption, porosity and bulk density for the mortars



although this was less pronounced variation, reaching a maximum reduction of 3% to mortar with 1.4% of admixture.

Figure 6 shows the mortar pore size distribution obtained by mercury intrusion porosimetry. There was a reduction in the number of pores with dimensions in the range of 10^2 – 10^5 nm with the presence of the admixture, probably by filling these voids with the polymer, as observed on microscopic images. However, in the range of

pores above 10^5 nm, there was a significant increase in the volume of pores in the modified mortars, especially for the highest levels of admixture, e.g. A1.4. This pore size range corresponds exactly to the pores entrained by surfactant admixtures [31].

Water absorption by capillarity is related to the permeability of mortar. In Figure 7, a reduction of absorption by capillarity was observed for all mortars with the addition of the polymer,

Figure 6 - Mortar pore size distribution obtained by mercury intrusion porosimetry

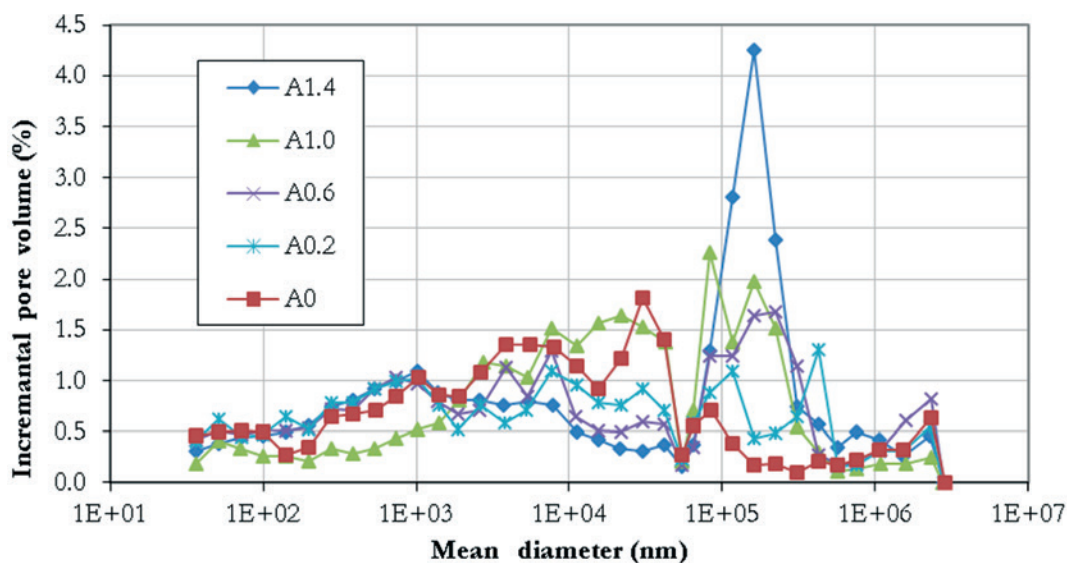
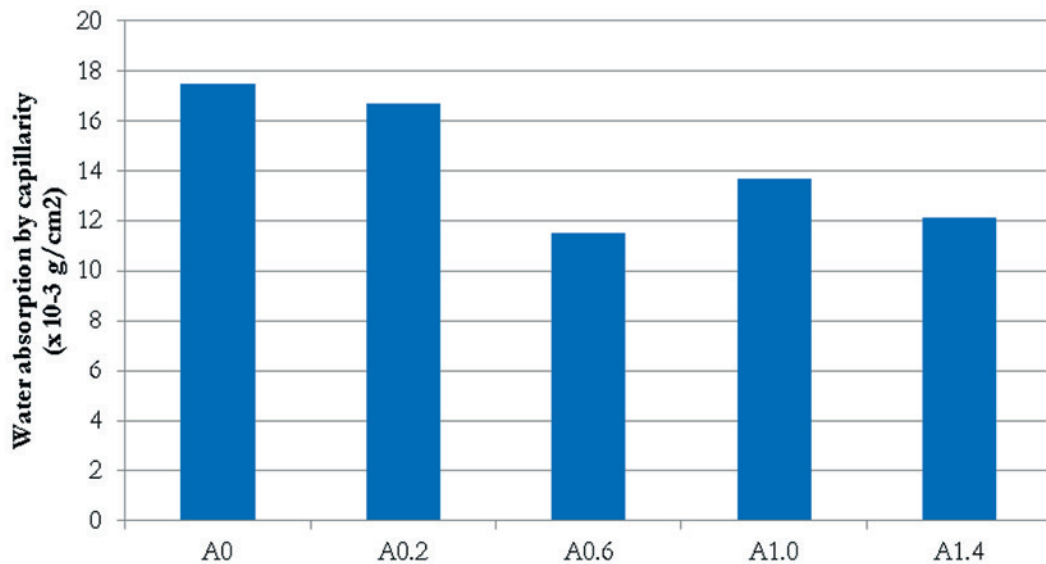


Figure 7 - Average values of water absorption by capillarity for the mortars

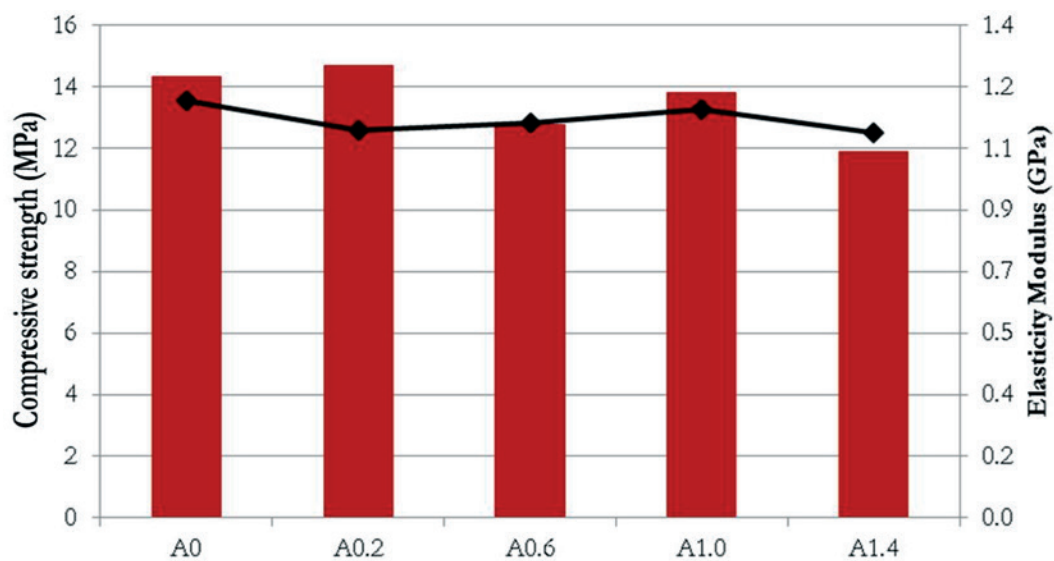


decreasing by 34% to the level of 0.6% admixture. Despite the elevation of porosity of the modified mortars, the polymer presence reduced the permeability, possibly explained by non-interconnected pores, and also by the formation of polymeric film in the microstructure of the mortars [17,32], as can be seen in the micrographs.

From Figure 8, there was a reduction in compressive strength with

the incorporation of the polymer; however, this reduction was not significant, as could be expected considering the large increase of porosity of the modified mortars. The A0.2 mortar showed an increase in compressive strength, demonstrating that the polymer improves the mechanical properties, even in small dosages. For higher levels of polymer like mortars A1.0 and A1.4, the reduction in compressive strength was not significant. This may be a result of

Figure 8 - Compressive strength and average values of the elasticity modules at 28 days of age



the greater resistance from mortars to microcracking by the action of polymeric films formed, as emphasized by Ohama [17].

The use of sulfonated polystyrene led to a decreased elasticity modulus compared to the reference mortar. The maximum reduction was approximately 8% for the mortar with highest polymer content (1.4%). However, this reduction can be advantageous for applications where it is subjected to large deformations, which could cause cracking of the mortar. Thus, a modified mortar submitted to a particular deformation will be under lower tension one without SPS.

Note in Figure 9 that the tensile strength increased in bending with levels of the SPS, indicating an optimum range of dosage of 1% the admixture.

There was an increase in the tensile strength in bending for all levels of admixture, reaching 23.72% for the A1.0 mortar. Therefore, even with low levels of SPS, the presence of polymer in the mortar appears to form a film on the microstructure of the paste, which tends to improve its tensile strength, as pointed out by Ohama [17], Assunção et al. [19] and Royer et al. [20].

There was an increase of bond tensile strength for all mortars modified with SPS. Still, as observed in the tensile strength in bending, the largest content was 1%. It should be noted that the increase was 69% even for low levels of 0.2% admixture, reaching a 214% increase for mortar A1.0 relative to the reference mortar (A0). This stronger physical bond between the modified mortar and the substrate makes it a good adhesive mortar for the settlement of ceramic tiles, for example, where the resistance of adhesion should be high.

Figure 10 presents SEM images of the mortars. The micrograph of Figure 10a, at a magnification of 15000x, shows the microstructure of the mortar with the presence of hydrated cementitious compounds such as calcium silicate hydrate. Figures 10b–10e, with magnifications of 30000x, show the structure formed by the

polymer in the mortars modified with SPS. It can be seen in the micrographs the deposition of sulfonated polystyrene in the form of lamellae, and the presence of polymeric film (SPS) on the microstructure of the modified mortars. As the admixture content increased, modification in the microstructure of the polymeric film was observed in the mortars, from a lamellar structure to the more continuous polymeric film [33], as shown in Figures 10c–10e. In Figure 10c, it is possible to identify both types of polymer structures: lamellar structures in the center of the micrograph and continuous film to the right, indicated by the arrow.

In the micrograph of Figure 11, at a magnification of 2500x, the presence of polymeric film on pores was observed, which may have contributed to the improvement of this mortar's tensile properties (A1.0), where the microcracks in the modified mortar under stress are bridged by the polymeric films or membranes formed. This prevented crack propagation and, simultaneously, a strong cement hydrate-aggregate bond is developed [34].

4. Conclusions

Sulfonated polystyrene produced from discarded plastic cups presented excellent results as an admixture for mortars, improving properties both in fresh and hardened states. There was an increase of mortar fluidity with the incorporation of the admixture, reaching 20% with 1.4% of SPS. Water retention was also improved relative to the reference mortar until the level of 1.0% admixture.

In the hardened state, the admixture incorporation resulted in a significant increase of mortar porosity. However, this increase caused no reduction of the same order of the compressive strength and elasticity modulus. This was because the presence of polymeric film improved the resistance of mortars to microcracking, thus overcoming the negative influence of porosity on mechanical properties. This was especially evident in the tensile properties,

Figure 9 - Values of flexural strength and bond tensile strength at 28 days of age

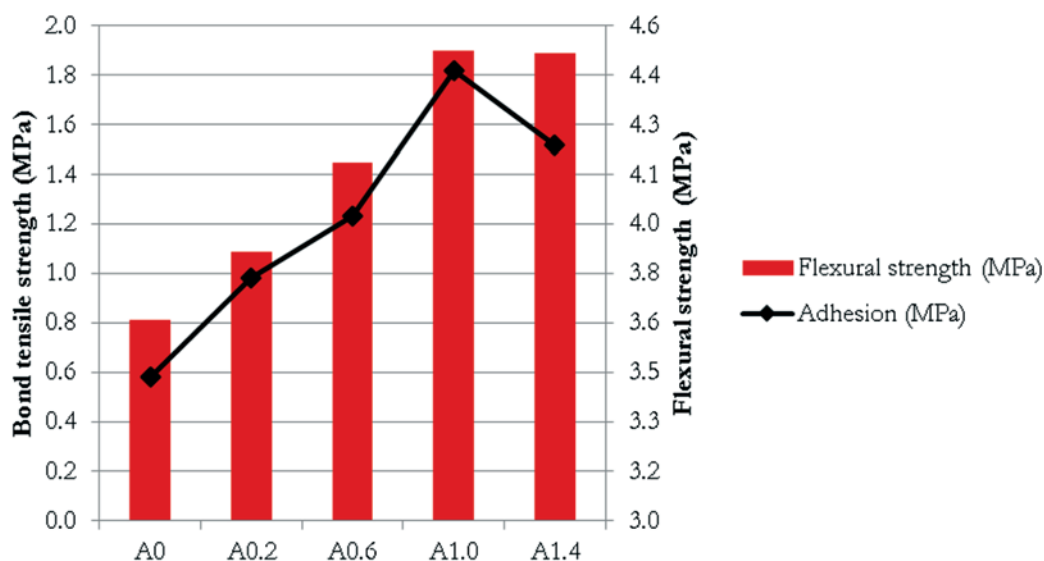


Figure 10 – Micrographs of mortar samples: (a) reference mortar, (b) mortar with 0.2% SPS, (c) mortar with 0.6% SPS, (d) mortar with 1.0% SPS and (e) mortar with 1.4% SPS

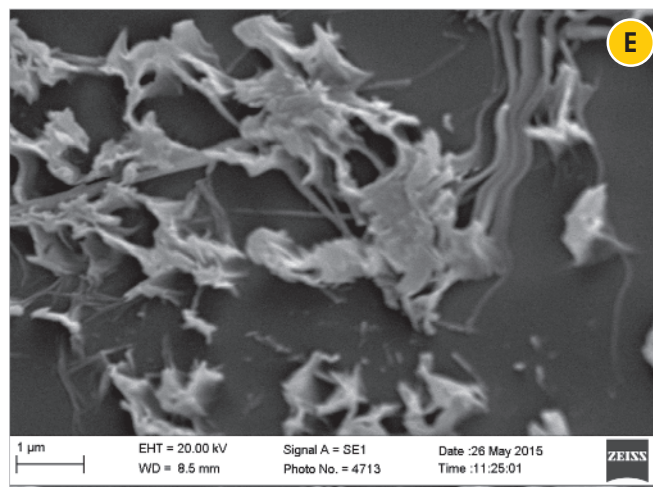
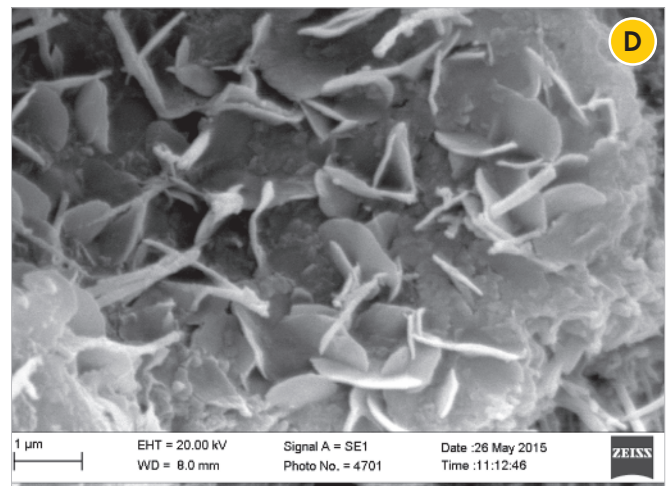
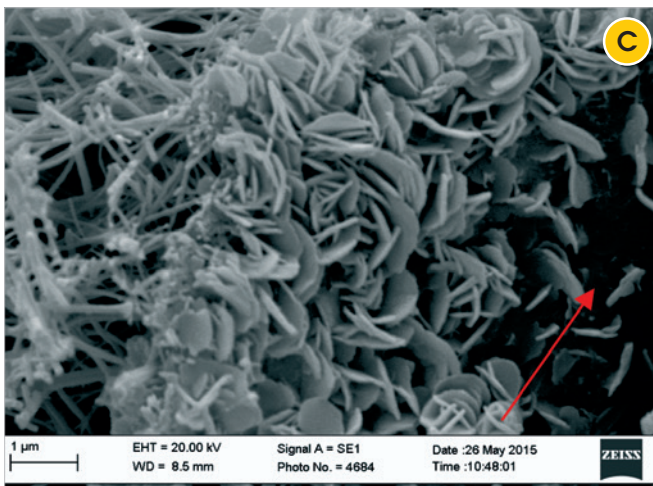
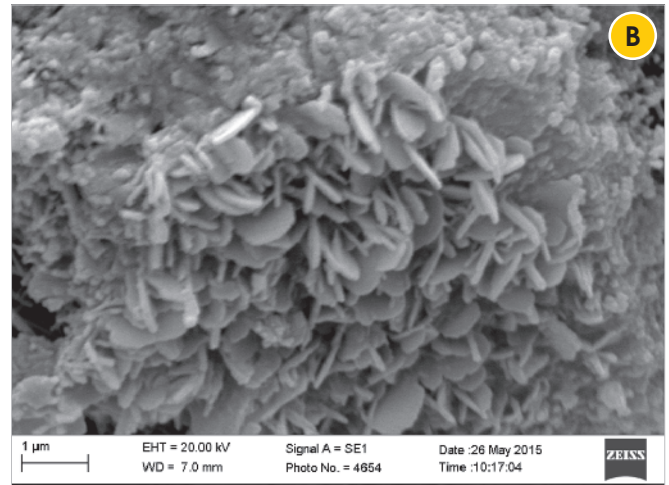
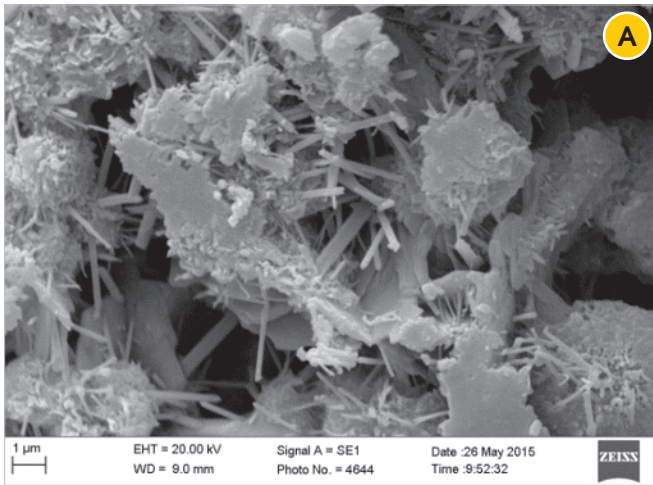
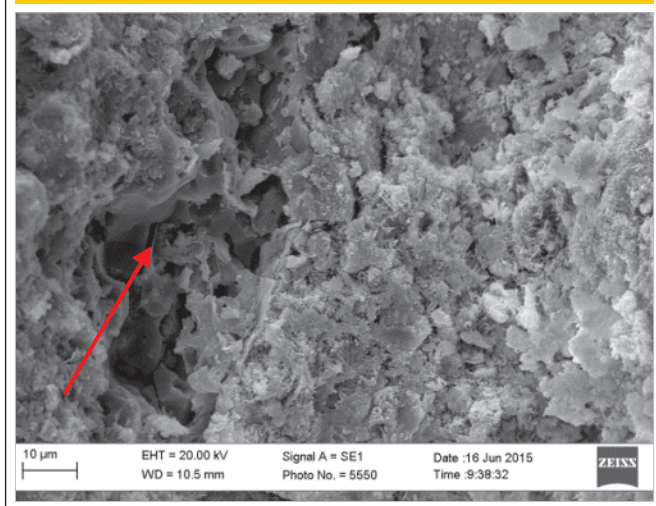


Figure 11 – Micrograph for mortar with 1.0% SPS



where the mortar modified with polymer showed an increase of about 24% in tensile strength, and above 200% of the adhesion resistance of the mortar with 1% admixture, relative to the reference. Another advantage observed in mortar modified with polymer was the reduction of water absorption by capillarity, showing the effect of the polymeric film in promoting the discontinuity of the formed pores. The exceptional increase of bond tensile strength of the modified mortar makes it a good adhesive mortar for applications where adhesive strength should be high. One should still emphasize the advantage of the sulfonated polystyrene admixture being produced from discarded plastic cups, preventing the disposal of PS in the environment. Furthermore, the production and application of better-performance mortars in the cladding of buildings, reducing the detachment of the cladding, a fairly common pathology, promotes higher sustainability of the construction.

5. Acknowledgments

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