Application of Cellulose Sulfoacetate Obtained from Sugarcane Bagasse as an Additive in Mortars

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ABSTRACT: In this study, cellulose sulfoacetate (CSA) was produced with cellulose extracted from sugarcane bagasse with sulfuric acid as a catalyst under homogenous conditions so that the derivative could be applied as an additive for mortar of Portland (Cimento Portland de alta resistência inicial, CPV-ARI) cement. The IR spectra showed that only precipitate C presented the characteristic band of sulfate groups (807 cm^{-1}) and was, therefore, the sulfoacetylated derivative. CSA presented a degree of acetylation of 2.70 and a degree of sulfonation of 0.063, and the presence of sulfate groups in the derivative displaced the position of C(6) to a lower field in the ^{13}C-NMR spectrum and influenced reduced viscosity as a function of concentration. The evaluation of CSA as an additive showed that for a polymer/cement ratio of 0.6%, an increase of 13.80% was achieved consistently, and an increase of 21.05% in the potential tensile of adhesion strength was also achieved. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 124: 510–517, 2012

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INTRODUCTION

There is an increasing demand for bioenergy in the world, and therefore, there is a frank expansion of alcohol production for fuel (ethanol). In this sense, Brazil, being the largest sugarcane producer worldwide, has also become the greatest producer of residual biomass, which is sugarcane bagasse.

In the state of Minas Gerais, sugar and alcohol activity is concentrated in the Triangle region, with about 70% of the industries. Currently, 36 mills are active in the state, and by 2012, there will be 52. Investments on the order of US$3 billion are to be made during the period. It is estimated that in the 2012/2013 harvest, 84 million tons of sugarcane will be processed, compared to the current 42.8 million tons. It is also expected that in 2012/2013, 4.2 billion L of alcohol and 4.6 million tons of sugar will be produced.[1] Each ton of sugarcane used generates 280 kg of bagasse. This industrial residue has been used to generate electricity and to produce second-generation ethanol. However, other possibilities that also aim at adding value to the residue are extremely important and should be investigated.

There are several studies in the literature on the use of sugarcane bagasse.[2–7] In sugarcane bagasse, the cellulose fraction is more abundant, corresponding to about 30–50%. This allows the residue to be used for chemical modification with a view to producing cellulose derivatives.[8]

Several cellulose derivatives can be produced from the chemical recycling of sugarcane bagasse, such as cellulose acetate, methyl cellulose, and cellulose sulfoacetate (CSA).[9–11] The cellulose derivatives have in common increased solubility in most organic solvents. The latter can be applied to a number of industrial processes, such as the recovery of heavy oils, effluent treatment, and concrete additives in civil construction.[12]

The derivative studied in this work was CSA obtained from sugarcane bagasse. The feasibility of producing CSA from this agroindustrial residue, as far as we know, has not yet been investigated in the literature nor has its use to produce additives for civil construction.

Water-soluble CSA, produced by the direct aceto-sulfonation of cellulose, was initially synthesized by Chauvelon et al.[13] The incorporation of sulfate groups into the glycosidic units confers high water solubility on the derivative. This solubility is due to the contribution of sulfate groups, which reduces the initial intermolecular interaction present in the juxtaposed glucose chains (hydrogen bond); this did not allow the solubilization of cellulose because these interactions were initially very strong.[14] Solubility in water is one of the main parameters of using this polymer in civil construction as a cement mortar additive.

In the CSA synthesis from sugarcane bagasse, a polyelectrolyte is produced, which, due to the presence of the sulfonic groups, can act as an additive. The action mechanism of some additives is based on the phenomenon of polyelectrolyte adsorption by the cement particles. In this process, the polyelectrolyte with sulfonic groups linked to the chain adsorbs to the cement particles and confers a negative liquid load on them. This effect causes repulsion between the cement particles and favors their dispersion in water and prevents cement flocculation; this improves the workability of the mixtures in a fresh state.[15–17] These polymers are called *plastifying additives*, and they allow one to use a smaller amount of water to prepare cement mortars and concrete. This makes them more resistant and reduces the amount of cement used in a work (for a lower cost and reduction of damage due to the high release of heat during hydration) and/or increases water viscosity, reinforcing the cohesion and stability of the cement-based systems. These factors are essential for preventing the excessive loss of water in the substrate, which can cause failures in the adhesion mechanism; they also improve the properties of the mixtures in the fresh state.

The sulfoacetate derivative was characterized by Fourier transform infrared (FTIR) spectroscopy, determination of the degree of substitution of acetyl groups (DS_{acet}) through a saponification reaction, determination of the degree of substitution of sulfate groups (DS_{sulf}) by elemental analysis, ^{13}C-NMR, and measures of viscosity. The application of CSA was also investigated as an additive for mortars of Portland CPV-ARI cement. The evaluation of this derivative as an additive was based on tests of consistency, mechanical strength to the compression of hardened mortar, and potential tensile of adhesion strength.

EXPERIMENTAL

Purification of the sugarcane bagasse

The cellulose was extracted from sugarcane bagasse according to the methodology developed by Filho et al.[8] as follows: 8 g of dry sugarcane bagasse was kept for 24 h in 150 mL of distilled water to remove the water-soluble extracts. Later, the bagasse was filtered and kept in 150 mL of sodium hydroxide (0.25M) for 18 h. Next, the mixture was vacuum-filtered, and the bagasse was placed in reflux with three successive changes of a mixture containing 20% (v/v) of nitric acid in ethanol, and this mixture was changed each hour. Once the reflux had ended, the mixture was filtered and washed with distilled water and then dried at 105°C for 3 h.

CSA synthesis

The cellulose obtained from purified sugarcane bagasse was sulfoacetylated according to the methodology described next, which was a modification of the Chauvelon et al.[13] procedure: 1 g of cellulose and 20 mL of glacial acetic acid were shaken at ambient temperature for 10 min. This procedure was repeated. After this first treatment, the residue was immersed in a mixture containing 20 mL of glacial acetic acid and 0.13 mL of sulfuric acid concentrate and shaken manually for 1 min. Then, 10 mL of acetic anhydride concentrate shaken manually for 1 min was added to the mixture. The system was then shaken for 30 min at 40°C. At the end of this time, a solution of acetic acid in water (7/3 v/v) was added to the mixture, and the mixture was shaken slowly for 30 min. It was centrifuged to separate the residue (precipitate A) from the supernatant. Slowly, 20 mL of water was added to the supernatant, and it was placed in the refrigerator for 16 h. The cellulose acetate formed (precipitate B) was separated by centrifuging, and the supernatant was neutralized with a solution of NaOH (4 mol/L). After neutralization, the volume of supernatant was reduced to three-quarters of the original volume and left to rest for the precipitate to form (precipitate C). This precipitate was washed to remove all of the sodium acetate produced during synthesis. The CSA was then dried in the oven at 50°C.

FTIR spectroscopy

The material, in powder form, was prepared in the configuration of KBr pastilles in a proportion of 1:100 (w/w) and analyzed in a PerkinElmer Spectrum 1000 instrument (Massachusetts, USA) with 4 cm^{-1} resolution and 28 scans.

Determination of the DS$_{acet}$

DS$_{acet}$ of CSA was determined with a saponification reaction.[18]

Elementary analysis

Carbon (C) and hydrogen (H) were determined with a CHN PerkinElmer 2400 elemental analyzer. The sulfur (S) was analyzed in the atomic emission spectrometry by plasma with induced argon. Oxygen (O) was determined by the difference of the percentages of elements determined previously.

^{13}C-NMR

The ^{13}C-NMR spectra of the CSA sample, in the solid state, were measured in a magnetic field of 7.04 T with a cross-polarization/magic angle spinning spectrometer 7 mm test tip of a Varian INOVA unit (Vernon Hills, Illinois, EUA). The measurements were performed in the Chemistry Department at the University of Coimbra in Portugal with a 4.5 kHz rotation, ^{13}C 75.43 MHz frequency, contact time of 0.7 ms with pulses of $\pi/2$ (a 4 µs length), and a waiting time of 1 s to perform another cycle.

Determination of the reduced viscosity

The reduced viscosity of CSA was determined with an Ostwald viscosimeter. Six determinations were performed at a temperature of 25°C for each dilution. The solutions were prepared in water in a concentration range of 0.2–1.9 g/L and filtered before measurement.

Studies of CSA application as an additive in civil construction

The mortars were prepared from Portland CPV-ARI cement according to the Brazilian technical standard NBR 7215,[19] which corresponds to ASTM C 109. The mortars were prepared by the mixture of the components, cement, sand, and water, in amounts of 1000, 3930, and 700 g, respectively. The polymer was previously solubilized in water and added to the cement/sand/water mixture at polymer/cement ratios of 0.2, 0.6, and 1% (m/m). The reference mortar was prepared without addition of the polymer.

The consistency of the mortars was measured with a table to determine the consistency index, according to technical standard NBR 7215.[19]

The tests of mechanical strength to compression were performed in a machine of tests of mechanical strength to compression Emic DL60000 (São José dos Pinhais/Paraná), where three hardened mortar references without additive and three hardened mortars of the samples with additives were broken at 7 and
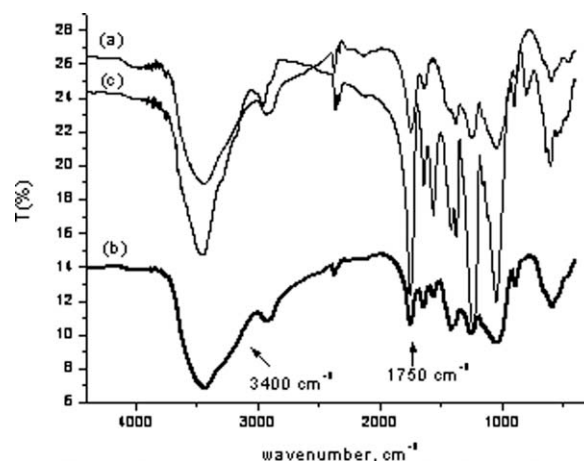

Figure 1 FTIR spectra for the materials produced: precipitates (a) A, (b) B, and (c) C.

28 days of curing, according to the technical standard NBR 7215.[19]

The tests to determine the potential tensile adhesion strength were performed in an instrument for pulling out and measuring the adhesion in mortars (Pavitest digital model, Belo Horizonte, Minas Gerais), according to the technical standard NBR 15258/2005.[20]

RESULTS AND DISCUSSION

CSA synthesis and FTIR spectra

According to Chauvelon et al.,[13] acetylation and sulfonation can be performed simultaneously, depending on the molar ratio of acetic anhydride to cellulose. The amount of acetic anhydride used reached 25 mol/mol of anhydroglucose. This value exceeded that used by Chauvelon et al.[13] The structure and chemical composition of the cellulose sources may have been responsible for the differences because we used sugarcane bagasse cellulose, and Chauvelon et al.[13] used microcrystalline cellulose and cellulose from maize and wheat bran to obtain the derivative. The use of the same amount used in the Chauvelon et al.[13] work resulted in a large amount of superficially acetylated material and a low amount of sulfoacetylated material, with only 6% of the initial cellulose being converted into sulfoacetylated derivative. The modification of this method enabled the production of three derivatives, which were evaluated with the FTIR spectra shown in Figures 1 and 2. Table I shows the assignments of the main absorption bands in CSA.

The FTIR spectra of the materials produced had a band at approximately 1750 cm^{-1}, which confirmed that all of the products were acetylated. With regard to the physical aspect of the materials produced, precipitate A presented a fibrous structure, which
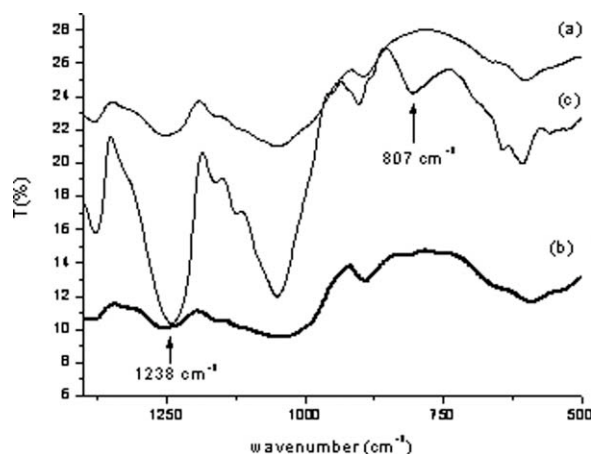
Figure 2 FTIR spectra for the materials produced (1400–500 cm^{-1} region): precipitates (a) A, (b) B, and (c) C.

was very similar to the original cellulose. Also, this material was not water soluble, which may have indicated only a superficial modification without any significant alteration of its crystalline structure. With regard to precipitates B and C, they were obtained in powder form. Although the physical aspects of precipitates A and B were visually different, their FTIR spectra were similar. These materials were also water insoluble. On the other hand, precipitate C presented greater changes in its FTIR aspects and solubility in water compared to precipitates A and B. Initially, it can be pointed out that precipitate C presented the highest degree of substitution compared to the acetyl groups among the three materials because its FTIR spectrum presented greater intensity of the band attributed to the carbonyl group compared to the band attributed to the stretching of the hydroxyl group at approximately 3500 cm^{-1}. Moreover, the fundamental aspect observed for this material was its increased water solubility. The main reason why this material was

more water soluble was that, besides being acetylated, sulfate groups were also incorporated into the glycosidic units. This could be proven by the presence of a band at 1238 cm^{-1}, a band of a sulfate ester attributed to the S=O and —COSO$_3$ stretching in the cellulose sulfate infrared.[21] This band was present in all of the materials evaluated, with greater intensity for precipitate C. The presence of this band was noted through the expansion of the spectrum of FTIR, shown in Figure 2. The presence of sulfate groups could be better evaluated by means of the band in the 810 cm^{-1} region.[22] For precipitate C, a band was observed at 807 cm^{-1}; this indicated that the sulfate groups were localized at position 6 of the glycoside ring.

Therefore, analyzing the FTIR spectra, we concluded that precipitate C was the sulfoacetylated derivative because the characteristic bands of the acetyl and sulfate groups were seen. Moreover, this derivative presented high solubility in water compared to the others. This was attributed to the presence of sulfate groups, which diminished the initial intermolecular interaction in the juxtaposed glucose chains (hydrogen bond) and did not allow the cellulose to become soluble because these interactions were initially very strong. Considering this finding, we performed the later characterizations and the study of its application in civil construction only with precipitate C.

Determination of the degree of acetylation and sulfonation of the CSA

DS$_{acet}$ of CSA was determined by a saponification reaction, and the value obtained was 2.70 ± 0.08, which corresponded to 42.20% of the acetyl groups. This value characterized precipitate C as a trisubstituted acetylated derivative. On the other hand, DS$_{sulf}$ was determined by a theoretical calculation from the results obtained by elementary analysis, shown in Table II, and the value obtained was 0.063. It was also estimated that there was a sulfate group for 16 units of anhydroglucose. Table II shows the percentages of carbon, hydrogen, oxygen, and sulfur for samples of cellulose acetate and CSA.

Comparing the percentages of sulfur of the two samples analyzed, we perceived that, for CSA, this percentage was approximately 5.5 times greater than for cellulose acetate, which presented only 0.12% S. This small percentage of S for the cellulose acetate sample could be attributed to the synthetic route of

TABLE I
Assignments of the Main Absorption Bands in CSA

Wave number (cm^{-1})	Assignment
3490	ν(O—H) cellulosic
2960	ν asymmetric (CH$_3$)
2880	ν symmetric (CH$_3$)
1750	ν(C=O) of ester
1634	δ of water
1429	δ asymmetric CH$_2$
1370	δ symmetric CH$_3$
1321	δ CH$_2$
1238	δ axial (C=O)
1157	ν(C—O)
1120	ν(C—O)
1052	ν(C—O)
807	δ S—O—C
909	δ angular (CH$_2$)
606	C—O of acetyl groups

TABLE II
Results Obtained by Elemental Analysis

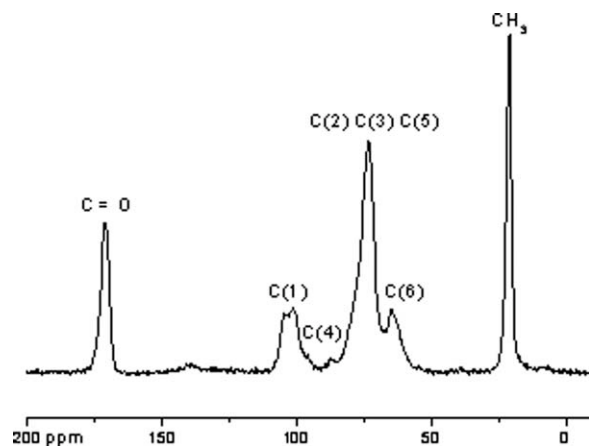| Sample	C (%)	H (%)	O (%)	S (%)
Cellulose acetate	47.99	5.53	46.33	0.12
CSA	44.75	5.50	49.10	0.65

Figure 3 ^{13}C-NMR spectrum of the CSA.

preparation of the cellulose acetate, which used sulfuric acid as a catalyst. This percentage was not enough to characterize the material as a sulfoacetylated derivative because the derivative, which presented a DS_{acet} of 2.84 ± 0.06 (43.40% acetyl groups), was not soluble in water; this was different from the sulfoacetylated derivative. Thus, it was classified as what was described by the literature, which was that cellulose acetates are usually insoluble in water for a degree of substitution greater than 1.[22] On the other hand, for CSA, even with a degree of acetylation higher than 1, 0.65% S was sufficient to change the physical and chemical characteristics in relation to the acetylated derivative, for instance, water solubility, which could be attributed to the contribution of the sulfate groups.

^{13}C-NMR

Figure 3 shows the ^{13}C-NMR spectrum of CSA. The resonance lines for the derivative could be attributed to C=O (175 ppm), C(1) (101 ppm), C(6) (65 ppm),
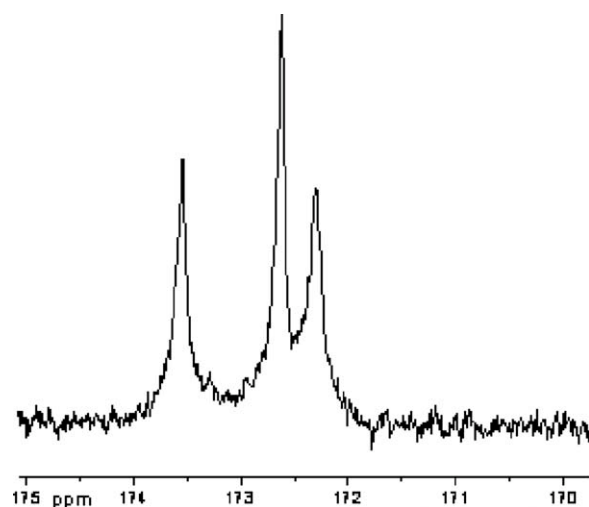
and CH$_3$ (21 ppm). The set of resonance lines located at 70–82 ppm belonged to C(2), C(3), and C(5). According to the literature,[22] the sulfate groups in CSA derivatives are located in the preliminary function of alcohol in the glucose units. The FTIR band at 807 cm^{-1} confirmed this observation. The chemical displacement of the resonance line of ^{13}C-NMR to C(6) provided additional information on changes compared to the spectrum of ^{13}C-NMR of cellulose acetates. For instance, a commercial cellulose triacetate shows a resonance line in 63.4 ppm,[23] whereas that of the CSA was at 65 ppm, which suggested that the substitution of the sulfate group in relation to the C(6) of the glucosidic ring displaced the resonance line to the low field.

The spectrum of ^{13}C-NMR could also be used to confirm DS_{acet} obtained by chemical means. When the resonance line of C=O (175 ppm) was expanded and when the region of 170 ppm to 175 ppm was analyzed (Fig. 4), the contribution of three lines of resonance to the carbonyl carbon was clearly perceived. This deployment in three lines was due to the substitution of acetyl groups in C(6), C(3), and C(2), which confirmed the high degree of acetylation obtained by chemical means.

Reduced viscosity

Figure 5 shows the reduced viscosity as a function of the concentration for CSA. The reduced viscosity increased for more diluted concentrations, less than 1 g/L; this indicated the effect of polyelectrolyte due to the presence of ionic sulfate groups, as already reported in the literature.[13]

Application of CSA as an additive in civil construction

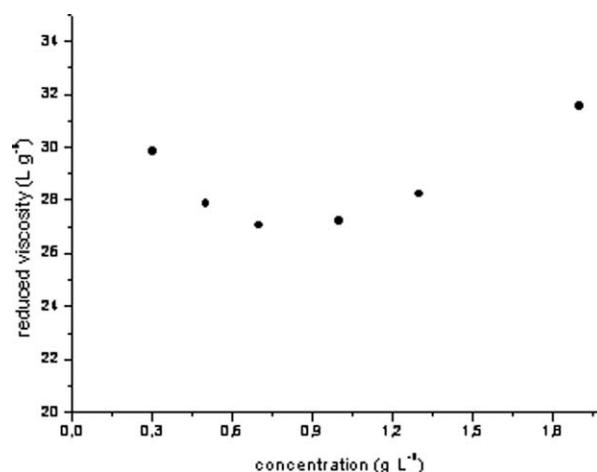Aqueous solutions of CSA were tested as an additive in mortars of Portland cement. The polymer/

Figure 4 ^{13}C-NMR spectrum of the CSA for the region from 170 to 175 ppm.

Figure 5 Determination of the reduced viscosity in water at 25°C of the CSA.
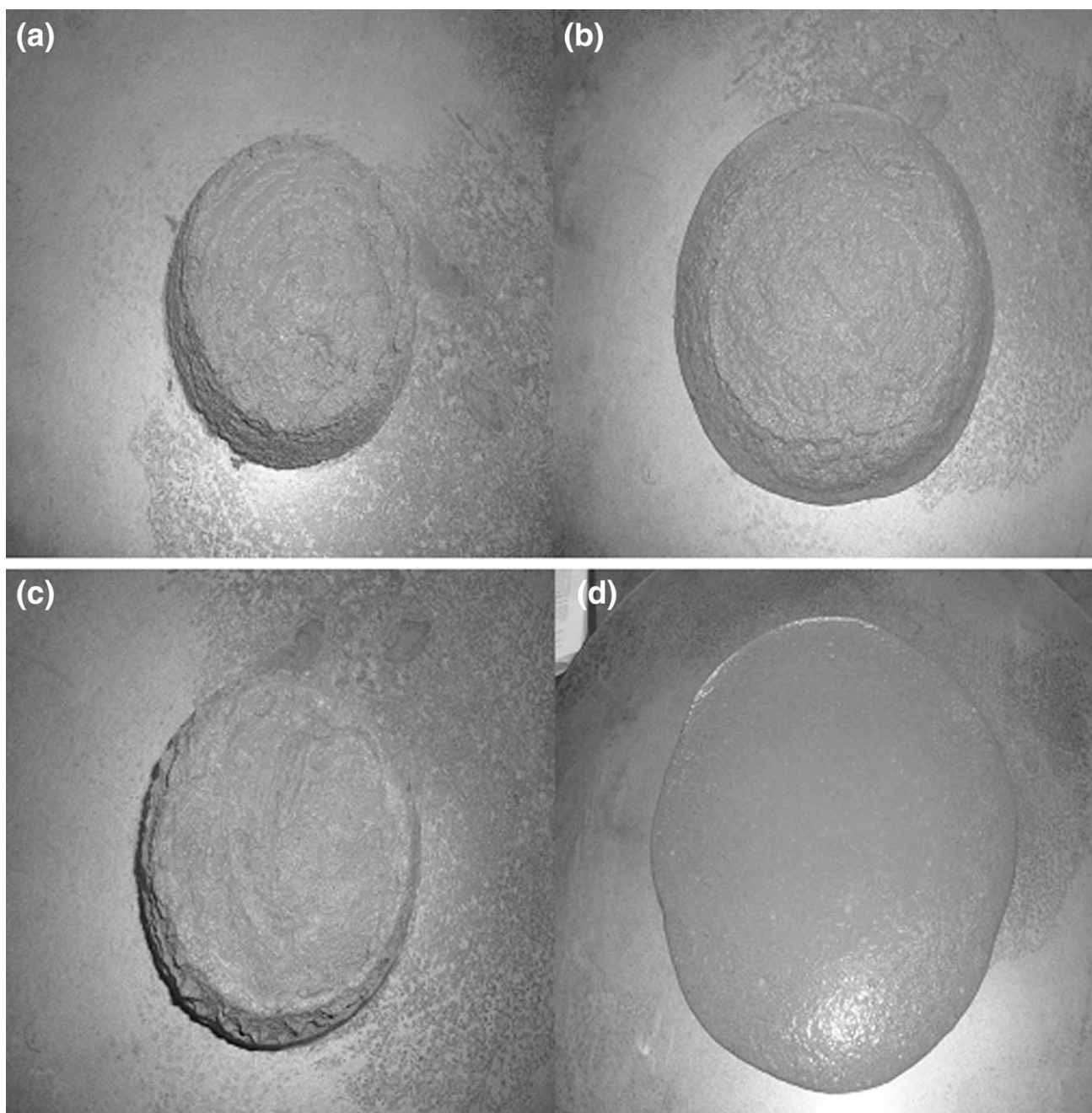
Figure 6 Consistency index test: reference mortar (a) before and (b) after the test and mortar with the polymer (0.6%) (c) before and (d) after the test.

cement ratios evaluated were 0.2, 0.6, and 1% (w/w), which were within the commercially recommended range, that is, between 0.05 and 1.2%.[24] For this purpose, we evaluated the workability by measuring the consistency index, the mechanical strength to compression, and the potential tensile of adhesion strength compared to the reference without the CSA solution.

The 0.2% polymer/cement ratio proved adequate for promoting increased mechanical resistance to compression and workability of the system, without any significant alteration of the potential tensile ad-

hesion strength, whereas the 1% ratio showed improved workability of the system, the potential tensile adhesion strength, but the mechanical resistance to compression was diminished. Considering these ambiguous behaviors, the best CSA dosage considered was 0.6% polymer/cement because this ratio presented satisfactory results in all of the tests performed, which is discussed later.

The value of the consistency index of mortars with CSA and of the reference without the polymer were 163.60 ± 0.50 and 176.30 ± 0.65 mm, respectively. In other words, the addition of the aqueous

solution of CSA resulted in a 13.80% increase in the consistency index compared to the reference. This increase was due to the improved mixture of mortar components because of better system lubrication; this was possibly based on the phenomena of polyelectrolyte adsorption by the cement particles. In this process, the polyelectrolyte with sulfonic groups linked to the chain adsorbs to the cement particles, giving them a negative liquid load. This effect provokes repulsion between the cement particles, favoring their dispersion in water and thus improving the fluidization and workability of the system.[17,24,25,26]

Figure 6 shows the mortars after the consistency index test. A homogeneous aspect and fluid of the mortar was seen, containing CSA.

The mechanical strength to compression of the hardened mortar with polymer at a 0.6% ratio remained virtually constant in relation to the reference. With this dose, there were increases of 3.84% at 7 days of curing and 0.31% at 28 days of curing in the compressive strength of the hardened mortar when compared to the reference.

The adhesive properties of the mortar with polymer were evaluated. The sample containing CSA at a 0.6% ratio and the reference, that is, without polymer, presented a potential tensile adhesion strength of 0.69 ± 0.20 and 0.57 ± 0.19 MPa, respectively. The mortar with the polymer showed an increased potential tensile adhesion strength of 21.05% compared to the reference; this indicated an increased adhesion of mortar to the substrate. This result shows the alteration of the characteristics of the mortar modified with polymer also in the hardened state. These modifications, also observed in the fresh state, were the result of the improvement in the dispersion and homogenization process of the mixture, which frequently leads to an improvement in the mechanical properties.[27] The value of potential tensile adhesion strength for the mortars modified with polymer, 0.69, was higher than the values found for the classification of mortars for tile setting and covering, according to Brazilian norm NBR 15258/2005.[20] Among those, the mortar with the best performance presented a resistance of adherence higher or equal to 0.3 MPa.

The results show that the cellulose extracted from sugarcane bagasse was a reasonable alternative for the production of CSA and that this polymer could be efficiently applied as an additive for mortars in civil construction because it increases the workability and adhesive properties of the mortars without a negative impact on the mechanical strength.

CONCLUSIONS

It was demonstrated that by adaptation of the Chauvelon et al.[13] procedure, it was possible to synthesize

CSA from sugarcane bagasse cellulose. An aqueous solution of CSA 0.6% w/w presented the best polymer/cement ratio to be used as an additive in mortars of Portland (CPV-ARI) cement. For this dosage, a significant increase of 13.8% was observed in the consistency index and an increase of 21.05% was observed in the potential tensile adhesion strength of mortar with polymer compared to the reference. These results show that the CSA produced from sugarcane bagasse cellulose could be effectively used as an additive to improve the workability and adhesive properties of mortars of Portland CPV-ARI cement.

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