



Production, characterization and evaluation of methylcellulose from sugarcane bagasse for applications as viscosity enhancing admixture for cement based material

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ABSTRACT

In previous works, methylcellulose (MC) was prepared from sugarcane bagasse cellulose in heterogeneous medium using dimethyl sulfate (DMS) as etherification agent. MC was produced in a range of degrees of substitution (DS) from 0.70 to 1.40 and the materials showed low water solubility. In this work methylcellulose was prepared with 5 h (MC5h) of reaction with reagent substitution at each hour. MC5h showed a DS of 1.89 ± 0.04 . An aqueous viscous suspension was produced with MC5h for application as viscosity enhancing admixture of cementitious adhesive mortars. It was observed a 40.37% increase on the consistency index (CI) and a 27.70% increase on the Potential Tensile Adhesion Strength. Such characteristics show the potential of this material for the utilization in situations that require good workability, improve viscosity and adhesive properties such as for tile setting in civil engineering.

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1. Introduction

The use of cellulose obtained from sugarcane bagasse for production of cellulose derivatives has been reported in the literature (Gurgel, Freitas, & Gil, 2008; Shaikh, Pandare, Nair, & Varma, 2009; Wang, Li, Xiao, & Wu, 2009). Our research group (GRP-UFU) has been working on demonstrating the viability of chemical recycling of waste sugarcane bagasse and newspaper journal to use as alternative cellulose sources for the production of cellulose acetate and methylcellulose (MC) (Cerqueira, Rodrigues Filho, & Meireles, 2007; Rodrigues Filho et al., 2007, 2008; Vieira et al., 2007).

Commercial MC is prepared by heterogeneous etherification with methyl chloride, its commercial importance can be observed through the wide use of this polymer in several areas that include food, paper, pharmaceutical, building, and painting industries. Chemical modifications of cellulose generally occur under heterogeneous conditions due the low solubility of cellulose in ordinary solvents (Ke, Zhou, & Zhang, 2006).

The degree of substitution (DS) of methylcellulose is defined as the average number of hydroxyl groups substituted by methoxyl groups in the anhydroglucosidic units. Li et al. (2002) showed that samples prepared with DS ranging from 1.4 to 2.0 presented solubility in water and could present a thermally reversible sol–gel

transition in the temperature range from about 50–70 °C. This behavior results from the heterogeneous distribution of substituents along the polymeric chain (Zhou, Xu, Wang, Qin, & Zhang, 2008).

Gel formation can also be observed during MC synthesis in heterogeneous conditions with dimethyl sulfate (DMS). The presence of this gel hampers the DS increase during the synthesis (Mansour, Nagaty, & El-Zawawy, 1994; Vieira et al., 2007) and in this case, the addition of fresh reagents and agitation can break the gel structure and increase the substitution by methyl groups (Rodrigues Filho et al., 2007). That was previously observed for MC with a DS increase from 0.70 to 1.40. In spite of the DS increase, the samples showed low solubility in water (Rodrigues Filho et al., 2007).

Due to the properties shown by the solutions of cellulose ethers in water, these systems have been widely used as admixtures in the production of mortars and concretes in building (Fu & Chung, 1996a, 1996b, 1997; Hou & Chung, 2000). This kind of polymer increases water viscosity, enhancing the cohesion and stability of cement based systems, improving the properties of fresh mixtures (Khayat, 1998). Several commercial products are sold under different trade names, e.g. Methocel[®], trademark of The Dow Chemical Company (Sarkar & Walker, 1995). These products are composed by methylcellulose and hydroxypropylmethylcellulose. Methocel[®] is used in several applications including admixtures for concrete grouts, concrete prepared for underwater repair of marine and hydraulic structures, and adhesive mortars for tile setting. Such

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mixtures reduce the separation of heterogeneous concrete constituents, providing stability in the fresh state. Cellulose ethers provide high viscosity, air incorporation and water retention. These factors are essential to avoid excessive loss of water into the substrate that could cause failures in the mechanism of adhesion. In the rigid state, the evolution of cement hydration and polymer film formation are fundamental processes for improving the quality of cement based materials.

Considering the aspect discussed above, in this work, the time of MC synthesis was modified from 3 to 5 h, maintaining the change of reagents during the synthesis (removal of reagent excess and subsequent addition of fresh reagents at each 1 h of reaction). MC was characterized by Fourier Transformed Infrared Spectroscopy (FTIR) and Differential Scanning Calorimetry (DSC). The application of sugarcane bagasse MC for producing a special mortar was evaluated in fresh state through consistency index (CI) and in the solid state (cure) through Potential Tensile Adhesion Strength of samples after normal cure of 28 days.

2. Experimental

2.1. Synthesis of methylcellulose

Cellulose (1.0 g), extracted as described elsewhere (Oliveira et al., 2007; Rodrigues Filho et al., 2007; Vieira et al., 2007), was mercerized using 20 mL NaOH solution (50%) for 1 h at room temperature. The NaOH excess was removed by filtration and acetone (9.0 mL) was added as solvent. Dimethyl sulfate (3.0 mL) was added drop-wise and the reaction was carried out at 50 °C. After 1 h of reaction, the system was filtered and fresh reagents were added (acetone and DMS), maintaining the same previous proportions. The same procedure was repeated for 5 h of reaction. At the end, the material was neutralized using acetic acid (10%), filtered, washed with acetone and finally oven dried at 50 °C for 6 h. The degree of substitution (DS) was determined as described elsewhere (Saliba, Rodrigues, Pilo-Veloso, & Morais, 2002; Vieira et al., 2007).

2.2. Preparation of methylcellulose suspension

Due to the unusual behavior of MC water solutions, the suspension used in the preparation of the mortar was produced in two stages: (i) mixing of MC in water at 80 °C in order to accelerate the process of water accessibility to the polymer; and (ii) the temperature of the solution was cooled to 4 °C, to increase the solubility of the polymer since in this temperature the water molecules are organized in enclosed structures that surround the hydrophobic groups in the polymeric chains, weakening the association between them (Haque & Morris, 1993; Kobayashi, Huang, & Lodge, 1999; Li et al., 2001). The procedure was summarized below:

A mixture of methylcellulose (6.0 g) with 200 mL water was kept at 80 °C for 1 h under stirring. Next, the suspension was cooled down to room temperature and stored in a refrigerator at 4 °C for 24 h. This amount of polymer was used to keep the ratio polymer/cement at 0.6% (w/w).

2.3. Fourier transformed infrared spectroscopy (FTIR)

The experiments were carried out using Shimadzu IRPrestige-21 equipment with step size of 4 cm⁻¹. Twenty-eight scans were collected using KBr pellets (1:100 (w/w)).

2.4. Differential scanning calorimetry (DSC)

DSC analyses were carried out in a Rheometric Scientific DSC-SP calorimeter, using 2 mg of powder methylcellulose in sealed

aluminum crucibles under nitrogen flow (20 mL/min), and heating ramp from 25 to 300 °C at 20 °C/min.

2.5. Mortar preparation

Commercial Portland cement (CPV-ARI Brazilian norm 7215) and medium sand were used for mortar preparations. Mortars were prepared with polymer-cement ratio (p/c) of 0.6% (w/w), a water-cement ratio (w/c) of 0.70 (w/w) and sand-cement ratio (s/c) of 3.93 (w/w). The polymer was previously suspended in water and added to the cement/sand/water mixture. A reference mortar was prepared without polymer addition.

2.6. Consistency index measurement

The consistency of fresh mortar was evaluated using a slump table, according to Brazilian standard NBR 7215, which is correspondent to ASTM C109.

2.7. Potential Tensile Adhesion Strength essay

The essay for determining the Potential Tensile Adhesion Strength was carried out according to NBR 15258/2005.

3. Results and discussion

3.1. MC synthesis and FTIR spectra

In a previous work, MC was produced using 3 h reaction, without changing the reagents, which presented DS of 1.20 ± 0.07 (Vieira et al., 2007). Later (Rodrigues Filho et al., 2007), with the change of reagents at each hour of reaction, a new MC sample (MC3h) reached DS of 1.40 ± 0.06. In this work, the sample prepared using 5 h reaction, MC5h, showed a DS of 1.89 ± 0.04, 35% higher than MC3h. This higher DS is related to the increase in the reaction time and substitution of the reagents by fresh ones at each hour of reaction. One important aspect that explains the increase of DS in this system is the destabilization of the gel that was formed during the reaction process, since MC shows the unusual property of forming gel during heating (Funami et al., 2007; Haque & Morris, 1993; Sarkar & Walker, 1995). During the chemical reaction the hydroxyl groups are replaced by methoxyl groups leading to an increase in the hydrophobic character of the system and in the hydrophobic interactions, which are favored at high temperatures. There is, in this case, an increase of viscosity of the reaction medium, as well as gel formation since the reaction is carried out at 50 °C and the temperature of sol–gel transition for methylcellulose ranges from 50 to 70 °C (Li et al., 2002). This phenomenon leads to a decrease on the accessibility of the methylant agent and a decrease in the DS value. Stirring and reagent substitution decrease the viscosity of the system improving the accessibility to the methylant agent and an increase in DS was observed for MC5h. In spite of the DS increase, water solubility of MC5h is still limited but higher than that presented by MC3h. It is important to notice that MC5h shows strong interaction with water what leads to a viscous solution.

Fig. 1 shows the FTIR spectra for cellulose, MC3h and MC5h. The chemical modifications of cellulose can be followed by changes in FTIR patterns. The main difference between methylated samples and cellulose is the modification in absorbance intensity of the bands at about 3500 cm⁻¹ assigned to O–H stretching and 2930 cm⁻¹ assigned to C–H stretching. As DS increased (MC5h), a higher ratio between C–H and O–H absorptions bands was observed, as follows: for cellulose $\nu(\text{C–H})/\nu(\text{O–H})$ was 0.32 while

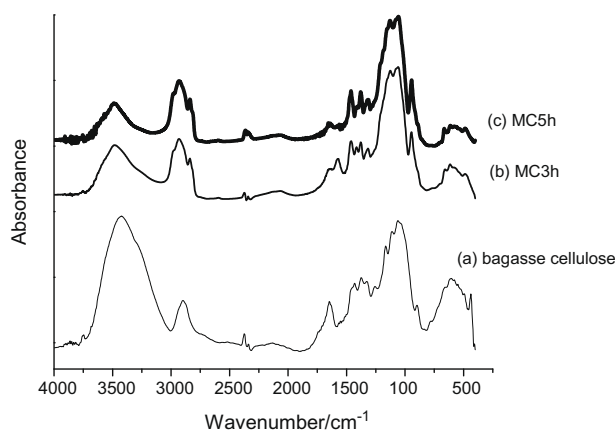


Fig. 1. FTIR Spectra of (a) Sugarcane bagasse cellulose, (b) Methylcellulose (MC3h – methylcellulose 3 h of reaction with reagent changes), and (c) Methylcellulose (MC5h – methylcellulose 5 h of reaction with reagent changes).

for MC3h $\nu(\text{C-H})/\nu(\text{O-H})$ was 1.04 and for MC5h $\nu(\text{C-H})/\nu(\text{O-H})$ was 1.10.

The region between 1550 and 1250 cm^{-1} presents better defined bands than those observed in the spectrum of non-modified cellulose. Some of those bands, such as the band in approximately 1420 cm^{-1} , assigned to the C–H bending has its intensity and shape associated to the increase of order in cellulosic materials. Besides, as the methylation process is carried out in heterogeneous medium the accessibility of the methylating agent to cellulose occurs predominantly in the amorphous regions. Then, part of the crystalline regions is preserved and new interactions can contribute to increase the degree of order of the system from the interaction of the methoxyl groups with the formation of hydrophobic blocks (Mansour et al., 1994; Zhou et al., 2008). Other modifications are observed in the region between 1500 and 500 cm^{-1} , which is particularly sensible to structural changes such as the alteration on the hydrogen bonds, occurred during mercerization and synthesis. This kind of cellulose modification can be evaluated in this region due to the introduction of new functional groups and due to DS increase. Methylcellulose spectrum is clearly modified when compared to unmodified cellulose, particularly regarding the presence of the bands at 1460, 1380, 1320, and 950 cm^{-1} (Zhbakov, 1966). The presence of an intense band at 1100 cm^{-1} assigned to C–O stretching of ether groups and the distinct pattern of this region in relation to unmodified cellulose is an important confirmation of methylation.

One important aspect observed in FTIR spectra is the presence of narrow and well-defined bands. This improvement can be attributed to the presence of ordered regions in MC. These characteristics were observed in DSC experiments as described in the next section.

3.2. DSC thermograms

Fig. 2 shows the DSC thermograms for MC3h (Rodrigues Filho et al., 2007) and MC5h. Both samples showed an endotherm ranging from 85.00 to 135.00 °C attributed to water desorption from the polysaccharide structure. The presence of this endotherm is typical of cellulosic derivatives due to interactions between water and residual hydroxyl groups. The enthalpy of water release for MC3h and MC5h are 72.00 and 44.47 J/g, respectively. The temperatures observed for the peak of water release for MC3h and MC5h are at 130.12 and 86.26 °C, respectively. These results confirm the highest DS obtained with the new methodology presented in this work.

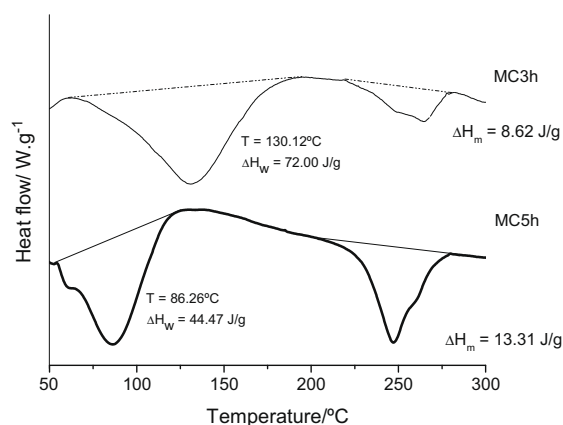


Fig. 2. DSC curves for methylcellulose MC3h and MC5h.

It was also observed in both thermograms an endothermic peak at around 250.00 °C, which can be predominantly related to fusion of crystalline regions of methylcellulose. In a previous work (Rodrigues Filho et al., 2007), the crystallinity of MC3h was confirmed by the crystalline pattern by X-ray diffractometry. Furthermore, a process of thermal decomposition occurs predominantly at 300 °C, what eliminates the possibility of the endothermic process at around 250 °C being predominantly attributed to sample decomposition. The behavior of this endotherm for MC5h confirms that this sample is semicrystalline. Although the values of enthalpy of fusion for both polymers are low, the difference between them can be attributed to an increase of MC5h crystallinity. Due to local associations and low uniformity on the distribution of methyl groups in cellulose, some cellulose crystallinity is preserved (Funami et al., 2007). This heterogeneity can be observed in the formation or preservation of crystalline zones that seem to increase with DS. The amorphous regions are more susceptible to chemical modification than the crystalline. The formation or preservation of some crystalline regions on MC could be responsible for the low solubility of the polymer (MC5h) in water.

3.3. Application of MC5h as viscosity enhancing admixture

The highest concentration that MC5h can be dissolved is 3%. Lower concentrations are not enough for increasing the medium viscosity. Therefore, was produced a solution using 6 g of methylcellulose for 200 mL solution. As the polymer amount for application in civil construction is calculated by the relation weight of polymer/weight of cement, it is obtained the 0.6% ratio, which is inside the range between 0.05% and 1.2%, which is commercially recommended.

In some of the papers of Chung et al. (Fu & Chung, 1996a, 1997; Hou & Chung, 2000), methylcellulose was used as admixture to improve some properties such as adhesion between the cement matrix, carbon fibers, and steel. The evaluated proportions were between 0.2% and 0.8% in relation to the cement weight. The 0.4% dosage showed to be adequate since it promoted an increase in the adhesion, stabilizing the system. In another paper (Fu & Chung, 1996b), methylcellulose effect was evaluated in relation to the mechanical properties such as traction and compression. A gradual increase was observed in the parameters related to the traction as the methylcellulose dosage increased. However, this effect was contrary in relation to the compression parameters.

Considering this ambiguous behavior, the best dosage of methylcellulose used in a system should be that in which it is observed a significant improve in a desired parameter with a negative alteration of little significance in relation to other parameter physically

important in the system, such as the resistance to compression. In this case, the 0.6% dosage was chosen for being in the range (0.4–0.8%) where the best results of application were observed by Fu and Chung (1996b).

MC3h was not used in the tests of mortar preparation due to its limited water solubility. A dispersion of this material do not modify the water viscosity and, therefore, would not accomplish the desired function which was modify the viscosity and consequently retain water in cement mortars.

The aqueous suspension of MC5h, 0.6% (w/w), was used as admixture for producing special mortars with adhesive properties for tile setting. The increase on workability of mortar was evaluated by consistency index (CI).

The CI values for the sample containing MC5h and the reference, i.e. without polymer, were 213.85 ± 0.70 and 152.35 ± 0.95 mm, respectively.

The addition of aqueous suspension of MC5h resulted in a 40.37% increase in CI when compared to the reference. An important aspect to explain the increase in CI is the improvement of the mixture components of the mortar due to better lubrication of the system. The fluidification is gradually modified when the mortar is kept still. In this case, the mixture increases its viscosity and starts to present a cohesive, “tacky” aspect.

Fig. 3 shows the mortars after CI test. It can be observe a homogeneous aspect of mortar with MC5h.

The adhesive properties of the mortar with polymer were evaluated. The sample containing MC5h and the reference, i.e. without polymer presented Potential Tensile Adhesion Strength of 0.83 ± 0.06 and 0.65 ± 0.16 MPa, respectively.

The mortar with polymer (MC5h) showed an increase in the Potential Tensile Adhesion Strength 27.70% higher than the reference. In addition to the changes in the fresh mortar there are changes in the cured state with the improvement of adhesion. That

was expected since commercial cellulose ethers work efficiently as binders in mortars used for tile setting (Jenni, Holzer, Zurbruggen, & Herwegh, 2005). The value of Potential Tensile Adhesion Strength, obtained for the mortar modified with polymer, 0.83 MPa, is in the range of mortars with higher performance found in the classification of mortars for tile setting and covering of walls and ceiling according to ABNT NBR 15258/2005, where the mortar with best performance presents a value of Potential Tensile Adhesion Strength higher or equal 0.30 MPa. Besides, the produced material from sugar cane bagasse cellulose presents a performance similar to commercial adhesive mortar, Super Cimentcola® Quartzolit which presents Potential Tensile Adhesion Strength value of 0.80 MPa.

The results show that the cellulose extracted from sugarcane bagasse is a viable alternative for producing methylcellulose and this polymer can be efficiently applied as an admixture (viscosity enhancing admixture) for mortars in the building industry.

4. Conclusion

The modification of reaction time for production of methylcellulose from sugarcane bagasse cellulose using heterogeneous route and DMS as methylant agent allowed the production of more substituted derivative than the sample produced with 3 h of reaction. Although the MC5h still presents low solubility in water, this property has been improved in comparison to MC3h. The aqueous viscous suspension of MC5h, 0.6% (w/w), was used as admixture in the mortar preparation. Based on the results of tests of CI and Potential Tensile Adhesion Strength, the presence of the MC5h improves the properties of mortars in fresh and cured state showing that the methylcellulose produced from sugarcane bagasse cellulose can be efficiently used as a viscosity-enhancing admixture.

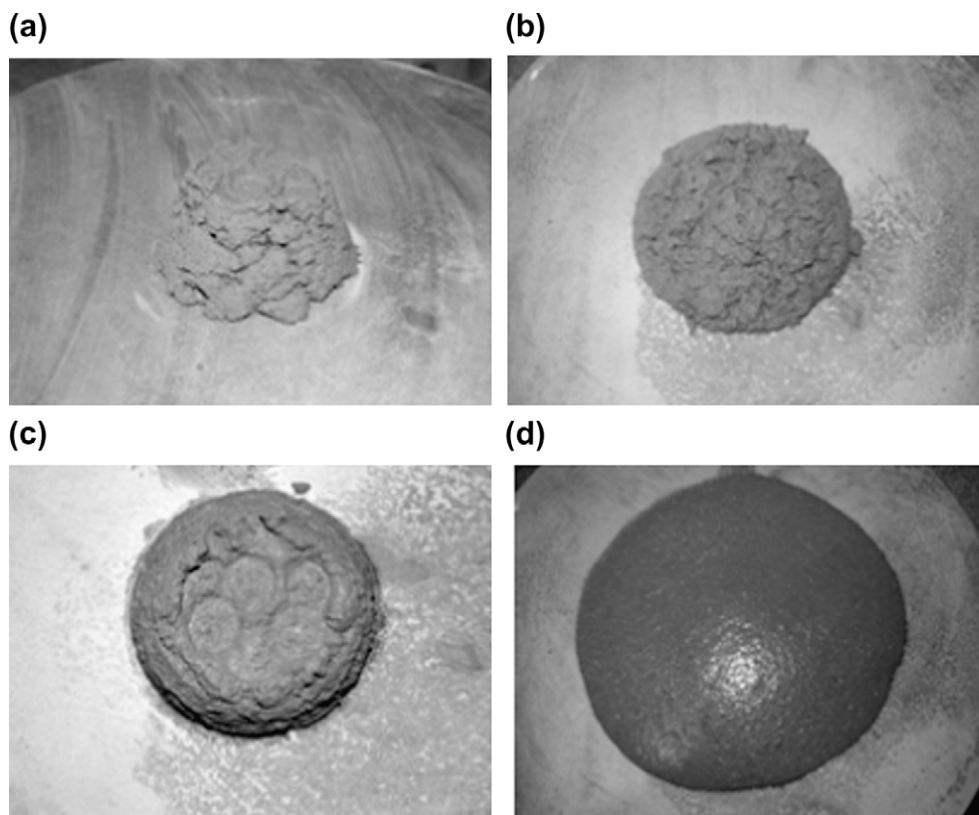


Fig. 3. Slump test: Reference mortar (a) before and (b) after the test and mortar with polymer (MC5h) (c) before and (d) after the test.

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