PREPARATION AND CHARACTERIZATION OF CELLULOSE/ HYDROUS NIOBIUM PHOSPHATE HYBRID

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> In recent years, increasing attention has been directed to the use of renewable resources, particularly of sugarcane bagasse. Considering the abundant availability of such lignocellulosic materials, relatively few attempts have been made regarding their utilization. Studies about properties and morphology, heavy metal adsorption, and membranes preparation have been conduced by this research group in order to use these materials. In this paper, cellulose fibers obtained from sugarcane bagasse were bleached and modified by hydrous niobium phosphate. Hybrids (cellulose/NbOPO₄.nH₂O) were prepared from metallic niobium dissolved in a fluoridric/nitric (10:1) mixture, to which cellulose sugarcane bagasse was added. Afterwards a concentrated orthophosphoric acid (85mL, 85% w/w) was added to precipitate hydrous niobium phosphate particles. This material was characterized by X-ray diffraction (XRD), thermogravimetry (TG/DTG), and differential scanning calorimetry (DSC) analyses, as well as scanning electronic microscopy (SEM) coupled to an energy dispersive spectrophotometer (EDS). Morphological studies of bleached cellulose revealed different sizes and arrangement of cells, showing that NbOPO₄.nH₂O was present in the cellulose structure. Thermal stability of the hybrid was observed up to approximately 200°C, and the cellulose decomposed at 300°C. These data will help finding new uses for these materials.

Keywords: Cellulose; Hydrous niobium phosphate; Sugarcane bagasse

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INTRODUCTION

In recent years, special attention has been given to agro-industrial residues and the re-use of residues in the preparation of new materials, as part of worldwide trends concerning environmental and economic viability. Research in the use of renewable natural resources has been carried out with lignocellulosic materials such as coconut fibres (Brígida et al. 2010; Bilba et al. 2007), sisal (Corradini et al. 2008; Martins et al. 2004), sugarcane bagasse (Paiva et al. 1999; Luz et al. 2007; Mulinari et al. 2009; Corradini et al. 2009), banana (Lui et al. 2009; Guimarães et al. 2010), jute (Saha et al. 2010), bamboo (Rao Mohan et al. 2010), and curauá (Spinacê et al. 2009; Monteiro et al. 2006). Alternative materials such as silica gel (Lazarin and Gushikem 2002), clays (Wang and Wang 2008), and agro-industrial residues have been used in the adsorption materials preparation of effluent treatment (Sene et al. 2002; Jorapur and Rayvanshi 1997; Mulinari et al. 2006), composites (Tita et al. 2002; Nogueira 2004; Klemm et al. 1996), components for the automobile industry, and for civil construction (Sanadi 2002; Young 1997).

Cellulose, as the world's most abundant renewable biopolymer, has attracted much attention because of its outstanding properties (Jin et al. 2007). Cellulose and its derivatives are frequently used in preparing composites due to their important advantages, such as low cost, availability, biodegrability, nonabrasive nature, and easy handling (Toledo et al. 2000; Pandey et al. 2000).

Moreover, the use of sugarcane bagasse residue is desirable economically as well as environmentally (Luz et al. 2007). Cellulose, the main component of this vegetal fiber, is a linear homopolymer made up of anhydro glucose units, which are linked through β (1-4) glycosides, forming a fibrilar structure (Klem et al. 1996; Fengel and Wegner 1989). Lignin is the second most abundant natural polymer. This macromolecule is chemically complex, composed by phenyl propane units connected by various types of linkages forming a branched structure containing diverse functional groups (Corradini et al. 1999; Carvalho et al. 2009). Hemicelluloses are heterogeneous polymers of pentoses (xylose, arabinose), hexoses (mannoses, gluose, galactose), and sugar acids (Saha 2003; Silva et al. 2009).

Sugarcane bagasse is an agro-industrial residue that is quite abundant in Brazil and can be reused or recycled in different applications. Many natural fibers have been used in the construction of automobiles, trucks, and railway cars (Chawla 1998). The residue generation occurs in high proportions in the agro-industry (Paiva and Frollini 1999). Several processes and products have been reported showing the use of sugarcane bagasse as a raw material (Padilha et al. 1995).

Many of the residues generated by the alchohol industries have been used as animal food or for energy generation and the surplus excess can be used in valuable applications, for example in new materials (Rijswijk et al. 2002; Tita et al. 2002; Mullinari et al. 2006; Luz et al. 2007).

Published studies have reported cellulose/metallic oxides, resulting in materials with such applications as immobilization of reagents and the preparation of thin films on the solid matrices surface such as Al₂O₃ (Alfaya 1999), niobium oxides (Campos 1996), zirconium oxide (Rodrigues Filho 1996), antimonium oxide (Toledo 2000), and titanium dioxide nanocomposite (Marques 2006). However, studies using hydrous niobium phosphate are not found. The objective of this paper is to prepare a cellulose/ NbOPO₄.nH₂O hybrid and to characterize it by X-ray diffractometry (XRD), thermo-gravimetry (TG/DTG), differential scanning calorimetry (DSC), and scanning electronic microscopy (SEM) connected to an energy dispersive spectrophometer (EDS).

EXPERIMENTAL

Materials Preparation

Preparation of the bleached cellulose

The bleached cellulose was obtained in the following way: sugarcane bagasse was pretreated with 10% sulfuric acid solution (350 L reactor at 120°C, 10 min), followed by

centrifugation with the purpose of separating the rich pentosans solution. Extracted lignocellulosic fraction was delignified with 1% NaOH solution (350 L reactor at 100°C, 1 h) to obtain a crude pulp. The crude pulp was bleached with acetic acid and sodium chlorite under stirring and heating, followed by filtration under vacuum and exhaustive washing with distilled water. This technique was used to remove residual lignin. Furthermore the bleached cellulose dried in a store at 50 °C, 12 h (Mulinari et al. 2006).

Preparation of the Cellulose/Hydrous Niobium Phosphate Composite

The preparation of Cellulose /Hydrous Niobium Phosphate hybrid material was similar to those used by Pereira (2009) with some modifications. Cellulose/ NbOPO₄.nH₂O was prepared by dissolving 5g metallic niobium in a mixture of fluoridric/nitric acid (50HF/5HNO₃) with a molar ratio of (10:1). The cellulose sugarcane bagasse was added to the solution; then concentrated orthophosphoric acid (85 mL, 85% w/w) was also added. The solution was heated in the steam bath until a crystalline precipitate was formed after approximately 48h. This suspension was treated with 200 mL of 5M nitric acid solution. The solid was washed with 100 mL distilled water and 100 mL ethanol solution. The product was dried at 50°C for 24h.

Characterization of the Materials

The hydrous phosphate niobium, the bleached cellulose, and the hybrid Cell/NbOPO₄.nH₂O were characterized by X-ray diffratometry (XRD), thermogravimetry (TG/DTG), differential scanning calorimetry (DSC) and scanning electron microscopy (SEM) connected to an energy dispersive spectrophotometer (EDS).

X-Ray diffraction (RXD)

The crystallinity of hydrous phosphate niobium, bleached cellulose, and cellulose/ NbOPO₄.nH₂O hybrid were evaluated by X-ray diffraction. X-ray diffractograms were obtained with a Rich Seifert diffractometer model XRD6000. Conditions used were: radiation CuK α , tension of 30 kV, current of 40 mA, and 0.05 (2 θ /5 s) scanning from values of 2 θ it enters 10–70° (2 θ).

Thermal analysis (TG/DTG)

TG/DTG curves were obtained by the use of a Shimadzu thermogravimetric instrument model TGA-50. Thermal behavior of the preparations was obtained by recording the TG/DTG curves in the range 25 to 900°C under conditions of nitrogen atmosphere using weighed samples in the range 5 to 10 mg, at 20°C min⁻¹.

Thermal analysis (DSC)

DSC analysis was performed using a calorimetric instrument DP Union, by the heating of the sample 5mg in temperatures in the range 25 to 550°C under nitrogen atmosphere at a rate of 10°C min⁻¹.

Scanning electron microscopy (SEM)

A JEOL JSM5310 model scanning electron microscope (SEM) was used to observe the modification on cellulose fibres, cellulose bleaching, and hydrous niobium

phosphate. Samples to be observed under the SEM were mounted on conductive adhesive tape, sputter coated with gold, and observed in the SEM using a voltage of 15 kV.

RESULTS AND DISCUSSION

Figure 1 shows the X-ray powder diffraction patterns of the three materials studied in this work, hybrid material (A), bleached cellulose (B), and hydrous niobium phosphate (C), respectively.

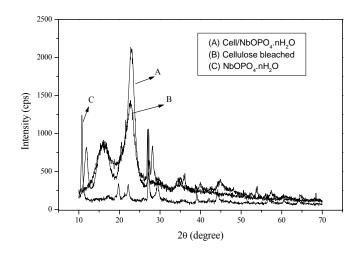


Fig. 1. X-ray diffractogram of bleached cellulose (A), Cell/NbOPO₄.nH₂O hybrid (B), and hydrous niobium phosphate (C)

Many differences between bleached cellulose and hybrid material can be observed in the x-ray diffractogram shown in Fig. 1. It is possible to observe a major diffraction peak for 2θ ranging between 22° and 23° , which corresponds to the cellulose (0 0 2) crystallographic plane. Curve spectrum Fig. 1A, corresponding to hybrid material, shows diffraction peaks at the following 2θ angles: 15.9° and 22.8°.

For bleached cellulose the same peaks were observed at 15.7° and 22.6°, which represent the typical XRD pattern for cellulose fibers (Uddin et al. 2007), in similar form to those reported for others lignocellulosic fibers, which present mainly the cellulose I structure, the common structure observed in plants (Guimarães et al. 2010). It can be observed that when the bleached cellulose was recovered with hydrous niobium phosphate, a gradual increase of hybrid material crystallinity index occurred (Fig 1a) associated to the crystalline character of the hydrous niobium phosphate (Fig. 1c).

According to Mwaikambo and Ansell (2002); Thygesen *et al.* (2005); El-Sakhawy and Hassan (2007); and Rodrigues Filho *et al.* (2007), this method to determine the lignocellulose fiber crystallinity index provides simple and fast information. The crystallinity index (*CI*) was calculated as the ratio of the intensity differences in the peak positions at 18° and 22° according to equation 1 (Rodrigues Filho et al. 2007),

$$CI = \frac{I_{(22)} - I_{(18)}}{I_{(22)}} \times 100 \tag{1}$$

where I_{18} is the intensity at minimum of the crystalline peak ($18^{\circ} < 2\theta < 19^{\circ}$) and I_{22} is the intensity at its maximum ($22^{\circ} < 2\theta < 23^{\circ}$). According to this method, the degree of crystallinity values were calculated (Tab.1). In the literature, the crystallinity values for some of these materials have been reported (Mulinari et al. 2009). The difference among the literature values for the crystallinity of cellulose, and also between the literature values and those reported in this study, can be attributed to the data manipulation used in evaluating the degree of crystallinity.

Table 1. Crystallinity Degree for the Bleached Cellulose and Cell/NbOPO ₄ .nH ₂ O
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Material	Degree Crystallinity		
Bleached Cellulose	56.35		
Cell/ NbOPO4.nH2O	79.82		

Figure 2 shows the TG curves of the, hydrous niobium phosphate (A), bleached cellulose (B), and Cell/NbOPO₄.nH₂O hibrid (C).

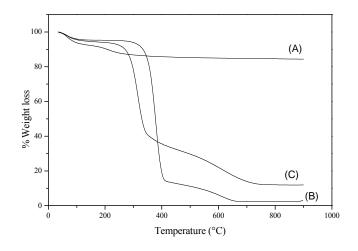


Fig. 2. TG curve: Hydrous niobium phosphate (A), bleached cellulose (B), and Cell/NbOPO₄.nH₂O prepared with boric acid (C)

The TG curve in Fig. 2A for the hydrous niobium phosphate shows a weight loss occurring in two distinct stages within the temperature range of 25°C to 550°C. In the first one, between the temperature of 40°C and 170°C, water molecules weakly connected to the matrix were eliminated (Morán et al. 2008). In the second stage, between 170°C and 550°C, water molecules more strongly connected to the material

were eliminated (Pereira and Da Silva 2009). The TG curve in Fig. 2B for the bleached cellulose shows two decomposition steps (Mulinari et al. 2008).

Figure 2C from the hybrid material exhibits degradation in two steps: the first between 40°C and 200°C, with a temperature of maximum weight loss of 64°, due to the evaporation of water in these sample (Bertoti et al. 2009), and the second loss between 200°C to 500°C with a the temperature of maximum loss equal to of 315°C. The results are indicated in Table 2.

Table 2. Results of the Thermogravimetric Curves (TG) with the Corresponding Temperatures to the Maximum Rate of Mass Loss in (dm) in the Respective Intervals of Temperature (*T*) with Losses of Mass (%) and Residue (*R*)

Material	m (%)	Δ <i>Τ</i> (°C)	d <i>m</i> (°C)	R (%)
NbOPO4.nH2O	7.1	40-170	68	85.3
	7.6	170-550	213	
	4.7	40-200	62	
Cellulose	84	200-500	378	2.8
	8.6	500-800	613	
Cell/ NbOPO₄.nH₂O hybrid	5.7	40-200	64	
	63.2	200-500	315	11.5
	19.6	500-800	460	

From Table 2 it may be observed that the percentage of residue in the hybrid increased with respect to the pureness of the cellulose, as indicated by the presence of inorganic material. In Table 2, it is shown that the presence of inorganic material reduced the stability of hybrid material. Therefore there was a decrease of the temperature (dm) from 378 to 315°C. Comparison of bleached cellulose and hybrid material TG curves shows that the hybrid material had less weight loss in comparison to the cellulose. The remaining 11.5% is attributable to the presence of inorganic material, confirming the presence of hydrous niobium phosphate on the cellulose surface.

Figure 3 shows thermograms of the first scan for hydrous niobium phosphate, cellulose sugarcane bagasse, and hybrid material. Based on this figure, is possible to observe that all thermograms exhibited an endothermic peak located near 120°C, which is attributed to the evaporation of water. Figure 3A shows two endothermic peaks, the first one at 131°C and the second one at 238°C; both are related to the enthalpy of material dehydration, which occurs in two phases. The first dehydration with $\Delta H_{des} = 235 \text{ J.g}^{-1}$, occurred by elimination of the water molecules weakly connected with matrix, and the second dehydration with $\Delta H_{des} = 44 \text{ J.g}^{-1}$ occurred by elimination of those water molecules more strongly connected (Pereira and Da Silva 2009).

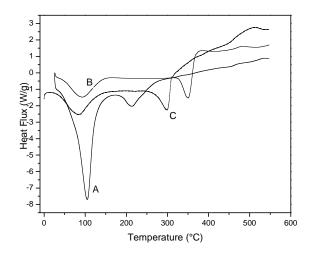
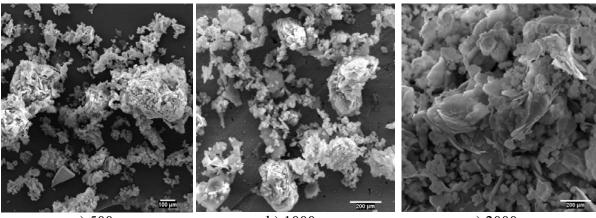


Fig. 3. DSC curve: Hydrous niobium phosphate (A), bleached cellulose (B), and Cell/NbOPO₄.nH₂O prepared with boric acid (C)

From DSC studies of the material (Fig. 3B and 3C), it can be seen that the peaks for the two material samples around 300°C can be related to the decomposition of cellulose, which is in agreement with the weight loss observed in this range in the thermogravimetric analyses of all samples. These findings confirm the results obtained by TG analysis.

In order to investigate the morphology of the obtained samples, comparison between the SEM images of hydrous niobium phosphate, bleached cellulose, and hybrid material are illustrated in Figs. 4 to 6.



a) 500x

b) 1000x

c) 2000x

Fig. 4. SEM of hydrous niobium phosphate backscattered with 500x (a), 1000x (b) and 2000x (c)

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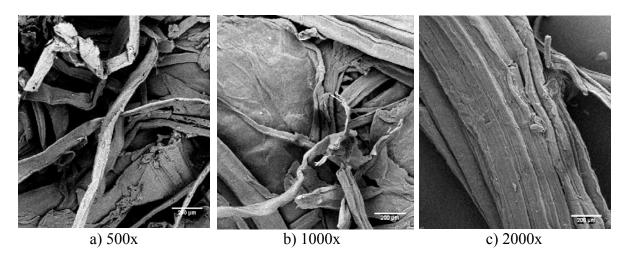
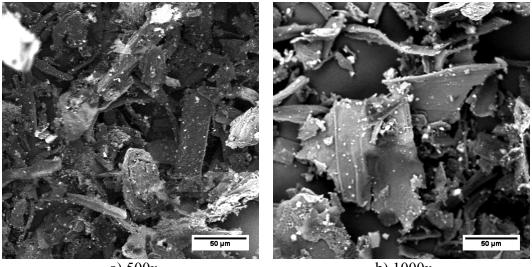


Fig. 5. SEM of bleached cellulose secondary electrons with 500x (a), 1000x (b), and 2000x (c)



a) 500x

b) 1000x

Fig. 6. SEM of cell/NbOPO₄.nH₂O hybrid secondary electrons with 500x (a), 1000x (b)

It can be seen that, as in the case of other natural fibers, all three materials studies here showed variations in their structure, namely, different sizes and shapes. Figures 6 to 8 are SEM images of hydrous niobium phosphate, bleached cellulose, and the cell/NbOPO₄.nH₂O hybrid under various magnifications. It was shown that hydrous niobium phosphate was mainly composed of agglomerated, small porous blades (Pereira and Da Silva 2009). The micrograph of bleached cellulose (Fig.5) shows a great amount of fibers present with flattened forms. At higher magnifications (Fig.5B), the fibers show helical winding of microfibrils attached to each other. The interior of these microfibrils shows a longitudinal array in some cases. Figure 8 shows the hydrous niobium phosphate dispersed on the cellulose fibers surface. It should be noticed, however, that the hydrous niobium phosphate was deposited in non-homogeneous form on the surface of to the cellulose fibers. From the EDS analysis the presence of the elements niobium, phosphorus, and oxygen was confirmed.

CONCLUSIONS

X-ray diffraction patterns of the cellulose and cell/NbOPO₄.nH₂O hybrid are similar to those reported for other lignocellulosic fibers, exhibiting mainly the cellulose I structure with crystallinity index values of 56.4% and 78.8% for cellulose and hybrid material, respectively.

Thermal stability of the material prepared is observed up to 200°C. A lower thermal stability is observed in comparison to bleached cellulose by itself. Indeed, the phosphate niobium precipitation on cellulose surface significantly decreases the thermal quality of material.

Morphological studies of the cell/NbOPO₄.nH₂O hybrid revealed different sizes and changes on the fiber surface. By EDS analysis the presence of the elements niobium, phosphorus, and oxygen in the composite was confirmed. Scanning electron microscopy showed that the hydrous niobium phosphate particles were not homogeneously dispersed on the cellulose surface.

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