Use of Sweet Sorghum Bagasse (*Sorghum bicolor* (L.) Moench) for Cellulose Acetate Synthesis

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The objective of this work was to synthesize cellulose acetate from sorghum bagasse, a promising raw material for the production of chemical inputs, both from a photosynthetic point of view and the maturation speed compared with that of sugarcane. The bagasse was treated with hydrogen peroxide, and then cellulose was isolated using sodium chlorite, acetic acid, and sodium hydroxide. The cellulose was subjected to an acetylation reaction, from which cellulose triacetate was obtained. By means of statistical analysis, it was observed that the conditions that generated the highest solubilization of lignin (62%) and higher yield from cellulose extraction (39.5%) were 60 °C, a 6% peroxide concentration, and 4 h. Cellulose acetate was obtained with a degree of substitution of 3.66 at 25 °C and 24 h. Fourier transform infrared spectroscopy, X-ray diffraction, thermogravimetry, differential thermogravimetry, and differential scanning calorimetry analyses confirmed that the obtained cellulose presented specific characteristics of this material. Also, the reaction of acetylation was confirmed through these techniques.

Keywords: Peroxide hydrogen treatment; Sorghum bagasse; Cellulose; Cellulose acetate

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INTRODUCTION

With increasing environmental emissions and the search for alternative renewable energy sources, many countries are promoting initiatives to make use of these sources a global reality. According to Heguaburu *et al*. (2012) and Candido *et al*. (2017) agricultural supplements have received special attention primarily because they are not used as food sources and because they are free of price fluctuations when compared to agri-food products that are also used for the production of chemicals.

Lignocellulosic biomass is composed mainly of polymeric components, including the carbohydrates cellulose (38% to 50%) and hemicellulose (23% to 32%), in addition to lignin (10% to 25%); lignocellulose has been extensively studied because it is an important raw material for the production of biofuels and chemical inputs; moreover, it has a high availability and is renewable (Perlack *et al*. 2005; Sawatdeenarunat *et al*. 2015; De Bhowmick *et al.* 2018). When this biomass is a residue, such as bagasse, it has possible added advantages because obtaining this raw material then has a low economic cost.

In Brazil, the most used lignocellulosic residual biomass for various purposes is sugarcane bagasse (Cabral *et al*. 2018; Cao *et al*. 2018; David *et al.* 2018). That is because this vegetable sugar is used as a raw material to produce sugar and ethanol by broth fermentation, and this residue is generated because of the high consumption of this biofuel. Sugarcane has not met the market demand because of the short crop and agronomic disadvantages (Machado *et al*. 2017; Alkimim and Clarke 2018).

Compared to sugarcane, a crop traditionally used in Brazil for the production of ethanol, sorghum bagasse is an agronomic and industrial option for this purpose. Sorghum resembles sugarcane in relation to energy production, since it stores sugars (sucrose, glucose, and fructose) directly fermentable in its stems with soluble solids contents ranging from 14 to 22 °Brix. The stems can be harvested from the plant at the same time as the grains, thus increasing the production of ethanol. The sorghum cycle lasts around 90 to 120 days, which allows more than one crop per year, and considering an average yield per crop of 50 $t.ha^{-1}$, it can be concluded that sorghum is a fairly large crop, which offers efficient production of biomass for the production of chemicals (De Nóbile and Nunes 2014; Idris *et al*. 2017). Similar to sugarcane, it is possible to obtain broth from sorghum for ethanol production, which makes it a promising raw material for the production of this biofuel (Castro *et al*. 2017; Appiah-Nkansah *et al.* 2018). However, residual lignocellulosic biomass from this raw material is generated as a process residue.

The sorghum culture, when compared with sugarcane, requires a lower amount of water because it has a good resistance to the climatic factors of a semi-arid region, namely heat and dryness. It is cultivated from seeds and has a more vegetative cycle of short crop yields, which presents a good yield per hectare, cultivation time, and lower harvesting time (3 months to 5 months), as well as offering a great opportunity for better land use (De Nóbile and Nunes 2014; Idris *et al.* 2017). Thus, sorghum saccharine is a promising raw material for the production of chemical inputs, both from a photosynthetic point of view and the maturation speed compared with that of sugarcane.

The production of chemicals from lignocellulosic materials for commercial use depends basically on two initial factors: the lignocellulosic source and pretreatment process chosen for the initial isolation of the polymers from that source. Pretreatment with hydrogen peroxide has been widely used because it has an oxidizing potential that degrades lignin and partially hydrolyzes the carbohydrates present in the structure of plant cell walls, which leaves the structural components of the biomass, such as cellulose, more exposed and therefore available for other chemical processes (Rabelo *et al*. 2014). Because hydrogen peroxide is completely degraded into molecular oxygen and water, use of this compound is an alternative for acid replacement in pretreatment (Yuan *et al*. 2018).

Boonterm *et al.* (2016) studied the extraction of cellulose from rice straw using chemical treatment with sodium hydroxide at different concentrations, Das *et al.* (2016) studied the isolation of rice husk cellulose using K-10/LiOH montmorillonite solution, and bleaching was carried out with 2% hydrogen peroxide. Manzato *et al*. (2017) also used sodium hydroxide, but with a second treatment with sodium hypochlorite. Wang *et al.* (2018) used sulfuric acid and hydrochloric acid in the treatment of residual cotton fabric fibers for cellulose isolation.

Among the chemicals that can be obtained from cellulose, cellulose acetate is one of the most important because it is an ester with wide applicability, such as a matrix for drug release, sensors, and the protection of optical films, as well as a separation membrane in the hemodialysis process (Cerveira *et al.* 2018; Ghorani *et al.* 2018; Ioniţă *et al*. 2018). This ester is derived from a renewable source of biomass and, if it comes from residual biomass, the final cost of this chemical can be reduced when compared with production from wood pulp (Candido and Gonçalves 2016).

In the acetylation reaction for the production of cellulose acetate, reaction between acetic anhydride and the hydroxyl groups, the degree of substitution (DS) of the cellulose affects several factors, such as the material crystallinity, biodegradability potential, and solubility in other solvents (Candido *et al.* 2017). Depending on the DS, cellulose acetate has different industrial applications (Liu *et al.* 2018; Wang *et al*. 2018). Thus, the thermal, physical, chemical, and mechanical properties of cellulose acetate are directly related to the degree of substitution of hydroxyl groups, according to Selih *et al*. (2007). In the cellulose diacetate, two hydroxyls of the polymer chain are substituted by acetyl groups, whereas in the triacetate three hydroxyls are substituted. In view of this context, the present work isolated cellulose from residual lignocellulosic biomass, which was saccharin sorghum bagasse (BSS), and synthesized and characterized cellulose acetate.

EXPERIMENTAL

Raw Material

The raw material used in this work was BSS (*Sorghum bicolor* (L.) Moench, variety IPA-EP17). The stems used in this research were provided by the Agricultural Research Institute of Pernambuco (Vitória de Santo Antão, Brazil). After the juice extraction process, the resulting residue was washed and oven-dried at 60 °C to a constant mass and stored in plastic bags for post-use.

Lignocellulosic Analysis of the Sorghum Bagasse

The methodology used for the lignocellulosic analyses was based on the TAPPI standards cited by Morais *et al*. (2010). The moisture, ash, extractives, lignin, holocellulose, hemicellulose, and α-cellulose contents of the material were characterized.

Cellulose extraction

The process of cellulose extraction from the BSS is shown in Fig. 1.

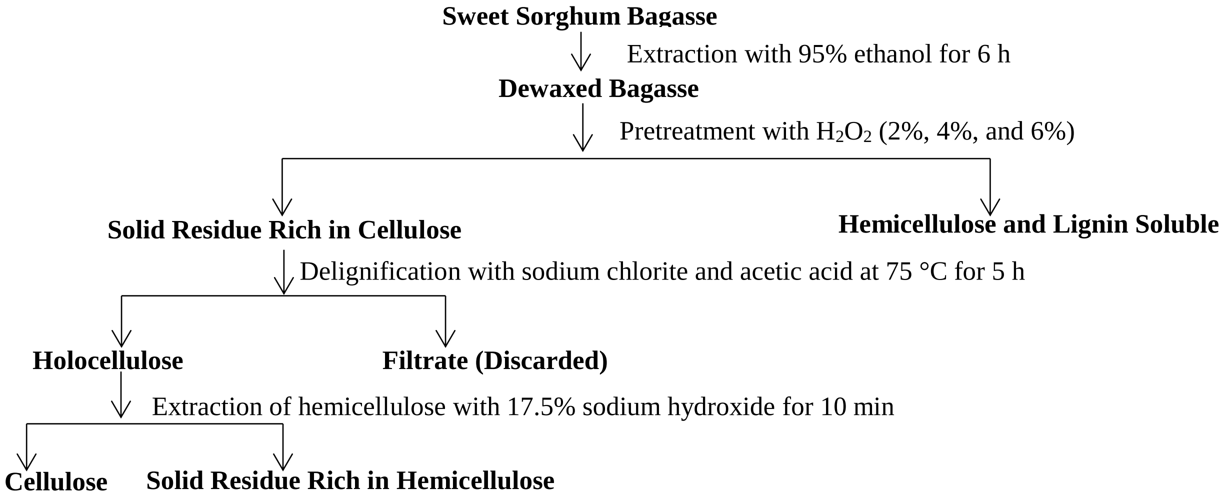

Fig. 1. Representative scheme of cellulose isolation

The dry bagasse was extracted with ethanol (95%) in a Soxhlet extractor for 6 h. The extractive free material was oven-dried for 24 h at 60 °C before pretreatment. Pretreatment with alkaline hydrogen peroxide was performed according to the method described by Xu *et al*. (2006) and modified by Brienzo *et al*. (2009). Ten grams of extractive free material were placed in an Erlenmeyer flask, magnesium sulfate was added at a concentration of 0.25% (w/w), 200 mL of hydrogen peroxide solution were added, the pH was adjusted to 11.6 with the use of 4 M sodium hydroxide, and the medium was stirred at 80 rpm.

The temperature (T), hydrogen peroxide concentration (C), and time (t) were used as independent variables (Brienzo *et al.* 2009) and fixed in bands that could define an experimental matrix for use as a tool in experimental planning to optimize extraction of the lignin fraction. Table 1 presents the real and coded levels of the experimental planning for the optimization of the BSS pretreatment.

Table 1. Real and Coded Levels of the Independent Variables for Cellulose Extraction

Variable	-1	0	+1
Temperature (°C)	20	40	60
H_2O_2 Concentration (%, w/v)	2	4	6
Time (h)	4	10	16

After reaching the reaction time, the material was filtered and the residue rich in cellulose was washed with distilled water until it reached a pH close to that of the washing water. It was then dried in an air circulation oven at 60 °C. The holocellulose was isolated using 3 g of this material, and 120 mL of distilled water were added to a 250-mL Erlenmeyer flask. The Erlenmeyer flask was kept in a water bath at 75 °C and 1.0 mL of acetic acid and 2.5 g of sodium chlorite were added. This same procedure was repeated two more times, with each hour of reaction adding 1.0 mL of acetic acid, after which time the reagents were added and the mixture was stirred for another 3 h. At the end of 5 h, the mixture was cooled to 10 °C, filtered, and washed with distilled water at 5 °C. The material was oven-dried at 60 °C for 6 h and cooled in a desiccator.

After drying, 1.0 g of holocellulose was transferred to a mortar in which approximately 15 mL of 17.5% NaOH solution (w/v) was added with 2 min of contact time between the solution and material. After contact, the material was crushed for 8 min. At this time, 40 mL of distilled water were added to the mortar and the contents were transferred to a funnel with constant stirring. Filtration was then done, wherein the solid material was cellulose and the filtrate was the soluble hemicellulose in the alkaline medium. The cellulose was dried at room temperature on glass plates.

Cellulose acetate synthesis

For synthesis of the cellulose acetate, the methodology was adapted from Meireles *et al.* (2010). Initially, 15 mL of glacial acetic acid were added to 1 g of the cellulose obtained from the bagasse. The medium was shaken at 170 rpm for 30 minutes, taking care that the temperature of the reaction medium remained at 25 °C. All experiments were performed in triplicate.

Thereafter, a solution containing 0.1 mL of concentrated H_2SO_4 in 6.6 mL of glacial acetic acid was added and stirred for 15 min under the same conditions. The supernatant was removed, and 15 mL of acetic anhydride were added, stirred, and returned to the starting flask with the material. The solution was stirred for another 30 min and allowed to stand. At the end of the time specified for each experiment and defined in the experimental planning matrix, distilled water was added to the reaction medium until a precipitate no longer formed. The mixture was vacuum filtered and washed with distilled water to remove the residual acid. The material was oven-dried at 45 °C for approximately 6 h. The time and temperature used were based on the work of Meireles *et al.* (2010) and were adapted so that the variables were fixed for the definition of an experimental matrix containing these variables. The response variable was the DS. Table 2 presents the real and coded levels of the experimental planning for cellulose acetate synthesis.

Table 2. Real and Coded Levels of the Independent Variables for Cellulose Acetate Synthesis

Variable	-1	0	+1
Temperature (°C)	25	40	55
Time (h)	8	16	24

Determination of the degree of substitution of the cellulose acetate

The DS determination was performed for all of the samples acetylated by a saponification reaction, according to the procedure described by Filho *et al.* (2008).

In this study, 5.0 mL of 0.25 mol/L sodium hydroxide and 5 mL of ethanol were added to 0.1 g of cellulose acetate, and the mixture was allowed to stand. After 24 h, 10 mL of 0.25 mol/L hydrochloric acid were added and the solution was allowed to stand for another 30 min. Then, the solution was titrated with sodium hydroxide using a phenolphthalein indicator. This procedure was performed in triplicate.

The percentage of acetyl groups that were substituted in the cellulosic chain was calculated according to Eq. 1,

$$AG\ (\%) = \frac{[(V_{\mathrm{bi}} + V_{\mathrm{bt}})\mu_{\mathrm{b}} - (V_{\mathrm{a}}\mu_{\mathrm{a}})]M}{m_{\mathrm{ac}}}\ x\ 100\% \tag{1}$$

where AG is the percentage of acetyl groups (%), V_{bi} is the volume of NaOH added to the system (L), V_{bt} is the volume of NaOH spent in titration (L), μ_{b} is the NaOH concentration (mol/L), V_{a} is the volume of HCl added to the system (L), μ_{a} is the HCl concentration (mol/L), M is the molar weight of the acetyl group (43 gmol/L), and m_{ac} is the weight of the cellulose acetate sample (g).

The determination of the DS by chemical means was based on the determination of the acetyl groups that were substituted in the cellulosic chain. Therefore, the DS was determined from the AG value with Eq. 2 (Appaw *et al*. 2007).

$$DS = \frac{3.86\ x\ AG\ (\%)}{102.40 - AG\ (\%)} \tag{2}$$

Analytics

X-ray diffraction

Diffractograms were obtained using X-ray diffraction (XRD) (D8 Advance, Bruker, Karlsruhe, Germany). The conditions of the analyses were room temperature (27 °C), Cu Kα radiation (1.5418 Å), a voltage of 40 kV, current of 30 mA, temperature range of 5 °C to 50 °C, and speed of 0.2 °C/s.

Fourier transform infrared spectroscopy

The mean infrared spectra were obtained using Fourier transform infrared (FTIR) spectroscopy (Vertex-70, Bruker, MA, USA). The samples were processed into KBr pellets with 64 scans in the region of 400 cm^{-1} to 4000 cm^{-1} with a 4 cm^{-1} ratio resolution.

Differential scanning calorimetry and thermogravimetry analysis

Thermogravimetric (TG) curves were obtained with a Perkin Elmer STA 6000 apparatus (Perkin Elmer, Waltham, USA). The differential scanning calorimetry (DSC) curves were obtained with a thermogravimetric modulus (Q600, TA-Instruments, New Castle, USA). Both measurements were performed under a nitrogen flow of 20 mL/min at a heating rate of 10 °C/min to 600 °C in an alumina crucible with a sample mass of approximately 4.0 mg.

RESULTS AND DISCUSSION

Compositional Analysis of the *in natura* Sorghum Bagasse

The lignocellulosic composition of the BSS used in this work was 39.59% ± 0.27% α-cellulose, 32.05% ± 0.53% hemicellulose, 20.36% ± 0.35% lignin, and 2.17% ± 0.05% ash (Table 3). These values were similar to those found in the literature for other BSS varieties (Idris *et al*. 2017; Mishra *et al*. 2017; Yue *et al*. 2017). The cellulose and hemicellulose were the major components of the cell wall structure of the BSS, and totaled approximately 71.6% of the dry mass. Within the context of biorefinery, this composition makes BSS an interesting raw material for the production of bioproducts.

The lignin content found (20.4%) suggested the need for a treatment step aimed at solubilization for the cellulosic and hemicellulosic fractions to become more susceptible to later stages. The ash content (2.17%) was related to the amount of minerals and inorganic matter (calcium, potassium, magnesium, and phosphorus) that can be used as sources of nutrients for various processes (Jamari and Howse 2012).

When compared with sugarcane bagasse, it was observed that BSS had cellulose, hemicellulose, and ash contents lower than those of the residue (36.7% α-cellulose, 26.1% hemicellulose, 24.4% lignin, and 1.3% ash) (Golbaghi *et al*. 2017). Compared with wood pulp and the basic material used for the production of cellulose acetate in the industry, the cellulose content for coniferous wood and hardwoods was higher than that of the BSS, at 42% ± 2% and 45% ± 2%, respectively, but these values were not that different (Iranmahboob *et al*. 2002). In this sense, because it presented a lignocellulosic composition similar to that of the mentioned woods, BSS residue may be an alternative source for the production of cellulose acetate.

Sorghum Bagasse Pretreatment

For each treatment performed, lignocellulosic characterization of the solid residue was also performed to observe if the treatment used was able to solubilize the hemicellulose and lignin, as well as to determine the cellulose content of the residue. The results are shown in Table 3.

Table 3. Composition of the *in natura* BSS and after Treatment with Alkaline Hydrogen Peroxide

Exp.	*T* (°C)	H_2O_2 (%, w/v)	*t* (h)	Cellulose (%)	Hemicellulose (%)	Lignin (%)
in natura	-	-	-	39.59 ± 0.27	32.05 ± 0.53	20.36 ± 0.35
1	-1 (20)	-1 (2)	-1 (4)	45.63 ± 0.75	31.69 ± 1.17	18.34 ± 0.11
2	+1 (60)	-1 (2)	-1 (4)	50.20 ± 1.31	31.78 ± 0.02	11.65 ± 0.10
3	-1 (20)	+1 (6)	-1 (4)	53.50 ± 0.71	26.08 ± 0.72	17.43 ± 0.35
4	+1 (60)	+1 (6)	-1 (4)	61.11 ± 1.12	21.33 ± 0.65	7.74 ± 0.33
5	-1 (20)	-1 (2)	+1 (16)	51.23 ± 1.02	30.98 ± 1.10	12.34 ± 0.03
6	+1 (60)	-1 (2)	+1 (16)	56.18 ± 0.51	24.95 ± 1.50	11.89 ± 0.01
7	-1 (20)	+1 (6)	+1 (16)	52.33 ± 0.39	27.01 ± 0.41	13.77 ± 0.10
8	+1 (60)	+1 (6)	+1 (16)	55.63 ± 0.32	25.89 ± 1.47	11.77 ± 0.63
9	0 (40)	0 (4)	0 (10)	51.63 ± 1.26	28.58 ± 2.23	14.54± 0.38
10	0 (40)	0 (4)	0 (10)	52.02 ± 1.60	28.55 ± 0.10	14.99 ± 0.35
11	0 (40)	0 (4)	0 (10)	53.55 ± 0.90	27.18 ± 1.02	15.67 ± 0.65

Values in parentheses are the real values for the variables

The cellulose content found in the pretreatment solid residues ranged from 45.6% to 61.1%, and the maximum percentage of cellulose found was at 60 °C with a 6% hydrogen peroxide concentration for 4 h. At the same conditions, there was solubilization of hemicellulose from 32.0% (before pretreatment) to 21.3% (after pretreatment), which corresponded to a total solubilization of 33.4%. Lignin was also removed from the material and went from 20.4% to 7.7%, which corresponded to a 61.98% lignin removal. The same trend was observed by Rabelo *et al.* (2011), but with higher values, when studying the pretreatment of sugarcane bagasse with a hydrogen peroxide concentration of 7% and temperature of 50 °C. The cited authors achieved a 96% increase in the cellulose, hemicellulose solubilization of 46.2%, and a 73% lignin removal rate. Correia *et al.* (2013), when studying hydrogen peroxide pretreatment of cashew bagasse at a concentration of 4.3% and 35 °C, obtained a 60.5% increase in the cellulose content, 44% lignin removal, and there was no remarkable change in the hemicellulose content.

In the present study, the lignin removal percentage ranged from 9.9% to 62.0%, according to the conditions in the experimental design. Figure 2 displays a Pareto chart, which shows the effects of the independent variables and their combinations on the lignin removal percentage. The intensity of each effect can be seen, which is represented by the length of each bar. The vertical dashed line represents a *p*-value of 0.05, which corresponds to a 95% confidence level.

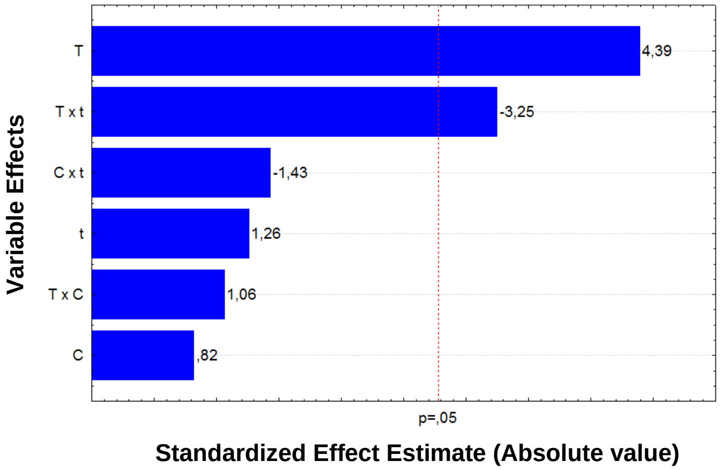

Fig. 2. Pareto chart of the variable effects on the percentage of lignin removal in the pretreatment of the BSS with alkaline hydrogen peroxide

Figure 2 shows that at the 95% confidence level, the temperature variable and the interaction of the temperature and time were statistically significant. With the values of the statistically significant regression coefficients, the lignin removal percentage can be estimated with Eq. 3 (linear regression model of the experimental data-coded model).

$$Removed\ Lignin\ (\%) = 33.18 + 11.56T + 3.31t + 2.79TC - 8.55Tt - 3.76Ct \tag{3}$$

An analysis of variance (ANOVA) was then performed with the parameterized coefficients to verify if the coded mathematical model was statistically significant. The data is shown in Table 4.

Table 4. ANOVA for the Lignin Removal Percentage in the Pretreatment of the BSS with Alkaline Hydrogen Peroxide

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Squares	*F* Ratio
Regression	1917.672	5	383.53	4.02
Residual	259.330	5	1.65	
Total	2177.002	10		
R^2 (%)	88.09		$F_{calculated}$ = 20.32	$F_{tabulated}$ = 5.05

From the data presented in Table 4, it was seen that the planning was statistically significant at the 95% confidence level because the $F_{calculated}$ was superior to the $F_{tabulated}$, with a *F* ratio of 4.02 (Rodrigues and Iemma 2014).

Figure 3 shows the response surface for the lignin removal percentage using the Eq. 3 model with the lignin removal percentage in the pretreatment as the dependent variable, while maintaining the concentration at the highest value of 6% (level = +1).

Figure 3a shows that when pretreatment was performed at the highest concentration level of 6% hydrogen peroxide, a reaction temperature of 60 °C, and a lower time of 4 h, it was possible to reach the maximum lignin removal percentage of approximately 62 %. Figure 3b shows that the percentage of cellulose reached its maximum within the range studied (61.11% ± 1.12%, experiment 4) under the same conditions of the lignin removal percentage.

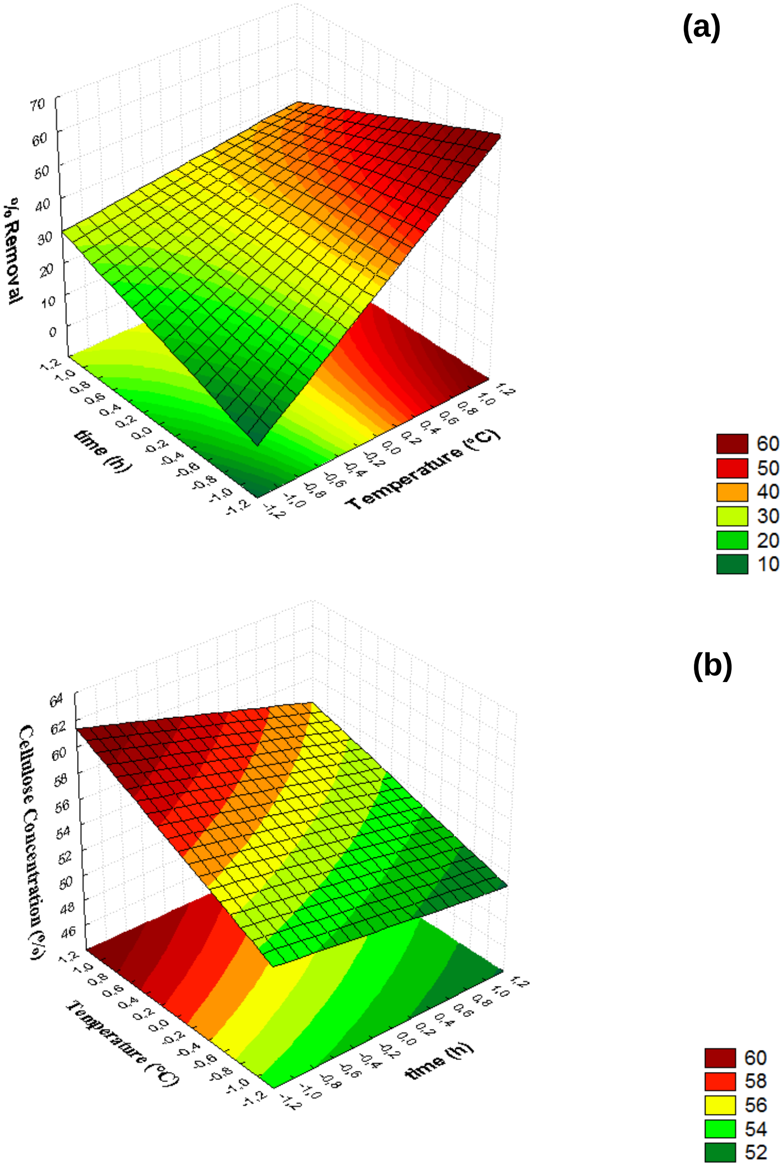

Fig. 3. Response surface for the lignin removal percentage (a) and cellulose concentration percentage (b) as a function of the temperature and time, with the concentration held at the +1 level (6%)

Cellulose Extraction

Using the solid residue resulting from the pretreatment that presented the highest cellulose percentage, it was possible to isolate the carbohydrate. Thus, the yield obtained, based on the initial dry mass of the material, was 39.5%. Regarding the efficiency of the pulp cellulose recovery process, it was verified that this was the maximum value. This result showed that the pretreatment used for delignification was actually effective and that even cellulose obtained from two consecutive treatments with sodium chlorite and sodium hydroxide did not show a significant loss. Sun *et al*. (2004) studied the isolation of cellulose from sugarcane bagasse using sequential treatments of hydrogen peroxide and sodium hydroxide, and achieved a yield of 45.9%.

Cellulose Acetate Synthesis

Table 5 shows the results obtained for the acetyl groups percentage, as well as the DS of the cellulose acetate derived from the BSS according to each experiment.

Table 5. Mean Values Obtained Experimentally for the DS from the BSS

Experiment	*T* (°C)	*t* (h)	Acetyl Groups (%)	DS
1	25 (-1)	8 (-1)	22.2	1.1
2	55 (+1)	8 (-1)	35.0	2.0
3	25 (-1)	24 (+1)	47.6	2.8
4	55 (+1)	24 (+1)	46.4	2.9
5	40 (0)	16 (0)	40.3	2.5
6	40 (0)	16 (0)	37.2	2.2
7	40(0)	16 (0)	41.0	2.6

When analyzing the results in Table 5 for the DS, it was observed that experiments 5, 6, and 7 were classified as cellulose diacetate because they presented a DS between 2.2 and 2.7, and experiments 3 and 4 were classified as cellulose triacetate because they had a DS greater than 2.8 (Heinze and Liebert 2001; Šelih *et al*. 2007; Candido *et al.* 2017; Liang *et al*. 2018).

According to Cerqueira *et al*. (2010), acetates frequently used in practice are diacetates and triacetates. Knowing that the solvents in which these substances are soluble according to the DS, this classification parameter makes it possible to choose the best solvent to be used. Triacetates are soluble in a wide range of solvents.

By analyzing the data, it was observed that the higher DS values were obtained in the experiments with longer reaction times, a fact that was also observed by da Silva (2014), who synthesized cellulose acetate from sugarcane bagasse and produced cellulose triacetate with a DS of 2.84 after a reaction time of 24 h at 50 °C. Candido *et al*. (2017) obtained cellulose acetate with a DS of 2.72 from sugarcane straw with a reaction time of 24 h.

The influence of the temperature and reaction time on obtaining cellulose acetate with the highest DS was statistically analyzed, and Fig. 4 shows the effects of the independent variables and their combinations on the DS at a 95% confidence level.

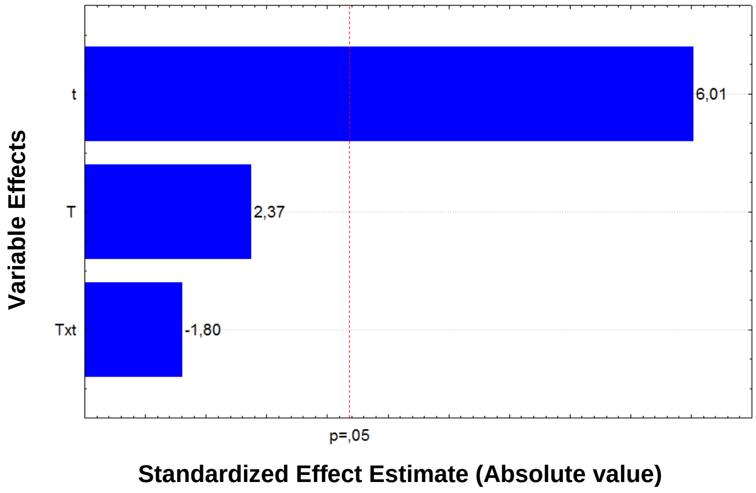

Fig. 4. Pareto chart for the cellulose acetate DS from the BSS where *t* represents time, *T* temperature and *T*x *t* the interaction between temperature and time.

By analyzing the Pareto chart, it was determined that only the time was statistically significant at a 95% confidence level. With the statistically significant regression coefficients, it was possible to estimate the DS with Eq. 4, which represents the linear model.

$$DS = 2.31 + 0.27.T + 0.68t - 0.20t.T \qquad (4)$$

The ANOVA (Table 6) with the parameterized coefficients showed that $F_{\text{calculated}}$ was superior to $F_{\text{tabulated}}$, with a *F* ratio higher than 1. The model was statistically significant at a 95% confidence level (Rodrigues and Iemma 2014).

Table 6. ANOVA for the DS

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Squares	*F* Ratio
Regression	2.3366	2	1.1683	4.33
Residual	0.1557	4	0.0389	
Total	2.4923	6		
R^2 (%)	94,0		$F_{\text{calculated}}$ = 30.03	$F_{\text{tabulated}}$ = 6.94

Figure 5 shows the response surface for the DS as a function of the reaction time and temperature. By analyzing the response surface, it was observed that increasing the reaction time and decreasing the temperature resulted in the maximum DS for cellulose acetate.

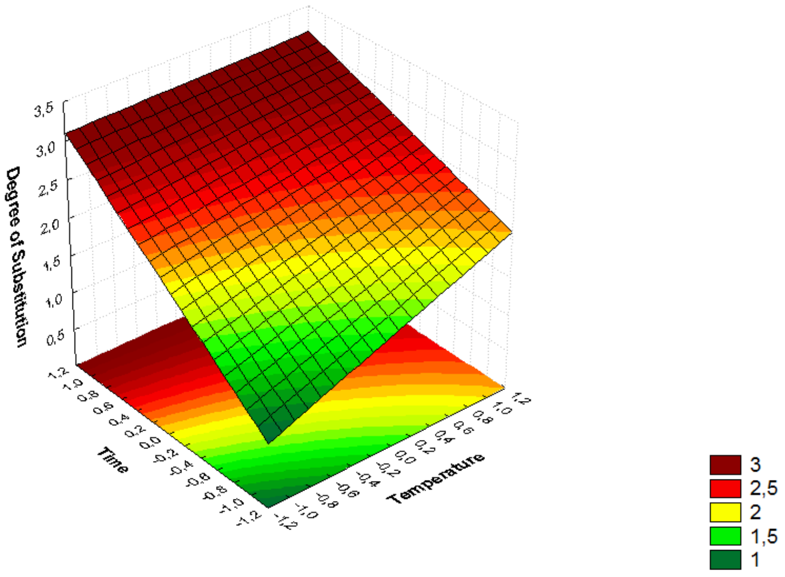

Fig. 5. Response surface described by Eq. 4

Characterization of the *in natura* Sorghum Bagasse, Treated Bagasse, Cellulose, and Cellulose Acetate

X-ray diffraction

The XRD analysis, presented as diffractograms in Fig. 6, was performed on samples of *in natura* BSS, treated bagasse, cellulose, and cellulose acetate.

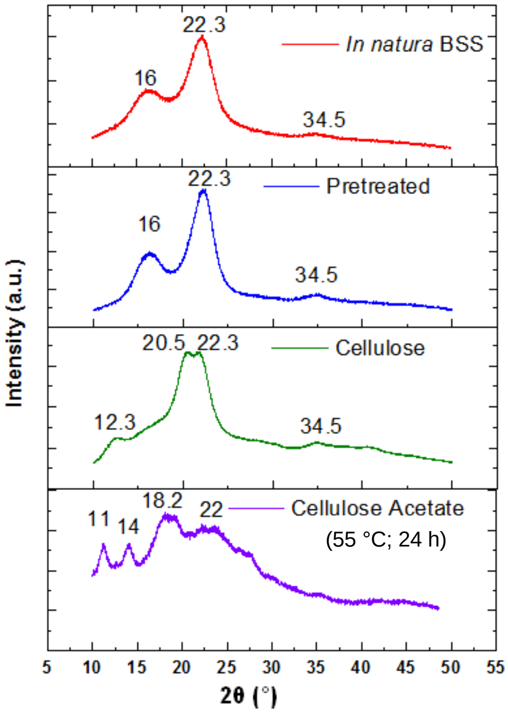

Fig. 6. XRD patterns of the *in natura* BSS, treated bagasse, cellulose, and cellulose acetate

When analyzing the diffractograms in Fig. 6, three reflections at the 2θ values of 16°, 22.3°, and 34.5° in the BSS and treated BSS were observed and attributed to partially crystalline materials containing cellulose. The crystalline structure was attributed to the interactions between the hydrogen bonds and Van der Waals forces between the molecules (Yabushita *et al*. 2014).

From the cellulose diffractogram, the cellulose had a more crystalline reflection at a 2θ value of 22.3° that was broader and more defined than that of the BSS and pretreated bagasse, which showed that hemicellulose and lignin were removed, and the cellulose presented a crystalline phase. The same behavior was observed by Owolabi *et al.* (2017), who studied the effect of pretreatment on obtaining cellulose from palm leaves, as well as by da Silva (2014), who studied the production of cellulose from sugarcane bagasse.

When analyzing the cellulose acetate diffractogram, four reflections at 2θ values of 11°, 14°, 18.2°, and 22° were observed. These reflections were characteristic of cellulose acetate (Shaikh *et al*. 2009). The first two peaks were attributed to cellulose disorders when acetylated, and the peaks between 18° and 22° were attributed to triacetates (Fan *et al.* 2013), which corroborated the DS data obtained for the cellulose acetate. The observed modification next to the reflection at 22° was identified as the crystallinity reduction of the material when compared with the diffractograms of the *in natura* BSS, treated BSS, and cellulose (Shaikh *et al*. 2009; Fan *et al*. 2013). This fact was closely related to the presence of acetyl groups in the structure, which caused disorganization of the repeated units of ordered arrangements that diffracted the crystalline regions.

Fourier transform infrared spectroscopy

Figure 7 shows the mean FTIR spectra of the *in natura* BSS, treated bagasse, cellulose, and cellulose acetate. Figure 4 shows that there were similarities in the band regions at 3340 cm^{-1}, 2900 cm^{-1}, 2340 cm^{-1}, 1632 cm^{-1}, and 1050 cm^{-1}, which indicated that the pretreatment did not compromise the configuration of the cellulose molecule because the cellulose sample was similar to the *in natura* BSS (Owolabi *et al.* 2017).

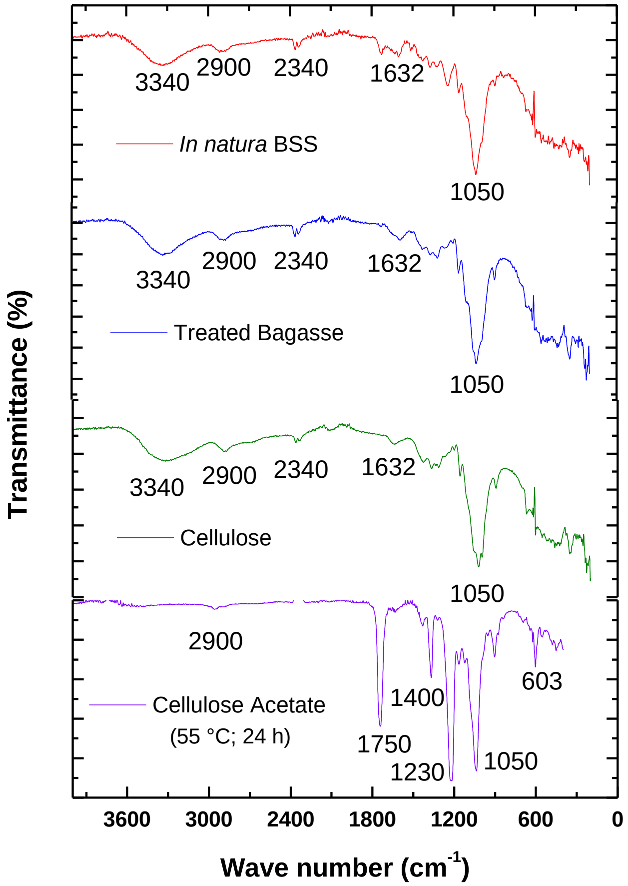

Fig. 7. FTIR spectra of the *in natura* BSS, treated bagasse, cellulose, and cellulose acetate

According to the spectra, characteristic cellulose and lignin bands were observed in the samples at 3340 cm^{-1}, except for cellulose acetate, which was attributed to intra and intermolecular hydrogen bonds (O-H cellulose stretching). The bands corresponding to 2900 cm^{-1} and 1632 cm^{-1} present in the *in natura* BSS, treated BSS, and cellulose samples corresponded to C-H stretching in the methyl and methylene groups and to C=O, respectively. These bands were characteristic of lignin. The bands at 1750 cm^{-1}, corresponding to the carbonyl group present in the cellulose acetate, and 1230 cm^{-1} were attributed to CO stretching in the acetate. The band at 603 cm^{-1} confirmed the presence of CO absorption bands from the acetyl groups (Malheiro 2014).

The bands at approximately 1050 cm^{-1} were characteristic of C-O-C bonds, which represented the cellulose ring-like pyranose vibration. They increased in intensity when the *in natura* bagasse and cellulose acetate were compared, which indicated purification of the samples. Vinodhini *et al.* (2017) found similar FTIR profiles for cellulose acetates, and so did Carvalho (2009), who studied cellulose acetate extraction from sugarcane bagasse.

Thermogravimetric analysis and differential scanning calorimetry

Figure 8 shows the mass loss (TG) curves and its first derivative (DTG) for the *in natura* BSS, treated bagasse, cellulose, and cellulose acetate obtained from the sorghum.

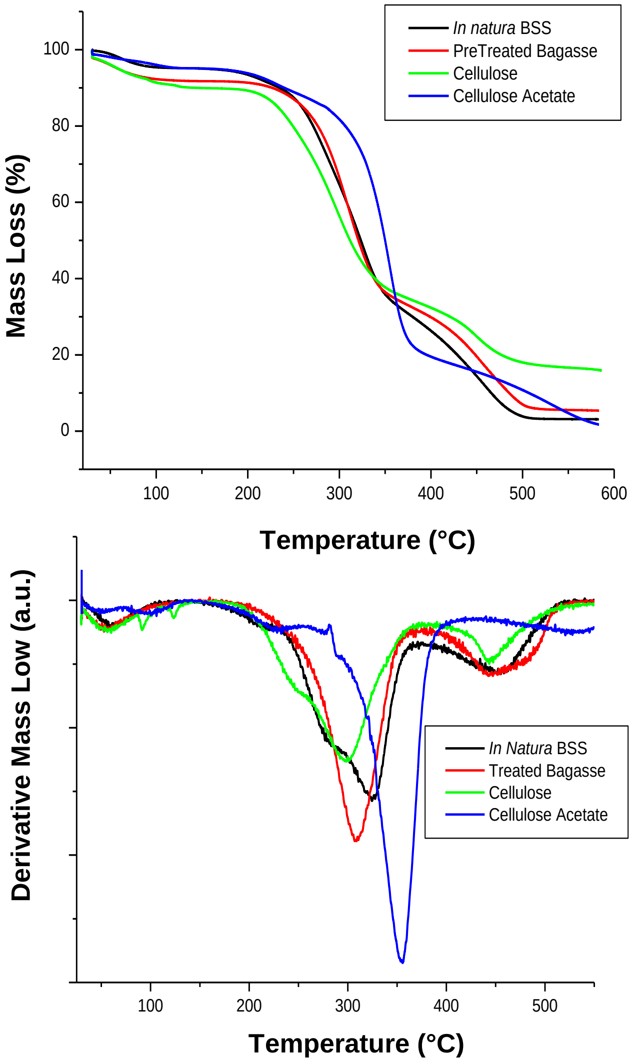

Fig. 8. TG (a) and DTG (b) of the *in natura* BSS, treated bagasse, cellulose, and cellulose acetate

When analyzing the TG/DTG curves in Fig. 8 of the *in natura* bagasse, it was observed that there were four stages of mass loss. In the first stage, there was a mass loss of 4.8% that was from the evaporation of water from the material at temperatures below 135 °C. The second stage occurred from 135 °C to 287 °C and was attributed to hemicellulose degradation (Shen *et al*. 2010), with a mass loss of 24.0%. The third stage occurred from 287 °C to 350 °C and was related to cellulose degradation (Vamvuka *et al*. 2003). Finally, the fourth stage was attributed to the decomposition of cellulose and lignin, with a mass loss 31.7% from 350 °C to 581 °C. There was a peak at 454.5 °C.

When analyzing the TG/DTG curves of the treated bagasse, three main stages were observed, in which the first stage of mass loss occurred from 30 °C to 154 °C and was characteristic of the dehydration of the material, with a mass loss of 7.8%. The second stage of mass loss occurred from 154 °C to 341 °C, and occurred because of the decomposition of hemicellulose and cellulose, with a mass loss of 53.1%. Finally, the third stage was the decomposition of cellulose and lignin, and it occurred from 341 °C to 583 °C, with a 33.1% mass loss. The same number of stages and similar mass loss values were observed when comparing the data with that from Owolabi *et al.* (2017), who studied the delignification of palm leaves using sodium hydroxide at concentrations of 1.5% to 5%.

With the TG/DTG cellulose curves, it was possible to verify the presence of three main stages. The first stage (< 120 °C) was attributed to water loss, whereas the second (120 °C to 383°C) and third stages (383 °C to 583°C) were because of the decomposition of cellulose polymer chains and the decomposition of cellulose and lignin, with mass losses of 56.1% and 17.8%, respectively.

The TG curve of the cellulose acetate exhibited a profile similar to that found for cellulose isolated from BSS. However, it was possible to infer that the decomposition of this acetate occurred in only one stage, from 154 °C to 366 °C with a mass loss of 67.0%. Candido *et al*. (2017) reported a similar temperature range and similar mass losses for acetate from sugarcane bagasse. A breakdown of the glycosidic bonds occurred and the acetate group volatilized to acetic acid.

Differential scanning calorimetric analysis of the *in natura* bagasse, treated bagasse, cellulose, and cellulose acetate was also performed (Fig. 9). Using this technique, it was possible to identify the transitions that occurred in the samples, if there was absorption or energy released during each of the transitions.

In the DSC curve of the cellulose acetate, there were three transitions, two endothermic and one exothermic. The first transition with a temperature of 37 °C represents the water vaporization, the second transition had an exothermic peak at 355 °C must be mainly related to the degradation of the acetylated lignin and hemicellulose derivatives (Filho *et al.* 2008). This peak can also correspond to degradation of cellulose acetate according to Candido *et al*. (2017).

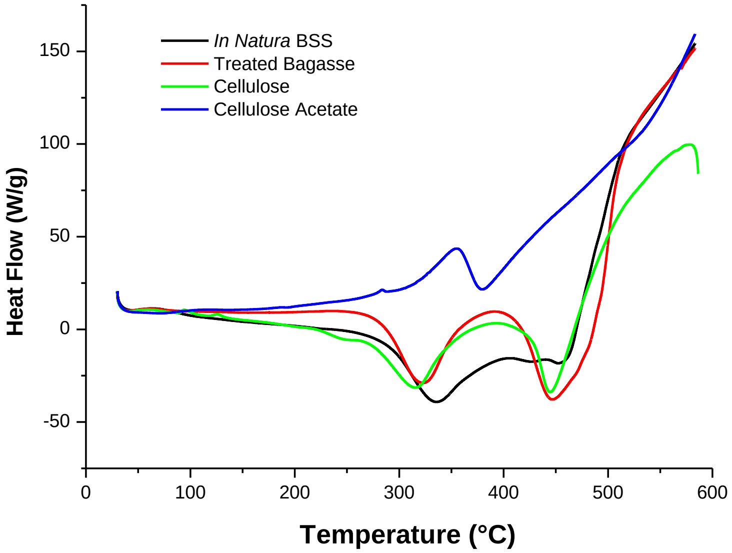

Fig. 9. DSC curves of the *in natura* BSS, treated bagasse, cellulose, and cellulose acetate

When analyzing the *in natura* bagasse and treated bagasse curves, three transitions were observed in each, and all of them were endothermic. The first transition with an endothermic peak at 40 °C for both curves was attributed to the removal of water from the BSS structure. The second stage presented endothermic peaks at 336 °C (*in natura*) and 325 °C (treated BSS), and was related to cellulose heating without breaking connections (Cerqueira *et al*. 2010). Finally, the third stage had endothermic peaks at 455 °C (*in natura*) and 450 °C (pre-treated), and was related to the breakdown of cellulose macromolecules (Filho *et al.* 2008).

Evaluating the DSC curve obtained for cellulose, endothermic peaks were present at 34 °C from water vaporization, 318 °C from cellulose heating, and 444 °C from the breakdown of this polymer molecule. It was also noted that in this same curve, an exothermic peak started to form at 575 °C and was probably related to the degradation of cellulose and lignin (Campos 2015).

CONCLUSIONS

1. The performance of the saccharin sorghum bagasse (BSS) pretreatment was enhanced by an increase in the alkaline hydrogen peroxide concentration and reaction temperature, which made it possible to achieve the maximum lignin removal percentage and cellulose yield when the time was fixed at the lower level.
2. The conversion of the cellulose extracted from the BSS into cellulose acetate produced cellulose diacetates and triacetates.
3. The X-ray diffraction (XRD) analysis showed characteristic peaks of lignocellulosic materials. The Fourier transform infrared (FTIR) analysis demonstrated that the hydrogen peroxide pretreatment removed hemicellulose and lignin, and it did not modify the cellulose structure by much. The thermogravimetric (TG) and differential thermogravimetric (DTG) curves showed mass losses similar to those reported in the literature, which were confirmed by the differential scanning calorimetry (DSC) curves.
4. In the present study, the valorization of sorghum residue, a renewable source that contains a considerable percentage of cellulose and whose production is quite extensive in Brazil, was sought for determining diverse applications in addition to those already considered.

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