Kraft Pulping and Bleaching of *Eichhornia crassipes* (Mart.) Solms (Water Hyacinth)

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Eichhornia crassipes (water hyacinth) was pulped by means of a kraft pulping process with reagent loads of 10 and 20% on a dry matter basis to determine yield, rejects, kappa number, and ash. Fiber classification, brightness, opacity, and viscosity were measured in the brown pulp. Bleaching was performed by means of an $O_1O_2D_1(PO)D_2HD_3$ sequence. Yield, kappa number, pH, ash, brightness, opacity, and viscosity were evaluated in the bleached pulp. Finally, a microanalysis of inorganic elements was carried out in both the bleached and unbleached pulp ash. The highest kraft pulp yield was 26.4%, with a 10% reagent load at 120 °C and 30 minutes cooking. It was determined that *E. crassipes* cellulosic pulp contains large amounts of fines. Results of the bleaching sequence indicate low brightness (58.0 %) and low viscosity (6.43 cP). The most abundant inorganic elements in the ash of both bleached and unbleached pulp were Ca, Mg, P, and Si. These results suggest that *E. crassipes* biomass might complement cellulosic fibers in pulping processes of low yield, such as the wood fibers used to produce handmade paper.

Keywords: Cellulosic pulp; Kappa number; Bleaching process; Inorganic substances

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INTRODUCTION

Pulp and paper production is almost exclusively based on wood. However, a growth in paper demand, together with a decline in the supply of fibers from forests, are forcing the pulp and paper industry to find alternative sources of fibers that are both technical and economically viable to complement forest resources (Jahan *et al*. 2008). The production of paper and paperboard in the world has increased as well. In 2009, the production was 371 million tons, while in 2015, 400 million tons were produced (FAO 2017). Likewise, in 2013 the Mexican paper industry required 5 million 144 thousand tons of fiber in order to produce 4 million 513 thousand tons of paper (INEGI 2013), increasing to approximately 6 million 822 thousand tons of fiber for the production of 5 million 956 thousand tons of paper by 2018 (Cámara del Papel 2018). Therefore, the global scarcity of fibrous resources has aroused great interest in the use of non-conventional fibrous raw materials (straw, sugar cane bagasse, bamboo, and miscellaneous raw materials) (Atchison 1996), (*Ricinus communis*, *Cyperus papyrifera*, *Typha domingensis*, *Agave tequilana*) (Escoto-García *et al*. 2013), which can be used to obtain cellulose for further paper production. Anupam *et*

al. (2016) present a classification of non-woody fibers that could be used for the manufacture of paper: agricultural residues (sugarcane bagasse, cotton stalks, rice straw), natural growing plants (bamboo, reeds, sabai grass, kahi grass), non-wood crops grown primarily for its fiber content such as bast fiber (jute, hemp, kenaf), leaf fiber (sisal), and seed hair (cotton fiber, cotton linter).

E. crassipes is a floating aquatic plant, native to the Amazon basin in Brazil (Barrett 1980). It is an invasive plant that has spread within tropical and subtropical regions of the world (Villamagna and Murphy 2010), with extremely rapid proliferation (Malik 2007). Biomass of this plant amounts up to 1,800 to 2,700 tons of wet raw material or to 90 to 135 tons of absolute dry biomass per hectare (Shoyakubov and Aitmetova 1999). A mat of these medium-sized plants may contain approximately 2,000,000 plants per hectare with a weight between 270 and 400 tons (Malik 2007). Furthermore, it is estimated that over a 6-month period, 125 tons of wet weight are produced in an area of one hectare (Istirokhatun *et al*. 2015). Moreover, this invasive plant has been reported to cause serious ecological impacts, such as a loss of diversity and a hybridization with native species, alterations in ecosystem processes, and an increase in pests and diseases. Also, it can cause serious economic difficulties to navigation and irrigation systems (Rodríguez 2006; Villamagna and Murphy 2010; Mahamadi 2011; Stiers *et al*. 2011; Nguyen *et al.* 2015).

Due to its alarming reproductive and propagation capacity, *E. crassipes* is considered a threat to biodiversity (Istirokhatun *et al.* 2015; Tan *et al*. 2015). Even after the use of traditional mechanical methods for its elimination and phytoremediation of contaminated water, the problem of how to use this valuable lignocellulosic resource in a reasonable and efficient way remains (Feng *et al*. 2017).

Some previous scientific reports on this aquatic plant have dealt with topics of, for example, saccharification processes (Abdel-Fattah *et al*. 2012; Reales-Alfaro *et al*. 2013), obtaining bioethanol (Nigam 2002; Masami *et al*. 2008; Aswathy *et al*. 2010; Satyanagalakshmi *et al*. 2011; Bergier *et al*. 2012; Ganguly *et al*. 2012; Manivannan *et al*. 2012; Awasthi *et al*. 2013; Singh and Bishnoi 2013; Manivannan and Narendhirakannan 2014), acid-catalysed hydrolysis (Girisuta *et al*. 2008), biosorbent (Mahamadi 2011; Murithi *et al*. 2014; Vijetha *et al*. 2014), nutritional value (Mako *et al*. 2011; Saha and Ray 2011), pyrolysis process (Promdee *et al*. 2012), and antimicrobial activity of its extracts (Thamaraiselvi and Jayanthi 2012).

Regarding the application of *E. crassipes* for pulp and paper manufacture, the following reports stand out: Bagnall *et al*. (1974) proposed the use of fibers from this aquatic plant to make paper, as well as a nutrient absorber in treated wastewater, forage feed, and compost. Nolan and Kirmse (1974) obtained cellulosic pulp through four chemical processes, including the kraft process. Widyanto *et al*. (1983) propose the use of water hyacinth as an absorbent of pollutants in paper factories and to cultivate this plant to complement the raw material to make pulp and paper. Jeododibroto *et al*. (1983) studied the morphology of *E. crassipes*, obtained soda pulp, and carried out a chlorine bleaching process. Das *et al*. (2013) propose the use of this aquatic plant as an alternative raw material for the pulp and paper industry. Recently, the authors' work team has carried out some studies on the chemical composition of water hyacinth (Fileto-Pérez *et al*. 2013; Fileto-Pérez *et al*. 2015; Lara-Serrano *et al.* 2016; Pintor-Ibarra *et al.* 2018). Thus, the aim of this research is to obtain cellulosic pulp by means of a kraft pulping process followed by a bleaching sequence, in order to use *E. crassipes* pulp as a complement to wood cellulosic fibers and as an alternative raw material for cellulosic fibers used in paper production. In

the present study, in addition to other results, the ash analysis of the *E. crassipes* pulp is reported, and a bleaching sequence was applied, which was not previously reported.

EXPERIMENTAL

Materials

Samples of *Eichhornia crassipes* were collected at Cuitzeo Lake, located in the State of Michoacán, Mexico, between 19°53'15" and 20°04'34" North latitude and between 100°50'20" and 101°19'34" West longitude. *E. crassipes* samples were washed with abundant water at constant current to remove contaminants such as soil, seashells, and small stones, among others. The biomass was dried outdoors in the shade until it contained approximately 12% moisture (TAPPI T 412 om-06 2006a). Fibers were manually separated from the plant.

Kraft Pulping Process

Pulping conditions were as follows: white liquor sulfidity 26%, active alkali 100 g/L, and a liquor to wood ratio of 12:1 with a 10 and 20% reagent load, on a dry matter basis. The kraft cooking process was carried out in a 15 L heated reactor bath (Jayme type, Stober Deutsch & Newmann, Germany) using a 2^2 factorial design with a central point (Gutiérrez-Pulido and de la Vara-Salazar 2004). Factor A was temperature (120 and 150 °C), and factor B was cooking time at T_{max} (10 and 30 min) (See Tables 2 and 3).

The obtained data were analyzed at a 95% confidence level and processed using the Statgraphics Version 4 Plus software to evaluate yield, rejects, kappa number (TAPPI T 236 cm-99 1999) and ash content (TAPPI T 413 om-93 1993). After each cooking stage, cellulosic pulp was washed with water at current flow and filtered on a 325-mesh sieve. The screened pulp was stored at room temperature for its later use. The residual black liquor obtained was filtered and stored for future research.

Only the brown kraft pulp with highest yield was screened in a diaphragm equipment (Type F1 117, Serie 1123, AB Lorentzen & Wettre, Stockholm, Sweden) with a 0.40 mm plate opening. The fiber classification was determined using Bauer-McNett equipment (AB Lorentzen & Wettre, Stockholm, Sweden) (TAPPI T 233 cm-06 2006). The following parameters were evaluated in standard sheets (60 $g.m^{-2}$) of high-yield brown kraft pulp, which were formed in a conventional semi-automatic TAPPI equipment (TMI Testing Machines Inc, Amityville, NY, USA) (TAPPI T 205 sp-02 2002): brightness (TAPPI T 452 om-02 2002), opacity (TAPPI T 519 om-02 2002), and viscosity (SCAN-CM 15:88 1988).

Bleaching Process

Bleaching was performed only to high-yield brown kraft pulp by means of an $O_1O_2D_1(PO)D_2HD_3$ sequence, which stands for oxygen, chlorine dioxide, peroxide oxygen, and sodium hypochlorite (Table 1). At the end of every bleaching stage, yield, kappa number (TAPPI T 236 cm-99 1999), pH (TAPPI T 625 cm-85 1984), and ash content (TAPPI T 413 om-93 1993) were determined. Brightness (TAPPI T 452 om-02 2002), opacity (TAPPI T 519 om-02 2002), and viscosity (SCAN-CM 15:88 1988) were determined in 60 $g.m^{-2}$ standard sheets only at the end of the bleaching sequence.

Table 1. Bleaching Stages Conditions for Brown Kraft Pulp

Conditions	O_1	O_2	D_1	PO	D_2	H	D_3
Oxygen pressure (atm)	5	5	--	5	--	--	--
Soda (wt.%)	4	5	--	0.5	--	0.2	--
Magnesium sulfate (wt.%)	0.5	0.5	--	0.5	--	--	--
Peroxide (wt.%)	--	--		3	--	--	
Chlorine dioxide (wt.%)	--	--	2.55		3	--	1.5
Sulfuric acid (mL)	--	--	--	--	--	--	0.2
Sodium hypochlorite (wt.%)	--	--	--	--	--	2	--
Consistency (wt.%)	10	10	10	10	10	10	10
Temperature (°C)	100	100	80	100	80	40	70
Time (min)	60	60	60	90	180	180	180
pH	11.28	11.42	4.91	11.06	3	11.04	4.40

Ash Microanalysis

Determination of inorganic elements in brown pulp ash and in ash after each stage of the bleaching process was performed using an X-ray spectrometer, connected to a Jeol JSM - 6400 scanning electron microscope (Tokyo, Japan), with operating conditions of 20 kV and 8.5s (Téllez-Sánchez *et al.* 2010).

RESULTS AND DISCUSSION

Kraft Pulping Process

The results obtained in each experimental run according to the design matrix are shown in Tables 2 and 3. The analysis of variance (ANOVA) for each response variable is presented in Table 4 (10 % reagent load), and Table 5 (20 % reagent load).

Yield

The ANOVA indicates that there was no statistically significant difference for yield, for both reagent loads used in the experimentation (Tables 4 and 5). Figure 1 shows the effect of temperature and time on yield during the kraft pulping process with 10 and 20% reagent loads on a dry basis.

Table 2. Results of the Design Matrix for 10% Reagent Loading

Run	Factor A	Factor B	Response variables			
	T (°C)	Time (min)	Yield (%)	Rejects (%)	Kappa number	Ash (%)
1	150	10	22.07	0.45	33.20	12.15
2	120	30	26.36	2.97	30.02	12.68
3	135	20	16.91	3.02	32.25	11.09
4	150	30	18.04	0.37	31.10	11.36
5	120	10	14.49	6.11	32.75	5.36
6	150	10	22.09	0.51	32.90	7.20
7	120	30	26.26	3.20	30.20	10.04
8	135	20	17.01	3.06	32.85	10.44
9	150	30	17.90	0.90	32.00	10.04
10	120	10	26.90	3.80	31.01	6.12

Table 3. Results of the Design Matrix for 20% Reagent Loading

Run	Factor A	Factor B	Response variables			
	T (°C)	Time (min)	Yield (%)	Rejects (%)	Kappa number	Ash (%)
1	150	30	18.53	0.36	31.6	5.14
2	150	10	18.24	0.17	23.00	16.62
3	120	10	18.61	0.29	20.60	7.01
4	120	30	17.61	2.10	27.85	8.04
5	135	20	15.85	0.24	27.40	18.45
6	150	30	18.58	0.39	32.00	5.10
7	150	10	18.21	0.18	23.06	16.79
8	120	10	18.67	0.92	21.01	12.15
9	120	30	18.50	3.00	27.50	6.19
10	135	20	16.01	0.25	28.10	14.50

Table 4. ANOVA for the Response Variables (10% reagent load)

Source	SS	DF	MS	F-Ratio	p-Value
Yield					
A: Temperature	24.19	1	24.19	1.22	0.3190
B: Time	1.13	1	1.13	0.06	0.8203
AB	47.29	1	47.29	2.39	0.1826
Blocks	15.10	1	15.10	0.76	0.4220
Total error	98.84	1	19.77		
Total (corr.)	186.55	5			
Rejects					
A: Temperature	23.98	1	23.98	33.96	0.0021
B: Time	1.47	1	1.47	2.08	0.2086
AB	2.05	1	2.05	2.90	0.1491
Blocks	0.21	1	0.21	0.30	0.6087<
Total error	3.53	1	0.71		
Total (corr.)	31.24	5			
Kappa number					
A: Temperature	3.41	1	3.41	4.94	0.0770
B: Time	5.35	1	5.35	7.75	0.0387
AB	0.04	1	0.04	0.05	0.8273
Blocks	0.01	1	0.01	0.02	0.8963
Total error	3.45	1	0.69		
Total (corr.)	12.25	5			
Ash					
A: Temperature	5.36	1	5.36	2.15	0.2026
B: Time	22.08	1	22.08	8.84	0.0310
AB	10.56	1	10.56	4.23	0.0949
Blocks	7.74	1	7.74	3.10	0.1385
Total error	12.48	1	2.49		
Total (corr.)	58.22	5			

Table 5. ANOVA for the Response Variables (20% reagent load)

Source	SS	DF	MS	F-Ratio	p-Value
Yield					
A: Temperature	0.003	1	0.003	0.00	0.9674
B: Time	0.03	1	0.03	0.02	0.9025
AB	0.42	1	0.42	0.21	0.6634
Blocks	0.13	1	0.13	0.07	0.8087
Total error	9.80	1	1.96		
Total (corr.)		5			
Rejects					
A: Temperature	3.39	1	3.39	15.47	0.0110
B: Time	2.30	1	2.30	10.49	0.0230
AB	1.52	1	1.52	6.94	0.0463
Blocks	0.25	1	0.25	1.14	0.3349
Total error	1.09	1	0.22		
Total (corr.)	8.56	5			
Kappa number					
A: Temperature	21.91	1	21.91	15.96	0.0104
B: Time	126.56	1	126.56	92.17	0.0002
AB	1.33	1	1.33	0.97	0.3705
Blocks	0.31	1	0.31	0.23	0.6548
Total error	6.87	1	1.37		
Total (corr.)	156.98	5			
Ash					
A: Temperature	13.16	1	13.16	0.67	0.4492
B: Time	98.70	1	98.70	5.05	0.0745
AB	41.59	1	41.59	2.13	0.2044
Blocks	0.03	1	0.03	0.00	0.9712
Total error	97.68	1	19.54		
Total (corr.)	251.15	5			

The lowest yield was 14.5% and the highest was 26.4%, both at a 10% reagent load (Table 2). For the 20% reagent load, the lowest yield was 15.8% and the highest was 18.6% (Table 3). These results demonstrate that the higher the reagent load, the lower the yield obtained. Similarly, Widyanto *et al.* (1983) reported a decrease in yield as the reagent load increased with soda pulping for *E. crassipes*. In Fig. 1a it can be observed that to maximize pulp yield, the combination of *T* (120 °C) and *t* (30 min) should be used. In Fig. 2a. it is observed that the effect of increasing the time is positive (the yield increases) as long as the temperature is lowered (120 °C). So, for the case of using 10% reagent load, the equation of the fitted model to maximize yield is (optimum value = 25.3%):

$$Yield = -8.06325 + 0.20825 * T + 2.22575 * t - 0.0162083 * T * t \quad (1)$$

where *T* is temperature (°C) and *t* is time (min).

For kraft pulping with 20% reagent loading, in Fig. 1b it is observed that to achieve maximum pulp yield, the combination of *T* (150 °C) and *t* (10 min) should be used. However, in Fig. 2b the effect of keeping the time low (10 min) on the highest yield is observed as long as the temperature is low (120 °C). It is known that the conclusion obtained only by the analysis of the main effects is not always correct (Gutiérrez-Pulido and de la Vara-Salazar 2004), as shown by these results. So, for the case of using 20%

reagent load, the equation of the fitted model to maximize yield is (optimum value = 18.2 %):

$$Yield = 21.9348 - 0.0290833 * T - 0.21225 * t + 0.001525 * T * t \quad (2)$$

where T is temperature (°C) and t is time (min).

The determination coefficients are low, both for pulping with 10% (R-squared is 47.0% and R-squared (adjusted) is 20.5%), and with 20% (R-squared is 5.6% and R-squared (adjusted) is 0.0%) reagent loading. In this case, low values that would indicate that the effect of these factors studied on pulp yield is small compared to the rest of the variability observed in the experiment, such once because the levels studied are narrow.

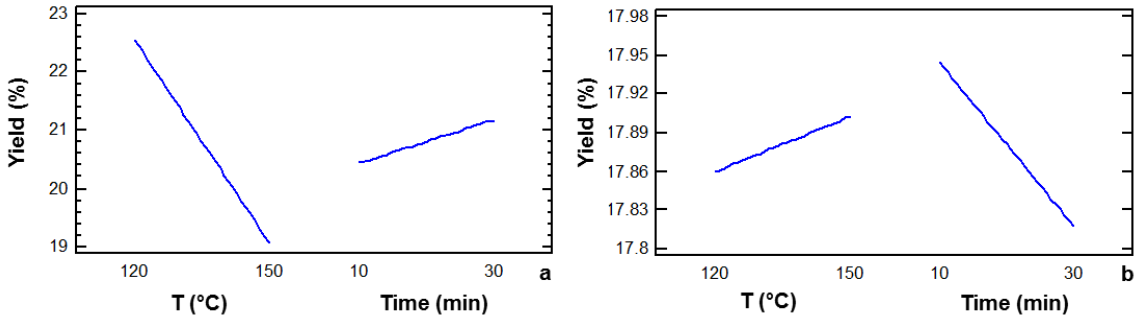

Fig. 1. Main effects on yield: a) 10% reagent load, b) 20% reagent load

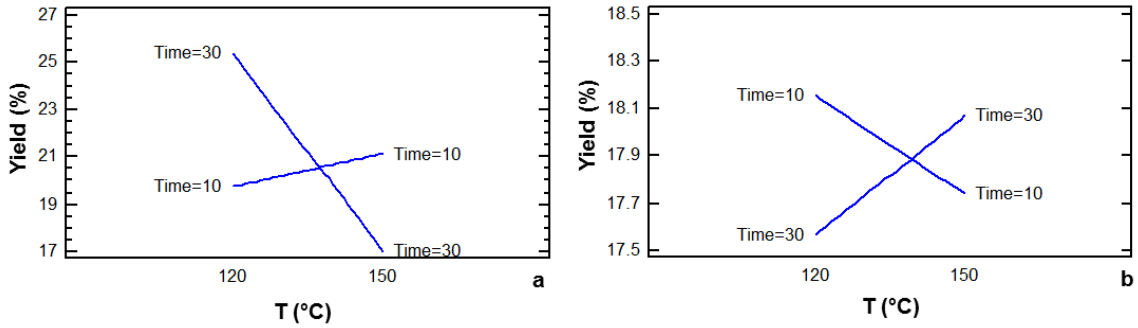

Fig. 2. Interaction effects on yield: a) 10% reagent load, b) 20% reagent load

Figure 3 shows response surface plots for the response variable (pulp yield) and gives a visualization of what the fitted model (Eqs. 1 and 2) means over the experimentation region. It can be seen that the points where the surface takes higher values are precisely in the best treatment that had been found (10%: 120 °C and 30 min; 20%: 120 °C and 10 min).

The highest yield (26.4%) obtained with a reagent load of 10% at 120 °C and 30 min (Table 2) was within the 15 to 30% yield range obtained for this aquatic plant as reported by Bagnall *et al.* (1974). Kumar *et al.* (2015) obtained a higher yield value (33.9%) in stems and leaves of water hyacinth. Compared to other raw materials, the yield obtained in this work was lower than that reported (43.7%) for *Cymbopogon winterianus* (citronella grass) kraft pulp (Sharma *et al.* 2017), and than that obtained (51.7%) for *Melia dubia* kraft pulp (Deepika *et al.* 2018). Moreover, Lara-Serrano *et al.* (2016) studied the chemical components of *E. crassipes* and concluded that if biomass is used as raw material in the cellulosic pulp manufacturing process, the yield would be low due to its low holocellulose

content. These low results contrast with the obtained yields for softwood and hardwood kraft pulping, which range from 44 to 51% (Libby 1980; Rutiaga-Quiñones *et al*. 1998; Juacida *et al*. 2002; Gabriel-Parra *et al*. 2018).

In addition, pulping conditions for wood and for water hyacinth are different. For wood, pulping conditions are a 20% reagent load, 90 to 120 min cooking time, and temperatures from 168 to 170 °C, contrasting with water hyacinth pulping conditions, which require lower operating conditions, resulting in lower pulping costs. This shows that the variability of organic chemical components in different fibrous species have different demands on reagents and pulping times (Juacida *et al*. 2002).

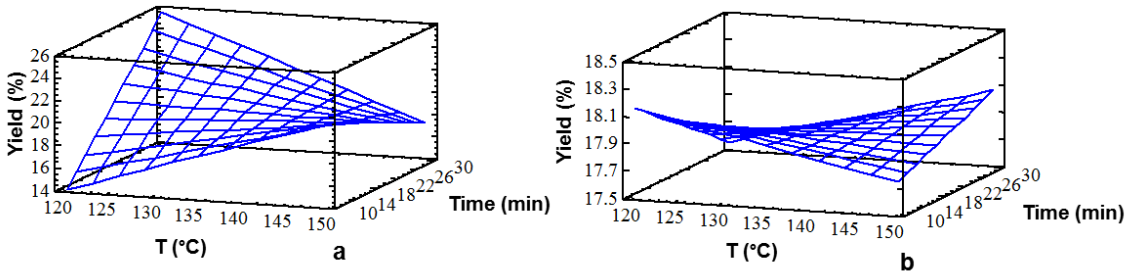

Fig. 3. Response surface for yield a) 10% reagent load, b) 20% reagent load

Rejects

ANOVA indicated that there were statistically significant differences for the response variable (rejects) (Tables 4 and 5). Figure 4a shows that the minimum of rejects was achieved with treatment *T* (150 °C) and *t* (30 min), while in the interaction plot (Fig. 4b) the effect of the shorter time was observed (rejects decreases) as long as the temperature was kept high (*T* = 150 °C). Then, the equation of the fitted model to minimize the content of rejections with optimal conditions (*T* = 150 °C and *t* = 10 min) was the following (optimum value = 0.63%),

$$Rejects = 27.9902 - 0.182917 * T - 0.4985 * t + 0.003375 * T * t \quad (3)$$

where *T* is temperature (°C) and *t* is time (min).

It is known that taking conclusions from the analysis of the main effects of the studied factors is not always the correct way (Gutiérrez-Pulido and de la Vara-Salazar 2004), so it is better to rely on the analysis of iteration effects, as shown by these results.

For the case of using 20% reagent load, the analysis of both the main effects (Fig. 4b) and the interaction effects (Fig. 5b), it is concluded that to minimize the amount of rejects, the combination of *T* (150 °C) and *t* (10 min) should be used. In this case, the equation of the fitted model to minimize rejects (optimum value = 0.04%) is as follows:

$$Rejects = -2.27375 + 0.01475 * T + 0.44625 * t - 0.00290833 * T * t \quad (4)$$

where *T* is temperature (°C) and *t* is time (min).

The determination coefficients obtained were the following: R-squared = 88.7% and R-squared (adjusted) = 83.0% (for 10% reagent load), and R-squared = 87.2% and R-squared (adjusted) = 80.8% (for 20% reagent load). This means that the studied factors, together with their interactions, explained a high percentage of variability observed in the response variable (rejects).

With a 10% reagent load in the kraft pulping process, rejects values ranged from 0.37% to 6.11% (Table 2). On the other hand, with a 20% reagent load, results ranged from 0.17 to 2.21% (Table 3). Thus, the higher the reagent load percentage, the lower the rejects percentage and the higher the obtained yield. MacLeod (2007) points out that when the variables reagent load and temperature increase, the number of rejects in the pulping process decreases. Furthermore, the number of rejects generated in the pulping process of *E. crassipes* was similar to those reported for softwood and hardwood species, which range from 1.5 to 3.4% (Juacida *et al*. 2002).

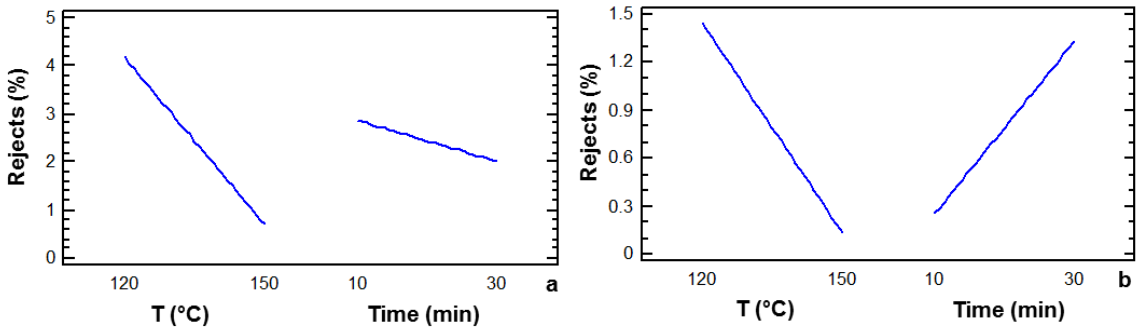

Fig. 4. Main effects on yield: a) 10% reagent load, b) 20% reagent load

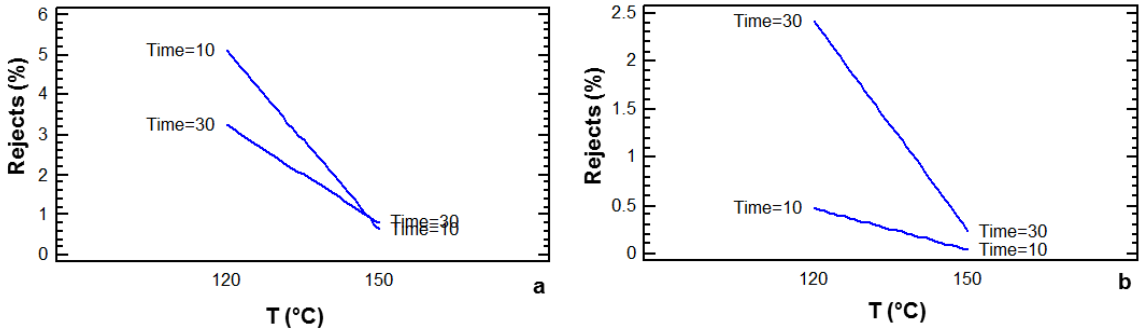

Fig. 5. Interaction effects on yield: a) 10% reagent load, b) 20% reagent load

The response surface plots for the response variable (rejects) visualize what the fitted model (equations 3 and 4) means in the experimental region (Fig. 6). It can be seen that the points where the surface takes lower values were precisely in the best treatment found (10%: 150 ° C and 10 min; 20%: 150 ° C and 10 min).

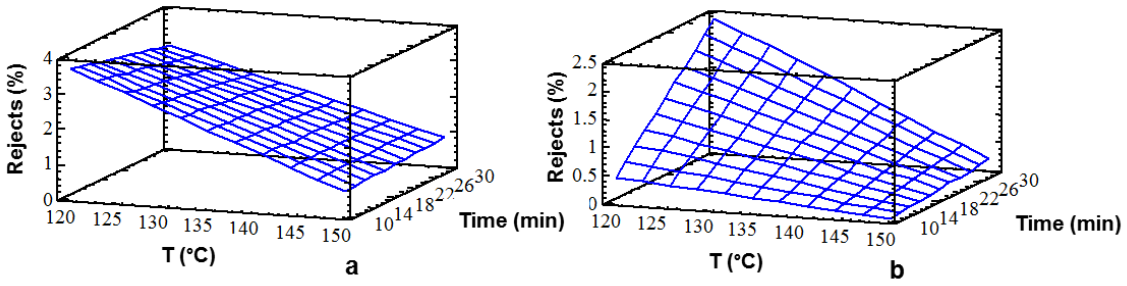

Fig. 6. Response surface for rejects, a) 10% reagent load, b) 20% reagent load

Kappa number

Statistically significant differences were found for the response variable (kappa number) only for the kraft pulp with 20% reagent load (Tables 4 and 5). Figure 7a shows the effects of the factors studied, and the minimum kappa number was obtained with 120 °C and 30 min. Likewise, the interaction plot (Fig. 8a) indicates that with a time of 30 min there was lower kappa number, as long as the process was carried out at 120 °C. For this case using 10% reagent load, the equation of the fitted model to minimize the kappa number (optimum value = 30.3) was as follows:

$$Kappa\ number = 28.8055 + 0.0345 * T - 0.1425 * t + 0.00045 * T * t \quad (5)$$

where T is temperature (°C) and t is time (min).

When using 20% reagent loading, it is seen in Fig. 7b that to achieve the low kappa number, the combination of 120 °C and 10 min must be used. Likewise, in the interaction plot (Fig. 8b) with 10 min of cooking kraft, a low kappa number was obtained, as long as the pulping process was carried out at 102 °C. Thus, the equation of the fitted model to minimize the kappa number was (optimum value = 21.2):

$$Kappa\ number = 12.6545 + 0.0425 * T - 0.0365 * t + 0.00316667 * T * t \quad (6)$$

where T is temperature (°C) and t is time (min).

The determination coefficients obtained were the following: R-squared = 71.8% and R-squared (adjusted) = 57.8% (for 10% reagent load), and R-squared = 95.9% and R-squared (adjusted) = 93.8% (for 20% reagent load). This means that the studied factors, together with their interactions, explained a high percentage of variability observed in the response variable (kappa number).

The amount of residual lignin in water hyacinth pulp also differed when using a 10% and a 20% reagent load, ranging from 30.0 to 33.2 (Table 2) and from 20.6 to 31.6 (Table 3), respectively, coinciding with several reports (Casey 1990; MacLeod 2007; Wan Daud *et al.* 2009; Kumar *et al*. 2015). In general, values obtained for kappa number were high (21 to 33). Therefore, it can be predicted that for bleaching *E. crassipes* cellulosic pulp, high amounts of reagents will be required. During cooking, *E. crassipes* cellulosic pulp darkened, agreeing with what by Bagnall *et al.* (1974) reported. Additionally, kappa number values obtained here are comparable to those reported for kraft pulping (Kumar *et al*. 2015) and for soda pulping (Joedodibroto *et al.* 1983) for the same aquatic plant. They are also within the range of 18.9 to 31.0 reported for kraft pulping of softwood and hardwood species (Rutiaga-Quiñones *et al*. 1998; Juacida *et al.* 2002; Torres *et al*. 2005).

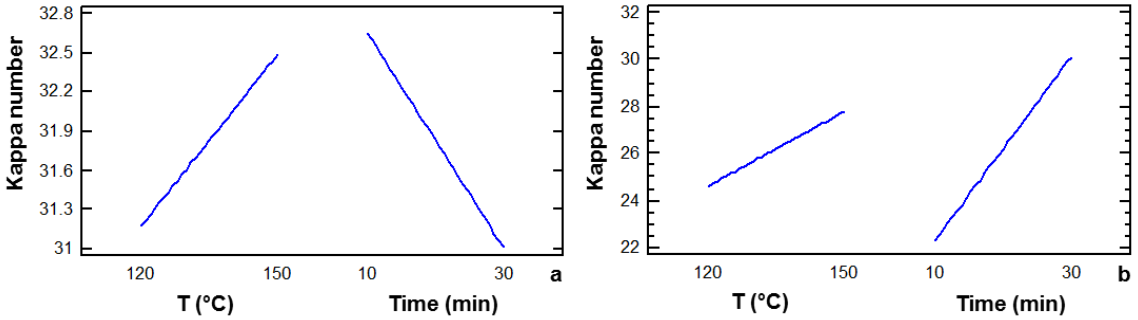

Fig. 7. Main effects on kappa number: a) 10% reagent load, b) 20% reagent load

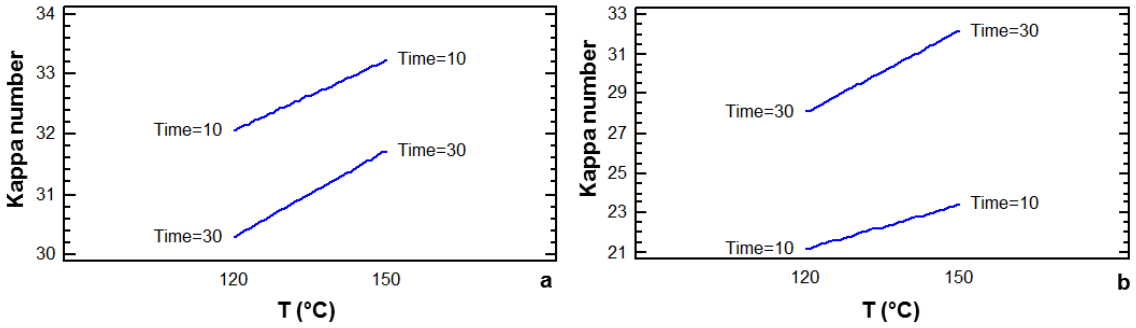

Fig. 8. Interaction effects on kappa number: a) 10% reagent load, b) 20% reagent load

The response surface plots for the response variable (kappa number) visualize what the fitted model (Eqs. 5 and 6) means in the experimental region (Fig. 9). It can be seen that the points where the surface takes lower values were precisely in the best treatment found (10%: 120 °C and 30 min; 20%: 120 °C and 10 min).

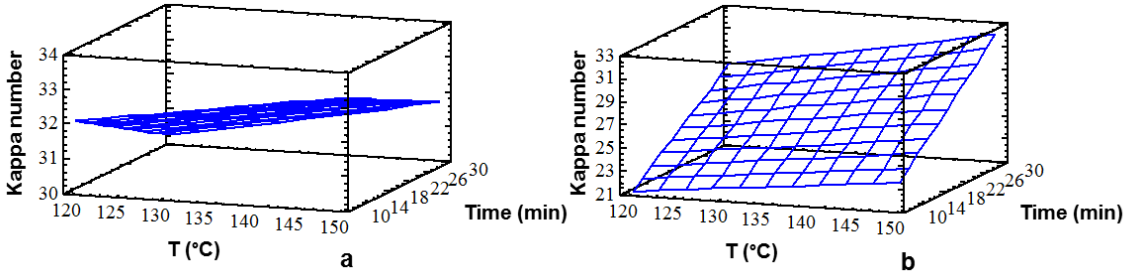

Fig. 9. Response surface for kappa number, a) 10% reagent load, b) 20% reagent load

Ash content in high-yield brown kraft pulp

Table 4 presents the ANOVA for the ash content at 10% reagent load, and Table 5 shows it for 20% reagent load. In the main effects plot it is observed that the combination of 120 °C and 10 min (Fig. 10a) should be used to obtain low ash content in the kraft pulp. Likewise, the interaction plot shows that a low ash content was obtained with a cooking time of 10 min, as long as the cooking process was carried out at 120 °C. Under these conditions, for a 10% reagent load, the equation of the fitted model to minimize the ash content is the following (optimum value = 6.0%):

$$Ash = -21.7207 + 0.20775 * T + 1.2 * t - 0.00765833 * T * t \quad (7)$$

where T is temperature (°C) and t is time (min).

When the pulping process was carried out with a 20% reagent load, the main effects plot shows that to obtain low ash content, the combination of 120 °C and 30 min should be used. However, in the interaction plot it is observed that maintaining a high time (30 min) low ash content was achieved, but always when the cooking temperature was 150 °C. This same effect was observed in the case of the response variables yield and rejects. For this case, with the optimal conditions of 150 °C and 30 min, the equation of the model adjusted to minimize the ash content (optimum value = 6.5%) was as follows,

$$Ash = -34.5585 + 0.3895 * T + 1.70075 * t - 0.0152 * T * t \quad (8)$$

where T is temperature (°C) and t is time (min).

For this case, the coefficients of determination obtained were as follows: R-squared = 78.6% and R-squared (adjusted) = 67.8% (for 10% reagent load), and R-squared = 61.1% and R-squared (adjusted) = 41.7% (for 20% reagent load). This means that the studied factors temperature and time, together with their interactions, explained a high percentage of variability observed in the response variable (ash content).

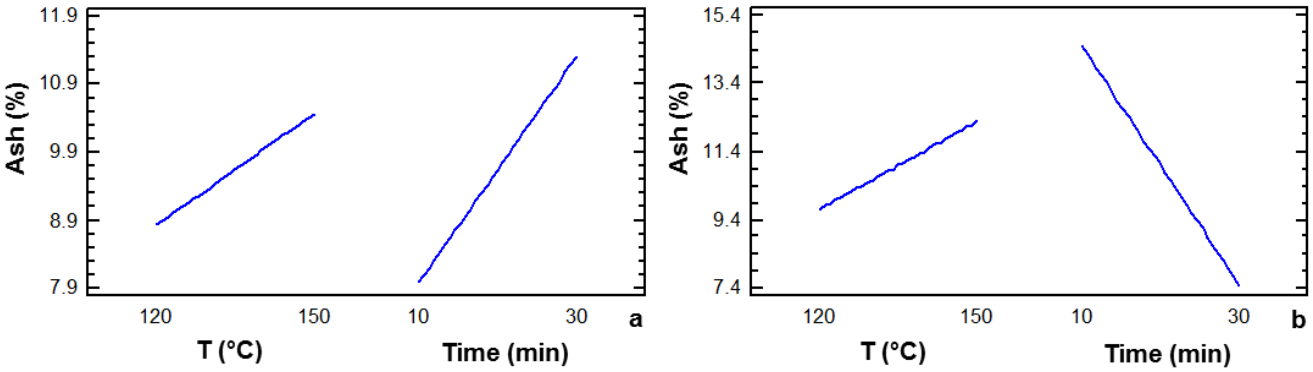

Fig. 10. Main effects on ash content: a) 10% reagent load, b) 20% reagent load

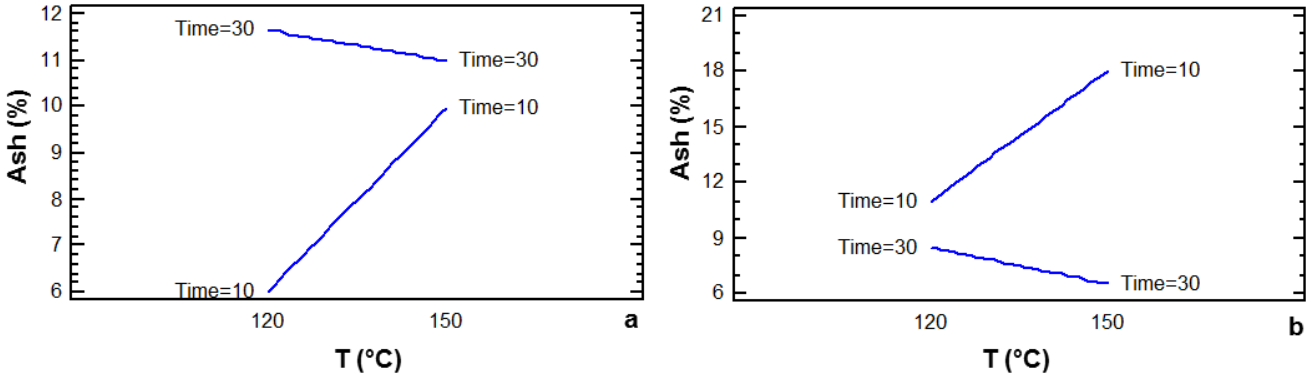

Fig. 11. Interaction effects on kappa number: a) 10% reagent load, b) 20% reagent load

Previous reported values of inorganic substances content in the *E. crassipes* range from 19.1 to 22.9% (Fileto-Pérez *et al.* 2013; Pintor-Ibarra *et al*. 2018). This high ash content is related to its natural characteristic to absorb and concentrate toxic minerals and metals from the aquatic environment (Mahmood *et al.* 2005). The values obtained for cellulose pulp vary from 5.7 to 10.8% when pulping with a 10% reagent load (Table 2), while when carrying out the process with a 20% reagent load the results ranged from 5.1 to 16.6% (Table 3). These results were lower than those of the original raw material due to the fact that some inorganic substances are soluble in alkali (Doldan *et al.* 2011) and were eliminated during the pulping process. These results do not show a reduction in the percentage of ash in the cellulosic pulp by increasing the load of chemical reagents during the kraft pulping process, as it occurs in a soda pulping process (Widyanto *et al*. 1983).

The response surface plots for the response variable (ash) visualize what the fitted model (Eqs. 7 and 8) means in the experimental region (Fig. 12). It can be seen that the points where the surface takes lower values were precisely in the best treatment found (10%: 120 °C and 10 min; 20%: 150 °C and 30 min).

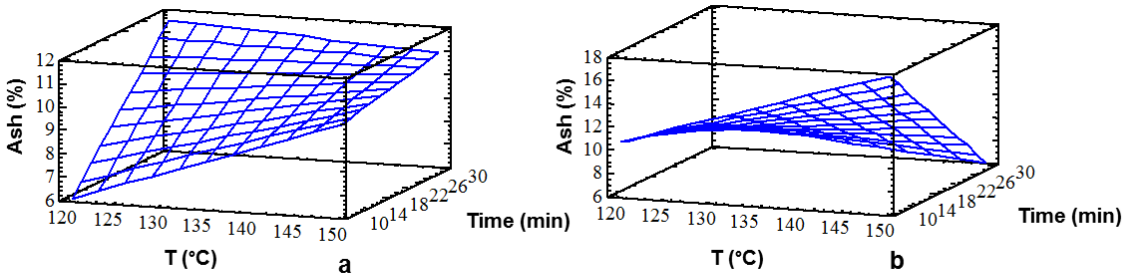

Fig. 12. Response surface for ash: a) 10% reagent load, b) 20% reagent load

Fiber classification

As shown in Table 6, *E. crassipes* fibers contained a high proportion of fines (47.8%), compared with *Pinus douglasiana* kraft pulp (Rutiaga-Quiñones *et al.* 1998), which has a long fibers retention of 87.1% in the 30 mesh. A high content of fines is also reported in cellulosic pulp of *Cymbopogon winterianus* (citronella grass) (Sharma *et al.* 2020). The high fines in *E. crassipes* is related to its structural fine elements, such as parenchyma and aerenchyma cells (Mahmood *et al.* 2005). Unfavorable results have been documented for drainage of *E. crassipes* pulp through sheet formers, due to its high content of fine cells (Bagnall *et al.* 1974). Although fine fibers may decrease drainage freedom, fines are needed because they increase contact zones of cellulosic surfaces and favor tensile strength of paper sheets (Swanson and Steber 1959; Casey 1990; Young 1991).

Brightness, opacity, and viscosity in high-yield brown kraft pulp

Brightness and opacity percentages (Table 6) were lower than those reported for *Pinus douglasiana* brown kraft pulp, with values of 32.9% and 99.5% for brightness and opacity, respectively (Rutiaga-Quiones *et al.* 1998), which is reflected in *E. crassipes* kraft pulp dark color. This coincides with previous literature reports that state that water hyacinth darkens during the cooking process (Bagnall *et al.* 1974). *E. crassipes* brown kraft pulp presented lower viscosity than kraft pulp from timber species. Viscosity value reported for *Pinus douglasiana* kraft pulp is 12.6 cP (Rutiaga-Quiñones *et al.* 1998) and for *Pinus tecunumanii* kraft pulp 34 cP (Torres *et al.* 2005). However, no previous data on literature about cellulosic pulp viscosity of *E. crassipes* were found. Table 6 shows the characteristics of the high-yield brown kraft pulp using a 10% reagent load.

Table 6. Characterization of High-yield Brown Kraft Pulp

Determination	Value
Moisture (wt%)	69.42 (±0.02)
Yield (wt%)	26.36 (±0.42)
Kappa number	30.02 (±0.42)
Ash (wt%)	9.97(±1.8)
Fibers classification (wt%)	
mesh 30	28.31(±0.19)
mesh 50	6.8 (±0.4)
mesh 100	8.88 (±0.02)
mesh 200	8.17 (±0.19)
<200	47.83 (±0.41)
Brightness (%)	6.41 (±0.41)
Opacity (%)	97.48 (±0.21)
Viscosity (cP)	10.12 (±0.07)

Bleaching Process

Yield and kappa number

During the bleaching sequence the behavior for brown kraft pulp of *E. crassipes* was drastically different (Table 7). To begin with, pulp yields were low compared to *Pinus douglasiana* kraft pulp during a bleaching sequence applied (97.3 to 99.2%) (Rutiaga-Quiñones *et al.* 1998). No data were available on the yield of *E. crassipes* pulp during the bleaching process. The initial kappa number (30.0) (Tables 6 and 7) meant a 66.9% pulp delignification with double oxygen stage, which is higher than the delignification of pulps exposed to a single stage (41.7% to 63%) (Poukka *et al*. 1999; Suchy and Argyropoulos 2002). Delignification for *E. crassipes* pulp was greater than the delignification of *Pinus tecunumanii* kraft pulp (62%), with a double oxygen stage treatment (Torres *et al.* 2005).

Pulp delignification was determined based on the initial kappa number (Table 7). Then a kappa number was measured after applying chlorine dioxide. After the first application, a 46.6% delignification was achieved. This was followed by a 39.7% delignification on the second application until a 93.5% delignification was reached. The final kappa number was close to zero, which is related to a good delignification. Nonetheless, the brightness of the bleached pulp of *E. crassipes* (58.0%) was low compared to the kraft pulp of *Pinus douglasiana* (85.7%) using the bleaching sequence $O_1D_1D_2$ (Rutiaga-Quiñones *et al.* 1998) and in comparison with *P. tecunumanii* (90%) using the sequence $D_0E_oE_{op}D_1ED_2$ (Torres *et al.* 2005). The low percentage of bleaching in the cellulosic pulp of *E. crassipes* could have been caused by the high ash content and some extractives. It has been reported in the literature that high quantities of these substances produce a yellowish color in the cellulosic pulp and they also react with the bleaching agents, causing problems in the bleaching stages (Rapson 1963; MacDonald and Franklin 1969; Grace *et al.* 1996).

pH of pulp in the bleaching sequence

Concerning pH, values varied depending on the chemical reagent used at each stage of the bleaching process (Table 7).

Ash content of pulp in the bleaching sequence

Regarding ash percent, the initial value (9.9%) in the brown kraft pulp was lower compared to the value achieved after the first stage of bleaching (13.9%). Based on the results, the inorganic content persists, carbohydrates are degraded by bleaching reagents, as shown in the decrease in ash percentage of each bleaching stage (Table 7).

Brightness, opacity and viscosity in bleached kraft pulp

The final brightness achieved in the *E. crassipes* pulp was low with high opacity (Table 7), and its use may not be viable due to the number of stages applied in the bleaching sequence. However, the final brightness was high compared to that reported for *Melia dubia* kraft pulp (42.5%) (Anupam *et al.* 2018). On the other hand, several papers and paperboards on the market, such as those used for packaging, do not require bleached cellulosic pulp. Therefore, the pulp from this aquatic plant could be incorporated into the cellulosic pulp obtained from wood species. Final viscosity (Table 7) was also low compared to the 8.4 cP value reported for *Pinus douglasiana* pulp in the final bleaching stage (Rutiaga-Quiñones *et al.* 1998), possibly because bleaching processes affect cellulosic pulp viscosity, as previously described (Rapson 1963; Casey 1990; Suchy and Argyropoulos 2002). Also the viscosity obtained was low compared to the value of 16.7

cP reported for *Cymbopogon winterianus* (citronella grass) kraft pulp (Sharma *et al*. 2017).

Table 7. Characterization of Brown Kraft Pulp of *Eichhornia crassipes* during the Bleaching Process

Determination	Brown kraft pulp	Bleaching stages						
		O_1	O_2	D_1	PO	D_2	H	D_3
Yield (wt%)		68.14	70.47	84.27	87.34	82.94	97.24	78.23
Kappa number	30.02	16.27	9.94	5.39	3.45	2.08	0.62	0.04
pH		8	9.91	5.31	9.40	5.22	10.59	4.30
Ash (wt%)	9.97	13.09	11.84	10.24	10.21	9.38	7.01	6.88
Brightness (%)	6.41	--	--	--	--	--	--	57.96
Opacity (%)	97.48	--	--	--	--	--	--	95.50
Viscosity (cP)	10.12	--	--	--	--	--	--	6.43

Ash Microanalysis in Brown Kraft pulp

The most abundant inorganic elements in the kraft cellulosic pulp were calcium, magnesium, phosphorus, and silicon, both for a 10% and a 20% reagent load (Table 8).

Table 8. Inorganic Elements Detected in *E. crassipes* Brown Kraft Pulp (at.%)

10% Reagent load					
Element	**150 °C y 10 min**	**135 °C y 20 min**	**120 °C y 10 min**	**120 °C y 30 min**	**150 °C y 30 min**
Calcium	48.28 ±0.30	49.76 ±0.14	46.48 ±0.16	50.19 ±0.58	46.99 ±0.29
Magnesium	13.35 ±0.46	14.28 ±0.22	15.31 ±0.27	14.56 ±0.17	16.59 ±0.48
Phosphorus	14.93 ±0.18	15.19 ±0.44	16.26 ±0.51	15.55 ±0.48	15.63 ±0.16
Silicon	14.68 ±0.37	11.95 ±0.70	12.89 ±0.48	12.78 ±0.99	12.61 ±0.25
Sodium	2.39 ±0.62	2.37 ±0.43	3.37 ±0.76	2.54 ±0.30	2.95 ±0.12
Sulfur	1.54 ±0.08	2.05 ±0.02	1.58 ±0.30	1.13 ±0.01	1.52 ±0.15
Iron	1.25 ±0.1	0.63 ±0.06	nd	0.80 ±0.22	0.69 ±0.02
Aluminium	1.69 ±0.69	1.11 ±0.48	1.65 ±0.38	1.05 ±0.02	1.47 ±0.06
Potassium	0.83 ±0.14	0.75 ±0.15	0.77 ±0.08	0.50 ±0.19	0.64 ±0.07
Manganese	0.73 ±0.33	1.31 ±0.07	1.05 ±0.03	0.63 ±0.10	0.65 ±0.01
Chlorine	0.29 ±0.04	0.22 ±0.08	0.57 ±0.14	0.22 ±0.12	0.21 ±0.04
20% Reagent load					
Element	**150 °C y 10 min**	**135 °C y 20 min**	**120 °C y 10 min**	**120 °C y 30 min**	**150 °C y 30 min**
Calcium	50.81 ±0.64	52.81 ±0.05	51.85 ±0.53	54.85 ±0.11	48.86 ±0.23
Magnesium	17.23 ±0.01	16.25 ±0.25	16.88 ±0.43	16.23 ±0.04	21.81 ±0.13
Phosphorus	12.06 ±0.03	12.88 ±0.11	13.34 ±0.42	12.99 ±0.30	13.01 ±0.02
Silicon	12.80 ±0.23	11.78 ±0.04	11.67 ±0.10	10.43 ±0.09	5.10 ±0.28
Sodium	2.04 ±0.25	1.47 ±0.25	1.80 ±0.44	1.43 ±0.14	4.02 ±0.12
Sulfur	0.35 ±0.05	0.80 ±0.02	0.74 ±0.30	0.76 ±0.05	5.36 ±0.18
Iron	1.36 ±0.07	1.20 ±0.12	0.80 ±0.02	0.90 ±0.04	nd
Aluminium	2.13 ±0.18	1.47 ±0.04	1.63 ±0.40	1.15 ±0.18	0.31 ±0.18
Potassium	0.28 ±0.002	0.39 ±0.03	0.46 ±0.04	0.45 ±0.03	0.61 ±0.04
Manganese	0.77 ±0.12	0.91 ±0.11	0.67 ±0.08	0.77 ±0.05	0.87 ±0.20
Chlorine	0.11 ±0.06	Nd	0.11 ±0.004	0.009 ±0.006	nd
nd = not detected					

Some of the elements detected in this work have also been found in *Melia dubia* kraft pulp (Deepika *et al.* 2018). In general, there were no significant variations in the concentration of the inorganic elements detected. Potassium and chlorine were the least concentrated in the pulp (Table 8), whereas in the unprocessed biomass of *E. crassipes*, they were reported as the most abundant elements (Pintor-Ibarra *et al.* 2018). This is supported by Doldan *et al.* (2011), who indicate that these inorganic elements are highly soluble in alkali. Recently, Pintor-Ibarra *et al.* (2018) discussed the effects of inorganic elements present in lignocellulosic materials used in the pulp and paper industry, reporting that calcium, magnesium, iron, phosphorus, silicon, aluminum, and chlorine can cause serious problems during the pulping process. It is also known that these chemical elements favor fouling formation on the surfaces of heat exchangers where the combustion of the black liquor takes place in the kraft process and in the digester. In addition, such chemical elements increase the corrosion in evaporator pipes and produce inert lime in the lime recovery cycle (Libby 1980; Grace *et al.* 1996; Sithole and Allen 2002; Vakkilainen 2005; Tran and Vakkilainen 2007; Doldan *et al.* 2011).

Table 9. Ash Microanalysis of Bleached Pulp in the Bleaching Sequence (at.%)

Elements	Bleaching stages						
	O_1	O_2	D_1	PO	D_2	H	D_3
Calcium	59.51 ±0.04	56.04 ±0.05	60.30 ±0.52	56.39 ±0.17	61.19 ±0.25	55.53 ±0.12	47.39 ±0.41
Magnesium	10.77 ±0.06	14.70 ±0.49	8.50 ±0.34	11.35 ±0.11	6.94 ±0.21	6.001 ±0.36	5.39 ±0.37
Phosphorus	15.006 ±0.5	12.47 ±0.36	16.50 ±0.17	14.29 ±0.14	16.13 ±0.30	18.09 ±0.28	18.56 ±0.08
Silicon	9.15 ±0.05	11.62 ±0.44	11.47 ±0.14	13.12 ±0.20	10.27 ±0.01	9.97 ±0.34	11.87 ±0.08
Sodium	1.34 ±0.03	2.37 ±0.15	0.65 ±0.08	1.82 ±0.20	1.13 ±0.50	2.38 ±0.31	6.13 ±0.10
Sulfur	1.05 ±0.04	Nd	nd	0.42 ±0.06	nd	nd	0.42 ±0.10
Iron	0.81 ±0.05	0.71 ±0.02	0.59 ±0.26	0.78 ±0.09	1.34 ±0.36	1.99 ±0.69	1.47 ±0.27
Aluminium	0.54 ±0.03	0.83 ±0.01	0.96 ±0.33	0.92 ±0.08	1.09 ±0.08	1.34 ±0.04	2.24 ±0.27
Potassium	0.84 ±0.19	0.51 ±0.07	nd	nd	0.39 ±0.09	nd	0.50 ±0.001
Manganese	0.95 ±0.01	0.72 ±0.05	0.76 ±0.21	0.85 ±0.01	1.38 ±0.24	1.26 ±0.06	1.87 ±0.35
Chlorine	nd	Nd	0.23 ±0.006	nd	0.09 ±0.10	nd	nd
Zinc	nd	Nd	nd	nd	nd	3.40 ±0.27	4.10 ±0.35
nd = no detected							

Ash Microanalysis of Bleached Kraft Pulp

Table 9 shows the inorganic elements detected in the ash of the bleached pulp after each bleaching stage. The most abundant were calcium, magnesium, phosphorus and silicon, the same as in the brown kraft pulp (Table 8). The presence of silicon, iron, copper, and manganese (approximately 1 microgram per gram of pulp), increase the oxidation speed of cellulose and react with bleaching agents, affecting aging and producing a

yellowish color in cellulosic pulp (Rapson 1963; Macdonald and Franklin 1969; Grace *et al*. 1996), which may cause the low degree of bleaching obtained for the *E. crassipes* cellulosic pulp (Table 7).

CONCLUSIONS

1. *Eichhornia crassipes* kraft pulp yield is low.
2. The highest kraft pulp yield was with the combination of T = 120 °C and t = 30 min.
3. The residual lignin content in the brown pulp is high.
4. To minimize the residual lignin, the combination of T = 120 °C and t = 10 min should be used.
5. It was observed that during the *E. crassipes* pulping process, the obtained pulp darkened.
6. Cellulosic pulp contains a high concentration of inorganic substances.
7. Fiber classification of the brown pulp indicates a high amount of fines, which may cause problems in the drainage stage of paper sheet production.
8. The final bleaching obtained by the bleaching sequence is low, which may be due to the high content of inorganic substances in *E. crassipes* biomass.
9. Calcium, magnesium, iron, phosphorus and silicon, were the most abundant inorganic elements in ash from both brown and bleached pulp.
10. Finally, the biomass of *E. crassipes* could be a complementary raw material to cellulosic fibers from wood species to produce handmade paper.

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This paper is dedicated to the memory of Fermín Correa Méndez (Universidad Intercultural Indígena de Michoacán, México).

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