Evaluation of the Chemical Components of *Eichhornia crassipes* as an Alternative Raw Material for Pulp and Paper

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Eichhornia crassipes biomass collected in Lake Cuitzeo, Mexico was analyzed to determine the chemical components (pH, ash, ash microanalysis, extractives, lignin, holocellulose, and alpha cellulose) in the whole plant, as well as segmented analysis in roots, stems, and leaves. The plant contained an ash content of 14.3 to 20.7% and extractives content from 21.8 to 35.6%. The inorganic elements detected were potassium (K), chlorine (Cl), calcium (Ca), sodium (Na), magnesium (Mg), silicon (Si), aluminum (Al), phosphorous (P), sulfur (S), manganese (Mn), iron (Fe), and titanium (Ti). In addition, low amounts of lignin (12.5 to 25.7%) and holocellulose (26.7 to 37.1%) were obtained. Thus, *E. crassipes* biomass could complement cellulosic fibers in pulping processes of low yield, such as the fibers used to produce handmade paper.

Keywords: Ash; Extractives; Lignin; Holocellulose; Water hyacinth

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INTRODUCTION

Cellulose and paper production is mostly based on wood. However, a growth in paper demand, along with a decline in the supply of fibers from the world's forests is forcing the pulp and paper industry to find alternative sources of fibers that are both technical and economically viable (Jahan *et al*. 2008). Global paper and paperboard production has increased from 371 million tons in 2009 to 400 million tons in 2015 (FAO 2017). The global shortage of fibrous resources has aroused great interest in the use of non-conventional fibrous raw materials (weeds, shrubs, and non-timber), which can be used to obtain cellulose for paper production (Nagaty *et al*. 1982; Agarwal *et al*. 1992; Atchison 1996; Escoto *et al*. 2013).

Eichhornia crassipes (water hyacinth) is a floating aquatic plant native to the Amazon basin in Brazil (Barrett 1980). It is an invasive aquatic plant that has spread widely in tropical and subtropical regions of the world (Villamagna and Murphy 2010), with extremely rapid proliferation (Malik 2007). Shoyakubov and Aitmetova (1999) reported that the biomass of this plant increases 1 kg/m^2 per day, which amounts to 1,800 to 2,700 tons of wet raw material or 90 to 135 tons of dry biomass per hectare. In a given area, the plant may double its size within 5 days (Malik 2007), whereas the number of plants doubles within 4 to 58 days (Epstein 1998). A carpet of medium-sized plants may contain 2,000,000 plants per hectare, weighing 270 to 400 tons (Malik 2007), and over a period of 6 months, 125 tons wet weight are produced in an area of 1 hectare (Istirokhatun *et al*. 2015). This invasive plant causes severe ecological and economic impacts, such as loss of diversity of native species, hybridization with native species, alterations in ecosystem processes, increased pests and diseases, and serious challenges in navigation and irrigation systems, (Rodríguez 2006; Villamagna and Murphy 2010; Mahamadi 2011; Stiers *et al*. 2011; Nguyen *et al*. 2015).

Because *Eichhornia crassipes* has an alarming rate of reproductive capacity and propagation, it is 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). Thus, the aim of this study is to determine the chemical composition of this aquatic specimen and find out if it might complement wood pulp resources as alternative raw material for cellulosic paper fibers.

EXPERIMENTAL

Materials

Eichhornia crassipes (Mart.) Solms was harvested from Lake Cuitzeo in Michoacán, Mexico (19°53'15" Latitude, 100°50'20" Longitude). The collected aquatic plants were washed with abundant water. The roots, stems, and leaves were separated and dried under shade. These materials were then milled and sieved to obtain a 40-fraction mesh meal (425 micron). The initial moisture content and the moisture content after the outdoor drying was determined by exposing the biomass at 105 ± 3 °C, according to the TAPPI T 264 cm-97 method (2000). Statistica software (v. 7.0, Palo Alto, CA, USA) was used to run a one-way ANOVA test and the Tukey test ($\alpha = 0.05$) to compare means and evaluate results. Mean values and standard deviation were reported.

Chemical Analysis

The pH value (Sandermann and Rothkamm 1959), mineral content (TAPPI T 211 om-93 2000), ash microanalysis (Téllez *et al*. 2010), and extractives content (Mejía-Díaz and Rutiaga-Quiñones 2008) of the dry biomass were determined. The total extractives content was determined by successive Soxhlet extractions using the following solvents: cyclohexane, acetone, methanol, and hot water at reflux for 6 h with each solvent. The solvents were recovered in a rotary evaporator under vacuum, and the respective extractives were placed in a desiccator at constant weight. Lignin, holocellulose, and cellulose in the extractive-free biomass were determined following the procedures reported by Runkel and Wilke (1951), Wise *et al*. (1946), and ASTM D 1103-60 (1981), respectively.

RESULTS AND DISCUSSION

Initial Moisture

Initial moisture content in the plant and stem were statistically the same, whereas moisture content for roots and leaves was lower (Table 1). The percentages obtained for moisture in the plant, roots, and stems were compared to those reported by Dantas-Santos *et al*. (2012), who reported moisture content in roots to be 91.3%, stem (rhizome and petiole) 93.2%, leaf 86.55%, and slightly lower than the 95% reported for plants in other works (Kumar *et al*. 2009; Bergier *et al*. 2012; Fileto-Peréz *et al*. 2013; Reales-Alfaro *et al*. 2013). The biomass of plants and grasses contains more than 50% moisture based on their dry weight, but intrinsic moisture (water that forms part of the biomass structure) is much lower (Tanger *et al*. 2013). There may be significant correlations between moisture content and mineral content of plants, since they use mineral ions to modulate the osmotic potential of the cells (Patakas *et al*. 2002; Arjenaki *et al*. 2012). Therefore, the high mineral content in *Eichhornia crassipes* may be related to its high moisture content. Table 1 shows the effects of moisture content of the raw materials in the pulp and paper industry.

pH

Table 1 describes the effects of pH on the pulping process and pulp and paper industry, showing a slight statistical difference on the pH of roots. Lara-Serrano *et al*. (2016) reported more acidic pH values in parts of this aquatic plant, roots (4.6), stems (4.7), and leaves (4.7), which may be due to the collection site. As it is known, acidic pH values may cause corrosion problems on the pulping process and pulp and paper industry (Table 1), however that is not the case with *E. crassipes*, since the pH values found in this study were almost neutral.

Ash

The ash content in the plant, root, and stem was statistically similar with significant differences in leaves, as shown in Table 1. The percentages of ash through the plant decreased according to the following order, stems > roots > leaves, coinciding with the greatest mineral absorption in the stem (rhizome) (Mahmood *et al*. 2005). The percentage of ash in *E. crassipes* and through its parts varied depending on the site of collection. For *E. crassipes* collected at the Tunal River in Durango, Mexico, and Lake Yuriria in Guanajuato, Mexico, Lara-Serrano *et al*. (2016) reported the following values: 26 and 14.6% in roots, 26.8 and 14.4% in stems, and 19.9 and 12.4% in leaves, respectively.

Whole plant ash content reported for samples collected in Lake Chapultepec in Mexico City was 19.1%, and 22.9% in Tunal River in Durango (Fileto-Peréz *et al*. 2013). Whereas the ash percentage in *E. crassipes* was higher than that found on other aquatic plants such as *Typha dominguensis* (4.9%) and *Cyperus papyrus* (7.05%) (Escoto *et al*. 2013), which have also been suggested for manufacturing handmade paper. The percentage of minerals in *E. crassipes* was higher than the value reported for wood used in the production of cellulose pulp. The percentage of minerals was 0.5% according to Macdonald and Franklin (1969) and 0.1 to 0.8% according to Fengel and Wegener (1989). This could be due to *E. crassipes* natural property to absorb minerals and toxic metals from aquatic environments, resulting in higher ash content than wood (Table 1). This can result in low quality pulp and additional problems on the bleaching process.

Table 1. Initial Moisture, pH and Mineral Content in *E. crassipes*

Test	Plant	Roots	Stems	Leaves	Effect on pulp and paper industry
Moisture (%)	92.75 ± 0.62 ab	92.71 ± 0.34 b	93.61 ± 0.23 a	85.34 ± 0.34 c	High moisture content in raw materials increases the weight of the plants, and consequently the transportation cost. It can also influence the manufacturing efficiency of paper pulp (MacLeod 2007). Low moisture content affects diffusion of the cooking liquor, low yield, and high percentage of rejects (Giacomozzi 2010).
pH	6.52 ± 0.11 a	6.82 ± 0.06 b	6.53 ± 0.16 a	6.31 ± 0.01 a	Low pH in biomass can interact with certain metals and this could cause corrosion of equipment in the pulping process (Fengel and Wegener 1984)
Ash (%)	19.37 ± 0.12 ab	19.02 ± 0.37 b	20.78 ± 0.26 a	14.37 ± 0.29 c	Ash can inhibit the manufacture of pulp, affect its aging, and produce a yellowish color in the cellulosic pulp (Rapson 1963; Grace *et al*. 1996). It increases the oxidation rate of the cellulose during bleaching, reacting with the bleaching agents. Thus, bleaching quality is affected (Rapson 1963; Grace *et al*. 1996).
Values with different lowercase literals in line are statistically different ($p \leq 0.05$)					

Microanalysis of Ash

Inorganic elements detected in *E. crassipes* are presented in Table 2. The most abundant element in the plant, root, and leaves was potassium, followed by chlorine. The stem showed the highest concentration of chlorine, followed by potassium. Potassium is found in higher concentrations in the stem and leaves of *E. crassipes* than other inorganic elements (Adeoye *et al*. 2001; Lara-Serrano *et al.* 2016). In contrast, calcium, potassium, and magnesium are the main elements present in wood (Fengel and Wegener 1989). Lara-Serrano *et al*. (2016) reported the presence of sodium, magnesium, aluminum, silicon, prosphorus, sulfur, cholrine, postassium, calcium, manganese, and iron in roots, stems, and leaves of *E. crassipes* collected at different sites. Silicates (aluminum, iron, magnesium, and calcium) can cause problems on the surfaces of heat transfer equipment where black liquor is burned in the Kraft process (Grace *et al*. 1996). Iron, copper, and manganese (approximately 1 microgram per gram of pulp) affect aging and produce a yellowish color in cellulosic pulp (Rapson 1963). Furthermore, *E. crassipes* has a strong tolerance to, as well as efficient adsorption of, heavy metals and nutrients such as nitrogen, phosphorus, and potassium (Feng *et al.* 2017). Table 2 describes the effects of minerals that may have effects on the pulp and paper industry.

Table 2. Ash Microanalysis of *E. crassipes* (atomic %)

Element	Plants	Roots	Stems	Leaves	Effect on Pulp and Paper Industry
Potassium	29.22 ± 1.24 a	23.94 ± 0.97 c	28.47 ± 0.79 a	37.29 ± 0.41 b	Favors the formation of incrustations (Giacomozzi 2010). Affects the quality of white cooking liquor (Sithole and Allen 2002). High concentrations can cause problems in the chemical reagent recovery boiler (Doldán *et al*. 2011).
Chlorine	21.42 ± 1.16 a	9.85 ± 0.18 c	35.27 ± 0.52 b	24.94 ± 0.93 a	High concentrations corrode the equipment and obstruct the passage of gas in the recovery boiler (Ulmgren 1987; Backman *et al*. 1995; Sithole and Allen 2002; Ferreira *et al*. 2003; Doldán *et al*. 2011).
Calcium	13.01 ± 0.56 a	10.85 ± 0.56 c	11.72 ± 0.67 a	13.52 ± 1.72 b	Favors the formation of incrustations on the surfaces of heat exchange where the combustion of the black liquor is carried out in the Kraft process and in the digester Increases the corrosion in evaporator pipes (Libby 1980; Grace *et al*. 1996; Rabelo *et al*. 2001; Sithole and Allen 2002; Vakkilainen 2005; Giacomozzi 2010).
Sodium	9.38 ± 0.74 a	9.16 ± 0.32 a	11.34 ± 0.59 b	2.2 ± 0.39 c	Favors the formation of incrustations (Giacomozzi 2010).
Magnesium	7.86 ± 0.62 a	8.69 ± 0.66 b	7.68 ± 0.67 a	9.22 ± 0.79 b	Produces slaked lime in the recovery cycle (Rabelo *et al*. 2001; Sithole and Allen 2002; Vakkilainen 2005).
Silicon	7.48 ± 1.19 b	18.48 ± 0.92 c	0.37 ± 0.11 a	0.91 ± 0.15 a	Affects the quality of bleaching (Rapson 1963). Produces slaked lime in the recovery cycle (Sithole and Allen 2002). They can generate fouling on heat transfer surfaces in the digester plant, chimneys and evaporators (Libby 1980; Rabelo *et al*. 2001; Vakkilainen 2005).
Aluminum	1.65 ±0.32 b	5.01 ±0.84 a	nd	nd	Favors the formation of incrustations (Giacomozzi 2010), increases corrosion on heat transfer surfaces in digestors, and can cause problems on black liquor boilers and evaporator pipes (Grace *et al*. 1996; Rabelo *et al*. 2001; Vakkilainen 2005). Produces slaked lime in the recovery cycle (Rabelo *et al*. 2001; Vakkilainen 2005).
Phosphorus	4.22 ±0.45 b	3.33 ±0.63 a	3.63 ±0.57 a	7.88 ±0.89 c	Produces slaked lime in the recovery cycle (Rabelo *et al*. 2001; Vakkilainen 2005; Tran and Vakkilainen 2007).
Sulfur	3.7 ± 0.52 b	6.04 ± 0.54 a	1.47 ± 0.23 d	2.1 ± 0.42 c	May cause problems in the chemical recovery system (Doldán *et al*. 2011).

Manganese	nd	0.88 ± 0.11	nd	nd	Affects aging and produces yellowish color in cellulose pulp (Rapson 1963). Has inhibitory effects on the reversal of gloss in cellulose (Rapson 1963). Causes negative effects in the bleaching and hydrolysis stages of the cellulose chains (Sjöström and Välttilä 1978; Lachenal *et al*. 1980; Soinnii *et al*. 1998; Torres *et al*. 2005; Giacomozzi 2010).
Iron	2.01 ± 0.33 a	3.54 ± 0.51 b	nd	nd	High content corrodes the pulp digester and equipment for evaporation, heat transfer, and on black liquor boilers (Grace *et al*. 1996; Torres *et al*. 2005). Affects aging and produces yellowish color in cellulosic pulp (Rapson 1963; Libby 1980). Causes reversal of gloss in cellulose (Rapson 1963). Causes negative effects in the bleaching and hydrolysis stages of the cellulose chains (Sjöström and Välttilä 1978; Lachenal *et al*. 1980; Soinnii *et al*. 1998; Torres *et al*. 2005; Giacomozzi 2010).
Titanium	nd	0.18 ± 0.04	nd	nd	no references were available
Values with different lowercase literals in line are statistically different ($p \leq 0.05$)					

Extractives Content

The percentages of extractives presented significant differences in each section of the plant (Table 3). The total extractives yield decreased from leaves > stems > roots. There were also significant statistical differences between the solvents used. The highest solubility was presented with the most polar solvents (methanol and water), coinciding with Lara-Serrano *et al*. (2016), who applied the same extraction sequence to this aquatic plant. Joedodibroto *et al*. (1983) used only hot water for the extraction stage and reported the following values: less than 24.71% for a mixture of stem-leaves and 28.56% for stem. Therefore, using extractive stages led to a higher efficiency of extractives. Extractive content of the most common pine species used in the pulp and paper industry range from 2.4 to 7.7% (MacDonald and Franklin 1969; Fengel and Wegener 1989) which coincides with values found in Mexican pine species, ranging from 7.6 to 8.2 (Bernabé-Santiago *et al*. 2013) and 6.3 to 7.07 (Pintor-Ibarra *et al*. 2017). As shown in Table 3, such values were much higher in *E. crassipes* than in *Pinus* species, which makes *E. crassipes* unsuitable to produce cellulose pulp. Fundamental research was done for wood as raw material for pulp and paper production in order to determine the effects of extractives on the pulping process (Rapson 1963; MacDonald and Franklin 1969; Fengel and Wegener 1984). In particular, *E. crassipes* has been studied to determine its pulp quality as an alternative raw material for paper production (Kumar *et al*. 2015), but in such study there is no discussion on the effects of its extractives. Therefore, this is the first study that researches why the cellulosic pulp yield of *E. crassipes* is so low and discusses the effects of extractives on the pulping process. Furthermore, extractives might also be used as a source of byproducts in commercial processes (Fileto-Pérez *et al*. 2015).

Table 3. Extractives Content in *E. crassipes* (%)

Extractives	Plant	Roots	Stems	Leaves	Effect on pulp and paper industry
Cyclohexane	2.21 ± 0.25 a	1.38 ± 0.26 b	1.78 ± 0.01 ab	3.14 ± 0.32 c	High extractives content may limit its use as pulp for paper since low-content raw materials are preferred (MacLeod 2007). Extractives could be a disadvantage in the pulping processes as they inhibit the manufacture of pulp, and certain extractives produce yellowing of the cellulose (Rapson 1963; MacDonald and Franklin 1969; Fengel and Wegener 1984); they cause problems in the bleaching stages (Rapson 196; MacDonald and Franklin 1969). Certain components of the extractives form complex with iron that can accelerate the corrosion of the cooking digester and participate in the corrosion of certain metals (MacLean and Gardner 1953; MacDonald and Franklin 1969; Libby 1980; Grace *et al*. 1996). Some extractives have been shown to inhibit sulphite pulping. However, the kraft pulping process produces extracts as byproducts that are commercially used (Grace *et al*. 1996). Some extractives combined with lignin may be resistant to delignification in pulping processes (David and Shiraishi 1991). Low extractives content decreases light absorption in paper fibers (Rapson 1963).
Acetone	1.65 ± 0.15 a	0.51 ± 0.15 c	1.62 ± 0.22 a	2.47 ± 0.08 b	
Methanol	14.33 ± 0.85 a	9.08 ± 0.92 c	17.96 ± 0.89 b	13.01 ± 0.46 a	
Hot water	13.78 ± 1.1 a	10.83 ± 0.7 c	13.60 ± 0.001 a	17.05 ± 0.32 b	
Total Extractives	31.98 ± 1.1 b	21.82 ± 0.73 c	34.97 ± 0.98 ab	35.69 ± 0.22 a	

Values with different lowercase literals in line are statistically different ($p \leq 0.05$)

Runkel Lignin

Runkel lignin values showed significant differences regarding each part of the plant (Table 4). The highest amount of lignin was present in leaves, followed by the roots, and finally the stem showed the lowest value. Lara-Serrano *et al*. (2016) also reported a lower percentage of Runkel lignin in the stem compared to the roots and leaves. Additional research on this aquatic plant shows less lignin in the stem (8.67%) and more in leaves (23.54%) (Joedodibroto *et al*. 1983). As for the whole plant, lower values of lignin obtained by another method have been reported, varying from 3.8 to 5.3%, depending on the collection site (Fileto-Peréz *et al*. 2013). The content of lignin in *E. crassipes* is low compared to that of the wood, which varies from 10.2 to 29.8% for different wood species (Fengel and Wegener 1989), and has a value of 25% for *Pinus* spp (MacDonald and Franklin 1969). Table 4 describes the effects of lignin in the pulp and paper industry.

Table 4. Lignin and Polysaccharide in *E. crassipes*

	Plant	Roots	Stems	Leaves	Effect on pulp and paper industry
Runkel lignin	19.95 ± 0.74 b	22.64 ± 0.8 ab	12.54 ± 0.02 c	25.70 ± 1.55 a	Low lignin content in lignocellulosic materials is desirable because it provides higher pulp yield and faster delignification (MacLeod 2007). Low lignin content in the pulp decreases the absorption of light, increases the bonding capacity on the surface of the fibers, flexibility, swelling (Rapson 1963).
Holocellulose	34.21 ± 0.19 a	32.94 ± 1.17 a	37.17 ± 0.76 b	26.78 ± 0.07 c	High content of cellulose and hemicelluloses favors yields in pulping processes (MacLeod 2007). However, high content of hemicelluloses can have negative effects because they can be hydrolyzed by alkaline attack during pulping (Rapson 1963; Libby 1980; Grace *et al*. 1996; MacLeod 2007). The color reversion of the bleached pulp is related to the high hemicelluloses content (Rapson 1963; Libby 1980). Hemicelluloses have a significant influence on the properties of fibers in papermaking, a high hemicellulose content increases the binding capacity of the fibers surface and the ease with which the fibers swell (Rapson 1963; Libby 1980). Hemicelluloses contribute greatly to the tensile, blast, and fold resistance properties of the paper sheet (Libby 1980).
Alpha cellulose	19.45 ± 0.19 c	27.94 ± 0.35 d	23.86 ± 1.42 b	21.94 ± 1.2 ab	High values of alpha-cellulose are favorable in the cooking processes in the production of cellulose pulp for paper (Libby 1980). Alpha-cellulose is relatively resistant to the extensive dissolution of pulp chemicals and the oxidizing agents used in bleaching (MacDonald and Franklin 1969; Libby 1980). It favors the whiteness of the whitened pulp, even with age (Rapson 1963).
Values with different lowercase literals in line are statistically different ($p \leq 0.05$)					

Holocellulose

Holocellulose represents the total fraction of polysaccharides; these components are made up of cellulose and hemicelluloses (MacDonald and Franklin 1969; Fengel and Wegener 1984). The highest percentage of holocellulose in *E. crassipes* was found in the stems (37.17%), followed by the roots (32.94%), and finally the leaves (26.78%) (Table 4). Dantas-Santos *et al*. (2012) reported minor carbohydrate values in roots (13.6%), stems (13.7%), and leaves (11.9%). Nonetheless, the percentage of holocellulose in wood is 70%, specifically 50% cellulose and 20% hemicelluloses (Macdonald and Franklin 1969), and 70 to 74% for the genus *Pinus* (Fengel and Wegener 1984). Table 4 shows some effects of cellulose and hemicelluloses in the pulp and paper industry. The high proportion of polysaccharides (cellulose and hemicelluloses) in the lignocellulosic materials favor the pulp yield for paper (MacLeod 2007), therefore the percentage obtained from holocellulose in *E. crassipes* (34.21%) favors little yield of cellulosic pulp. Lara-Serrano *et al*. (2016) also warn that if *E. crassipes* is used as raw material in the cellulosic pulping process, pulp yield would be low due to low polysaccharide content in this aquatic plant.

Cellulose

Alpha cellulose represents the non-degraded, high molecular weight cellulose fraction. The percentages of alpha cellulose through the sections of *E. crassipes* found are presented are Table 4. The values of alpha cellulose found were decreasing according to the following: roots > stems > leaves. Lara-Serrano *et al*. (2016) recorded the same tendency in roots, stems, and leaves of *E. crassipes*. In other aquatic plants, lower values of alpha cellulose have been recorded: 17.91% in *Cyperus papyrus* and 15.23% in *Typha domingensis* (Escoto *et al*. 2013). The effects of alpha cellulose in the pulp and paper industry are described in Table 4.

CONCLUSIONS

1. The basic chemical components of *Eichhornia crassipes* presented statistical differences among its roots, stems, and leaves sections.
2. Moisture content is a limitation since it increases the weight of the plants, and consequently the transportation cost if this material is intended to be used in a pulping process.
3. High percentages of ash, extractives, and low percentage of holocellulose affect the pulping yield in a negative way. In addition, inorganic elements found in *E. crassipes* may cause problems during the manufacture of cellulosic pulp and affect the bleaching stages. Whereas a low percentage of lignin contributes to rapid delignification.
4. *E. crassipes* biomass might complement cellulosic fibers in low yield pulping processes, such as the cellulosic pulp used to make handmade paper.

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