Evaluation of Process Variables Influence over Cellulose Extraction from Vegetable Waste using Response Surface Methodology

Liziane da Luz Seben*, Istefani Carísio de Paula*

* Production and Systems Department, School of Engineering, University of Rio Grande do Sul, Porto Alegre, Brazil

Email: liziprod@producao.ufrgs.br, istefani@producao.ufrgs.br

Abstract

The use of vegetable wastes as raw material for cellulose fibers production has revealed to be an attractive alternative to residue's valorization and reduction of environmental impact. Canned heart palms production (*Archontophoenix alexandrae*) in Brazil generates a large amount of wastes which could be a source of cellulose. With the aim to get a pulp extraction method adjusted to the canned heart palms processes' wastes, the objective in this paper is to develop a Response Surface Methodology (RSM) to evaluate the influence of the process variables over the cellulose pulp characteristics. Response variables selected for experimental design included the degree of delignification, capability and speed of water absorption, and fiber density. The pulp cellulose absorption characteristics were defined from specifications for use of it in hygiene products as diapers, for instance. Statistical tools were used to determine the influences of controlled variables over the response variables through the formulation of n-dimensional equations and surfaces, which allowed the optimization of the interest variables. The results have indicated that the pulps obtained from the processes proposed have not reached the quality characteristics required for the use intended. The values for the absorption variables showed to be lower than those established in the research hypotheses, but the RSM essays have established basis for further experiments accomplishment.

Keywords: Vegetable wastes; Canned heart palm; Response Surface Methodology (RSM); Pulp extraction; Design of Experiments.

1 Introduction

Design approaches that emphasize the economic, social and environmental dimensions, associated with advances in technology, make the efforts to find sustainable solutions more effective. One aspect referred to in the sustainable approaches is the reuse of process waste, as well as the minimization of its generation (PAULI, 1996; McDONOUGH and BRAUNGART, 2002). Waste recovery for the purpose of generating new products has been an object of research, since there is a significant variation in the quality of these products and due to the need to design processes that can efficiently effect this transformation. The most attractive aspect in relation to natural fibers is their positive environmental impact, since they represent a renewable resource and are produced with low energy consumption (JOHN and THOMAS, 2008). In line with this trend is the proposal to obtain cellulose from King Palm (*Archontophoenix alexandrae*) leaf sheaths, thereby taking advantage of and recovering an abundant by-product which results from the industrial processing of canned heart of palm. Three layers of leaf sheaths that surround the heart of palm constitute the waste from this process. The leaf sheaths of the King Palm are an agricultural waste, which are lignocellulosic in nature, like most of these types of plant waste (KEREM et al., 1992).

Several studies have been conducted to investigate different applications for the waste from the King Palm, since they can add value to waste left behind on the field and reduce environmental damage resulting from its accumulation (TONINI, 2004). Noteworthy among the research studies is the application of the middle leaf sheath for food products (TONINI, 2004; VIEIRA, 2006). In a research performed at the Rural Federal University of Rio de Janeiro (UFRRJ), the use of the *Euterpe edulis Martis* stem, was

investigated as a fibrous raw material alternative for producing cellulosic pulp by the kraft method (ANDRADE et al., 2000).

To carry out such a project, the leaf sheaths must undergo cellulose extraction using specific equipment. Normally, the most industrially-used chemical pulping of lignocellulosic raw materials employs alkaline reagents for transforming the fresh fiber into cellulose pulp (SIXTA, 2006; SOUZA et al., 2008). Pulping with soda (NaOH) and anthraquinone (AQ) as an additive is suitable and common for pulping processes in plants that are not subject to seasonality (ANTUNES et al., 2000). A variety of references can be found on treating plant waste with alkaline extraction using NaOH and even an additive for delignification, including: rice straw pulping (RODRÍGUES et al., 2010), corn husks pulping (REDDY and YANG, 2005), soybean husk pulping (REDDY and YANG, 2009) and banana fiber pulping (ELANTHIKKAL et al., 2010). Few researches were found for King Palm leaf sheaths (TONINI, 2004), but no specific study on cellulose fiber extraction.

Therefore, the object of study in this research is to develop a Response Surface Methodology (RSM) to evaluate the influence of the process variables over the cellulose pulp characteristics using alkaline pulping for King Palm leaf sheaths. This process uses NaOH as a delignifcation reagent and AQ as a process additive. Then an experimental design was proposed to examine the feasibility of extracting cellulose from King Palm leaf sheaths in order to obtain a fiber with absorbent capacity and low residual lignin content, as well as demonstrate the influence of the controllable process factors on the response variables analyzed.

2 Theoretical Review

This review deals with King Palm leaf sheaths and cellulose extraction methods. In the literature on lignocellulosic materials, there are few scientific publications available about the properties and behavior of their fibers.

2.1 King Palm

The high productivity of King Palm leads to the generation of large amounts of plant waste from processing. The heart of palm extraction process requires cutting down the palm tree, because only a small part inside the stem is used for commercial purposes and food consumption. Thus, recovering the waste that is generated is an attractive alternative for fomenting development in the sector, aligned with sustainability concepts, such as zero waste generation principles (PAULI, 1996; McDONOUGH and BRAUNGART, 2002). The main constituents of the plant are cellulose, hemicellulose and lignin (OKEKE and OBI, 1994). Of the different agro-industrial wastes with the potential to be used in bioprocesses, the middle sheath of the heart of palm, which has a high concentration of lignocellulosic material (TONINI, 2004), has been still little studied.

2.2 Industrial cellulose extraction processes

There are two alkaline pulping processes are the soda process and kraft process. In both NaOH is the largest delignifcation reagent in the cooking liquor, while the kraft process also contains sodium sulfide (Na_2S). The use of process additives has been a highly regarded option in alkaline processes since it enhances the efficiency of the pulping reactions and reduces the active alkali load and sulfur compounds. Studies with soda process showed that the action of AQ as an additive affords greater benefits in the pulpings done with a lower NaOH concentration, since the action of this additive can be masked by the presence of OH- ions in higher concentrations. These studies indicate that the effect of AQ is more pronounced in soda pulping as compared to kraft pulping (HOLTON, 1977).

The advantages of the alkaline processes over the others, lie in their adaptability to all types of wood for producing high quality pulps with excellent resistance properties, in addition to having an efficient system for recovering chemical reagents and energy. The soda process with the use of additives proves to be potentially favorable for application when the use of sulfur is restricted (SOUZA et al., 2008). In regards to

the concerns about applying this process, it should be noted that the excessively long cooking time, high temperatures and high NaOH concentrations used can promote the degradation of the wood carbohydrates, reducing the yield of the process (D'ALMEIDA, 1988; SIXTA, 2006). In industrial practice, temperatures in the 170°C range are recommended, but, according to D'Almeida (1988) the time and temperature factors are interdependent. If a higher pulping temperature is used, a shorter time period can be selected, or the opposite. Thus, process conditions are sought that promote delignification without excessive degradation of the cellulose fibers.

2.3 Design of Experiments

A design of experiments will be efficient if a scientific approach is applied in its planning. The planning process of the experiment is designed through the integration of statistical methods, so that the data collected can be analyzed statistically, leading to objective and valid conclusions (MONTGOMERY, 2001). Many kinds of design of experiments are suggested.

2.3.1 Response Surface Methodology (RSM)

Response Surface Methodology (RSM) is a combination of experiment design techniques, regression analysis and optimization methods that is widely applied in industrial surveys where a large number of variables of a system influences a fundamental feature of this system. According to Ribeiro (2007) (RSM) is well-suited for application in experiments where the intent is to optimize products and processes, because it generates the mapping of regions modeled through n-dimensional surfaces. For applying this methodology, Central Composite Design (CCD) offers certain advantages, in that it is recommended for the adjustment of quadratic models, making possible the study of linear and quadratic terms. Likewise, the alpha value can be set enabling the design to acquire properties of rotationality or orthogonality. In multiple regression equations, in order to compare the effects of the model's different controllable factors (terms), it is necessary to standardize their variation interval. The standardization is done by converting the actual levels of the investigation interval into coded levels of the interval.

3 Method

For the experimental research, the waste resulting from the processing of heart of palm was used, which consists of the leaf sheaths that surround it. Until now only the leaf sheaths are gathered by the company and the other elements remain on the field (stem, trunk and leaves), that is, on the properties of the heart of palm suppliers. In light of this, the study proposes utilizing the King Palm leaf sheaths, with the shipping cost built into the heart of palm processing cost, in other words, without extra expenses for the extraction process. From the results of the preliminary alkaline pulping tests on King Palm leaf sheaths, performed in a processing company in another study, the possibility was evaluated of conducting experimental designs in equipment used to extract cellulose from plant materials for bench-scale pulp production. It is important to mention that previous studies with the waste from the canned heart of palm process found that treatment via acid hydrolysis was a means of generating a product with high commercial value – inulin. This study is found in Patent 0606063-3 (FEPAGRO, 2008). Inulin, a natural sweetener, is present in the filtered liquor following the acid hydrolysis of the palm leaf sheaths.

Thus, the chemical pulping of the leaf sheaths, proposed in this paper, was developed in two ways: from fresh leaf sheath waste and after undergoing acid hydrolysis. The fresh crushed raw material was dried in an oven at 68°C and set aside in a container to be used during the tests. It was considered that the raw material, before drying, should have firm fibers and not be in a stage of decomposition. Similarly, for the acid hydrolysis stage, the already dry fresh raw material underwent the hydrolysis reaction in several rounds, after which it was dried in an oven, mixed and homogenized to then be used in the alkaline extraction tests. The acid hydrolysis process of the leaf sheaths (upper third of the stem of the palm tree from where heart of palm is removed) entailed attacking the fiber with an acid solution diluted in water. The conditions for the acid hydrolysis according to Patent 0606063-3 (FEPAGRO, 2008) are the following: liquid-sample ratio (10:1), acid concentration (3% v/v), pressure (1,5 atm), temperature (127ºC) and time in

the autoclave at the stipulated temperature (30 min) .

The experiment designed for alkaline extraction was a CCD, 2^k with star points and center points. The pulping temperature had to be maintained at 127°C, corresponding to the maximum operating limit that the equipment used was able to support. Reaction time, NaOH and AQ concentration were the controllable factors of the process, while the kappa number, absorption capacity, absorption speed and apparent pulp density were the response variables analyzed. The levels investigated, according to industrial parameters and those available in the literature (SMOOK, 1982; D'ALMEIDA, 1988; KHRISTOVA et al., 2004; SIXTA, 2006; KHIARI et al., 2009; RODRÍGUES et al., 2010). The intervals were established according to the CCD with an alpha value of 1.68 and two center points. The study points and their respective coded levels are listed in Table 1.

Table 1 - Points and controllable factors, in their high and low levels, and center point for the Central Composite Design (CCD)

Factors	***CONTROLLABLE FACTORS***	***Real Levels***		***Center Level***	***Star Level***	
		Low Level	**High Level**			
		(-1)	(+1)	(0)	(-1.68)	(+1.68)
X1	AQ concentration (%)	0.5	1.0	0.75	0.33	1.17
X2	Time (min)	120	150	135	110	160
X3	NaOH concentration (%)	15	29	22	10	34

Table 1 shows the controllable factors, intervals used and their respective levels analyzed in the experiments. The order of the trials was randomized and without repetition.

3.1 Laboratory description

The treatments were performed in the Plant Pathology laboratory located at FEPAGRO (Porto Alegre, RS, Brazil). Two kind of treatment were performed: acid hydrolysis reaction and alkaline pulping, and the first followed the patent recommendations. For both treatments it was used a vertical autoclave (BIOENG brand, model A50, number 274). The samples that had the acid hydrolysis treatment were neutralized in an aqueous medium with NaOH until reaching a pH close to 7.0 and then placed in an oven (BIOMATIC brand) for drying, in order to serve as raw material for the second round of experiments with alkaline treatment.

Following the preparation of the hydrolyzed material, the trials for cellulose extraction followed the CCD design points. First, the quantities of straw and water used in each experiment were determined. The trials were performed with 50 g of dry material and 500 ml of solution. The alkaline solutions were prepared from dissolving NaOH and AQ in distilled water, as per the predefined concentrations. The weighing of 50 g of plant material, whether hydrolyzed or fresh, after drying, was done on a digital analytical BEL MARK 500 scale with a maximum capacity of up to 500 g. The drying of the plant material was done in the same oven used in the previous stage, at a constant temperature of 68°C to constant weight. The autoclave available for carrying out the tests was regulated to operate at a constant temperature (127°C) and pressure (1.5 atm), which represent the maximum parameters permitted for operating the equipment. The autoclave has a 20-liter capacity and contains a basket which is capable of supporting two two-liter beakers. The experiments were done by placing the straw in heat-resistant beakers and then adding the alkaline solution at the set concentration. The equipment is used for bench-level experiments, since despite its small size it can reproduce industrially-applicable process parameters.

Once the experiment began, control over the time factor was maintained. The time established for each point was measured from the time the system reached a temperature of 127°C. At the end of the cooking time, the equipment was opening to remove the beakers, and then they were cooled. The material inside the beakers was separated, since it consisted of pulped material and black liquor. The pulp was filtered and washed three times with distilled water before undergoing a drying process. Drying took place in an

oven, whose temperature was maintained at 68°C for 24 hours. The black liquor was neutralized for later disposal. The solid part was analyzed to determine the response variables.

3.2 Analytical methods

Samples were taken in order to analyze the response variables: Kappa number, absorption capacity (g of water), absorption speed (g of water/s) and apparent pulp density (g/cm^3). All the samples were dried after collection and sent for analysis, which was done once for each sample. The analysis of the response variables was conducted at SENAI (Telêmaco Borba, PR, Brazil), which provides a specialized service for testing paper and cellulose pulp. It's worth noting that proposed analyses were a laborious and expensive task. The methods used for measuring each response variable are detailed described below.

3.2.1 Methodology for determining the Kappa number

This analysis provides an indication of the degree of pulp delignification. The analyses for determining the kappa number of pulp from King Palm leaf sheaths proceeded according to ABNT NBR ISO 302 (2005). To determine the kappa number, a gram of dried pulp was used for each test, and the tests were performed in triplicate for each sample. For more details see ABNT NBR ISO 302 (2005) specifications. Interval specification for kappa number in commercial sanitary pulps is 5 to 10, and the target value can be considered 8.

3.2.2 Methodology for determining water absorption capacity and time

For both the absorption capacity and absorption speed tests, 5 grams of cellulose are used, and the tests are carried out in that sequence, just once for each sample. The analyses proceeded in accordance with ABNT NBR 15004 (2003). Interval specification for absorption capacity is 8 to 14 g of water/ g dry pulp (FOELKEL, 2010), and he target value considered was 10 (g of water/ g dry pulp). Considering the absorption speed the interval specification for it is 3,2 to 20 g water/s (FOELKEL, 2010) and the he target value was 12 g water /s.

3.2.3 Methodology for determining apparent density

The determination of apparent density was done in accordance with ABNT NBR NM - ISO 534 (2006), with a 1.6 gram mass of cellulose fiber. The tests were performed just once for each sample and he interval values are 0,4 to 0,6 g/cm^3 (FOELKEL, 2010). The target value estipulated was 0,55 g/cm^3.

3.3 Obtaining the regression model and the Response Surface graphics

In multiple regression equations, in order to compare the effects of the model's different controllable factors (terms), it is necessary to standardize their variation interval, so they are codified. Using CCD, it was necessary to code each of the investigated controllable factors at high levels (+1) and low levels (-1), the center point (0) and the star points (± 1.68). The values of the star points (alpha) were calculated in order to confer the orthogonality condition to the project. All the results were treated with Minitab 15 statistical software in order to statistically evaluate, to the 15% significance level, which factors and interactions among the controllable factors have significant influence on the values obtained for the response variables. Response Surface graphics were generated on the basis of the influence of pairs of coded controllable factors on each of the response variables. They provided to define optimal conditions to the response variables.

4 Results and Discussion

Absorbent materials such as paper for sanitary purposes must display certain important characteristics to ensure the efficiency of their function. According to Jordan and Neves (1989), the most relevant properties of this kind of pulp are: high absorption speed and the ability to retain or absorb liquids. However, the pulp's ability to retain liquids is tied to its fibrous structure and arrangement thereof. The author states that the fibers of the material need to be long, porous and rigid. Foelkel (2010), who has studied the behavior of pulps for absorbent purposes, the specific volume, which corresponds to the inverse of the

density, is also an important feature. Pulps with a high specific volume or low density generally exhibit high water absorption.

In order to determine these parameters for the pulp obtained from the process waste (leaf sheaths), experiments were performed from November to February 2010. Sixteen tests were performed with fresh material and another sixteen with hydrolyzed material, which generated 32 different samples that were analyzed in a specialized laboratory. Only one test was performed for each experimental condition, since these are cumbersome and require a lot of time to perform. The results obtained for the response variables for each treatment, without and with acid hydrolysis of the raw material, are listed in Table 2 and 3, respectively. The statistically significant linear and quadratic effects, at the 15% level, were noted for the response variables. The value was set according to data presented in the literature for working with experiments with vegetable sources that involve chemical reactions (REZAYATI-CHARANI and MOHAMMADI-ROVSHANDEH, 2005; REZAYATI-CHARANI et al., 2006; ZENG et al., 2011). Researchers performed pulping with NaOH and AQ of palm fruit bunches after extracting the oil, through a design of experiments and with the generation of regression models to predict the value of the variables of interest of the pulp, accepting a statistical significance level of up to 20% (JIMÉNEZ et al., 2009).

Table 2 - Process factors and response variables for extracting cellulose with sodium hydroxide and additive

	CONTROLLABLE FACTORS AND CODING OF THE FACTORS						**RESPONSE VARIABLES**			
POINT	**AQ Concentration (%)**		**Time (min)**		**NaOH Concentration (%)**		**Kappa No.**	**Absorption Capacity[g]**	**Absorption Speed [g/s]**	**Apparent Density [g/cm3]**
1	0.5	-1	120	-1	15	-1	51.38	3.52	0.71	1.17
2	1	1	120	-1	15	-1	41.27	2.17	0.17	1.34
3	0.5	-1	150	1	15	-1	46.18	2.58	0.16	1.36
4	1	1	150	1	15	-1	66.50	2	0.51	1.27
5	0.5	-1	120	-1	29	1	42.49	2.49	0.14	1.3
6	1	1	120	-1	29	1	41.16	2.44	0.28	1.23
7	0.5	-1	150	1	29	1	61.68	3.82	0.6	1.07
8	1	1	150	1	29	1	60.04	4.83	0.91	1.21
9	0.33	-1.68	135	0	22	0	51.87	2.92	0.53	1.36
10	1.17	1.68	135	0	22	0	51.10	2.36	0.16	1.29
11	0.75	0	110	-1.68	22	0	48.40	3.43	0.45	1.28
12	0.75	0	160	1.68	22	0	50.48	1.8	0.02	1.24
13	0.75	0	135	0	10	-1.68	51.43	2.75	0.65	1.24
14	0.75	0	135	0	34	1.68	62.39	2.04	0.47	1.15
15	0.75	0	135	0	22	0	60.45	2.34	0.55	1.2
16	0.75	0	135	0	22	0	62.00	2.98	0.6	1.22

Table 3 - Process factors and response variables for extracting cellulose with sodium hydroxide and additive after acid hydrolysis

	CONTROLLABLE FACTORS AND CODING OF THE FACTORS						RESPONSE VARIABLES			
POINT	**Concentration AQ (%)**		**Time (min)**		**Concentration NaOH (%)**		**Kappa No.**	**Absorption Capacity [g]**	**Absorption Speed [g/s]**	**Apparent Density [g/cm3]**
1	0.5	-1	120	-1	15	-1	66.12	3.23	0.38	1.19
2	1	1	120	-1	15	-1	47.61	2.93	1.04	1.17
3	0.5	-1	150	1	15	-1	50.23	3.3	0.58	1.39
4	1	1	150	1	15	-1	61.5	4.05	0.8	1.34
5	0.5	-1	120	-1	29	1	43.73	3.48	1.47	1.12
6	1	1	120	-1	29	1	35.28	4.12	0.71	1.14
7	0.5	-1	150	1	29	1	43.27	3.73	1	1.21
8	1	1	150	1	29	1	49.23	3.32	1.36	1.06
9	0.33	-1.68	135	0	22	0	38.76	4.53	1.45	1.12
10	1.17	1.68	135	0	22	0	54.86	4.53	0.74	1.14
11	0.75	0	110	-1.68	22	0	25.17	3.86	0.17	1.27
12	0.75	0	160	1.68	22	0	22.41	4.13	0.37	1.45
13	0.75	0	135	0	10	-1.68	60.89	3.14	0.64	1.2
14	0.75	0	135	0	34	1.68	43.38	3.41	0.52	1.3
15	0.75	0	135	0	22	0	39.51	2.83	1.24	1.49
16	0.75	0	135	0	22	0	39.39	3.31	1.36	1.3

Once the laboratory results were obtained, a modeling was done through multiple regression, using only the significant effects indicated by the statistical analysis. Table 4 shows the equations generated by the experiments that only had the alkaline extraction stage.

Table 4 - Proposed models for the response variables of the experiments with alkaline extraction

Variable (Y)	Equation	R2 - coefficient of determination	p - value
Kappa number	Y= 61.298 + 4.51X2 – 4.342 X22	69.40%	X2: 0.059; X22: 0.114
Absorption capacity	Y= 2.6177 + 0.6038 X2X3	54.30%	X2X3: 0.090
Absorption speed [g/s]	Y= 0.5686 + 0.1625 X2X3	60.60%	X2X3: 0.113
Apparent density [g/cm3]	Y= 1.2119 - 0.0352 X3 – 0.0462 X2X3	61.90%	X3: 0.139; X2X3: 0.137

For the response variables (Y) it is possible to see the positive or negative influence through the signal "+" or "-", respectively, and the interactions and linear influences by the factors (X1, X2 and X3) in the

equations. However, the R^2 values which indicate how well the regression equation fits the sample data, is between 54.3 to 69.4%, and is the highest value found for the kappa number variable. This indicates that the models reveal a greater influence of external factors. Since the experiments were performed without repetition, it can be expected that the sample data generated will not be sufficient to describe models that manifest the influence of the controllable factors on the response variables analyzed. Table 5 presents the equations generated by the software for the response variables of the experiments that also had the acid hydrolysis stage followed by the alkaline extraction stage.

Table 5 - Proposed models for the response variables of the experiments with alkaline extraction after acid hydrolysis

Variable (Y)	***Equation***	***R2 - coefficient of determination***	***p - value***
Kappa number	Y= 38.618 - 6.107X3 + 4.61 X12 + 6.493 X32 + 5.524 X1X2	81.60%	X3: 0.034; X12: 0.141; X32: 0.054; X1X2: 0.108
Absorption capacity	Y= 3.1149 + 0.4077X12	67.30%	X12: 0.042
Absorption speed [g/s]	Y= 1.2702 – 0.2923X22	59.30%	X22: 0.080
Apparent density [g/cm3]	Y= 1.3984 + 0.05 X2 - 0.1019X12 – 0.0595 X32	74.50%	X2: 0.116; X12: 0.022; X32: 0.122

The regression models provide information about the linear effects and interactions. The regression analysis for the kappa number showed that the NaOH concentration influences in a linear way, causing the kappa number to decrease if there is any increase in the NaOH concentration. Data from the literature strengthen the proposed model, since an increase in the concentration of NaOH leads to a decrease in the kappa number, indicating a higher degree of delignification. On the other hand, studies have also found that the presence of a small amount of liquor affects the kappa number (ABNT, 2005), and for this reason, if the washing of the sample is insufficient, this variable can increase.

Based on the generated regression equations, which highlight the significant coefficients for the model, software was used for generating Response Surface graphs which highlight the interaction of pairs of controllable factors or even the quadratic effects for each response variable. These graphs with the effects of the factors are presented in:

https://docs.google.com/open?id=0B8VVSr93P98ibTZJY09oNTZRNzJtYWh1NW5hRjJIZw.

For the tests that only entailed alkaline extraction, the analysis of the AQ concentration versus time graph for the Kappa number is sloped, indicating the quadratic effect of the time variable. For the absorption capacity, absorption speed and apparent density variables, the graphs show the behavior of the interaction of time versus NaOH concentration. Meanwhile, the graphs of the experiments that contained the acid hydrolysis reaction followed by alkaline extraction, for the Kappa number, show the quadratic effects of the AQ and NaOH concentrations through the AQ concentration versus NaOH concentration graph. The positive interaction between the AQ concentration and time variables is shown through the graph with both factors.

For the absorption capacity variable, the quadratic effect of the AQ concentration variable is seen through the AQ concentration versus time interaction. The same graph was constructed for the absorption speed variable in order to reveal the quadratic effect of the time factor. These results indicate that it would be beneficial to find an optimal value between the time and AQ concentration factors, since both of them influence absorption capacity and speed, which are important response variables for pulp intended for sanitary purposes. They are important variables because from a practical point of view the pulp must meet

these parameters. Lastly, for the apparent density variable, an AQ concentration versus NaOH concentration graph was constructed to observe the slope that indicates the quadratic effects of these factors. For the analysis, areas can be proposed that lead to lower apparent density values, because generally that's where the most absorbent pulp is.

The generation of regression models as well as response surface graphs can lead to the analysis and proposal of favorable conditions for the quality of the pulps obtained. Then, with the help of the software it was possible to predict the optimization of the response variables as a whole, through multivariate optimization, from the reference values used commercially. The multivariate optimization was done via the models generated for each of the response variables, estimating the relative importance of each of these variables with numbers on a scale from 1 to 10. The absorption capacity variable achieved greater importance and was assigned a value of 10. Then, absorption speed the value was 7, apparent density the value was 4, while the kappa number variable had the lowest relative importance with a value of 1. The graphs generated for the behavior of each of the three controllable factors in relation to the response variables according to the proposed models presented in tables above can be showed in https://docs.google.com/open?id=0B8VVSr93P98iV0tNal8zOEtSTktpZzFZTnF3dnFCUQ. The optimization was done from the coding of the three controllable factors, abiding by the upper interval for the star point of 1.68 and the lower interval for the star point of -1.68. The specified upper and lower limits for the response variables, as well as the target value were specified in the methodology. Table 6 lists the values and experimental points for the optimized values of each response variable through multivariate optimization.

Table 6 - Values for the response variables from the experiments with alkaline extraction after acid hydrolysis after optimization

Response Variable	***Optimized values***		***Level for AQ concentration factor***		***Level for time factor***		***Level for NaOH concentration factor***	
	Without hydrolysis	With hydrolysis	Without hydrolysis	With hydrolysis	Without hydrolysis	With hydrolysis	Without hydrolysis	With hydrolysis
Kappa number	41.4784	95.7789	-1.68	-1.68	-1.68	-1.68	-1.68	-1.68
Absorption capacity	4.3219	4.2648	-1.68	-1.68	-1.68	-1.68	-1.68	-1.68
Absorption speed	1.0272	0.4458	-1.68	-1.68	-1.68	-1.68	-1.68	-1.68
Apparent Density	1.1406	0.8592	-1.68	-1.68	-1.68	-1.68	-1.68	-1.68

Even with optimization of the variables, it can be seen from the results presented that the pulps produced by the proposed processes do not meet the quality standards required for absorbent pulps. The very high kappa numbers are not in line with the reference numbers for pulps for sanitary purposes, which are usually between 5 and 10. The very long absorption times presented by the pulps led to low absorption speeds, which are less than or close to 1 g/s. In turn, the absorption capacity is also low for the samples, with maximum values close to 5 g of water/g of dry pulp. For the apparent density variable, the samples studied indicated values close to or slightly higher than 1g/cm3, likewise not meeting commercial reference values. Another factor pertains to the morphological characterization of the fiber, for proposing improvements in the experiments, as well as pretreatment of the fiber. Due to the limitations imposed by the process, regarding the use of temperatures not exceeding 127°C, the process conditions were unfavorable. The literature recommends the use of temperatures in the range of 170°C, but processing time can help in the delignification process if these temperatures are not possible. However, in the study, prolonged pulping time did not totally compensate the deficiencies in the delignification.

5 Conclusion

The study showed the possibility of obtaining cellulose pulp through alkaline pulping using the leaf sheaths of the King Palm. A design of experiments RSM was used, with an experimental design along the lines of CCD.For this purpose, models were generated for the response variables (kappa number, absorption capacity, absorption speed and apparent density) through multiple regression, using a statistical significance level of 15%. These models took into account the influence of three controllable factors – AQ concentration (0.33 to 1.17%), Reaction time (110 to 160 minutes) and NaOH concentration (10 to 34%) – on the response variables.

The results of the experimental design tests show that the pulps produced through the proposed processes do not meet the quality standards required for absorbent pulps. The very high kappa numbers are not in line with the reference numbers for pulps for sanitary purposes, which are usually between 5 and 10. The very long absorption times presented by the pulps led to low absorption speeds, which are less than or close to 1 g/s. In turn, the absorption capacity is also low for the samples, with maximum values close to 5 g water/g of dry pulp. For the apparent density variable, the samples studied indicated values close to or slightly higher 1g/cm3, likewise not meeting commercial reference values. In the practical and exploratory study, prolonged pulping time did not totally compensate the deficiencies in the delignification, since there was a limitation imposed by the process as far as using a maximum temperature of 127°C, lower than the temperatures recommended in the literature which range from 160 to 170°C. In light of this, for future works, it would be suggested to investigate temperature ranges from 150 to 180°C, with pulping time between 90 and 180 minutes, NaOH concentration within the investigated interval of 10 to 34% and AQ concentration from 0 to 1% in relation to dry matter. In the event there are still restrictions on the use of higher temperatures, the tests could observe the same AQ and NaOH concentration intervals but with larger investigation intervals for the time factor, in order to serve as indicators for analyzing the effect of this factor on the response variables.

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