INDEPENDENT JOURNAL OF MANAGEMENT & PRODUCTION (IJM&P)
http://www.ijmp.jor.br v. 8, n. 4, October - December 2017
ISSN: 2236-269X
DOI: 10.14807/ijmp.v8i4.648

SUSTAINABLE EXTRATION OF CELLULOSE PULP FROM AGRIBUSINESS WASTE: POOR PERFORMANCE IS BAD RESULT?

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Submission: 13/03/2017
Accept: 24/04/2017

ABSTRACT

Sustainable production is a recurrent theme in industrial engineering. Commercial production of canned palm hearts generates an amount of waste from the leaf sheaths that envelop the heart of palm, which can be used to produce cellulose pulp then reducing environmental impacts. This study aims to examine the feasibility of cellulose pulping from King Palm leaf sheaths 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 through the formulation of n-dimensional equations and surfaces which permitted the optimization of the variables of interest. The response variables were selected in order to characterize the fiber obtained according to degree of delignification, absorption capability and speed and apparent density. The results indicated that the pulps obtained from the processes proposed although didn't meet the quality standards required for absorbent pulps, since values are lower than those established in the research hypotheses, is very promising. This attempt raises the discussion around the role that industrial engineering professionals and researchers may play in the agribusiness waste recovery and recycling. Moreover it provides useful information for re-planning of experiments in search for extraction optimization of this or similar agribusiness wastes.

Keywords: Sustainable production; agribusiness waste; Design of Experiments; Response Surface Methodology (RSM); cellulose pulping; King Palm.

1. INTRODUCTION

Design approaches that emphasize the economic, social and environmental dimensions, associated with advances in technology, contribute to the development of more effective environment sustainable production solutions (SALVADOR et al, 2014; LYAKURWA, 2014). An aspect referred to sustainable approaches is the reuse of process waste, as well as the minimization of its generation (PAULI, 1996; PAULI, 1998; McDONOUGH; BRAUNGART, 2002; CNTL, 2003). Waste recovery for the purpose of generating new products has been object of research, since there is a significant variation in the quality of the waste if used as raw materials and due to the need of designing processes that can efficiently effect this transformation.

This paper focused on the examination of the alkaline cellulose pulping from the waste generated during the production of canned palm hearts, originating from the King Palm (*Archontophoenix alexandrae*). Three layers of leaf sheaths that surround the heart of palm constitute the waste from this process, consisting in 90% of the input material in the process and the other 10% is the portion commercialized by the industry. 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). The main components of lignocellulosic waste are cellulose, hemicellulose, lignin and nitrogen, in a smaller amount.

Studies have been conducted to investigate different applications for the waste from the King Palm, since they can add value to the waste left behind on the field and may reduce environmental damage resulting from its accumulation (TONINI, 2004). Noteworthy among the research studies is the application of the middle leaf sheath for producing hydrolytic enzymes by fungi from the genus

Polyporus (ISRAEL, 2005) and the waste used as substrates for cultivating fungi both from the *Pycnoporus sanguineus* species (BORDERES, 2006) and the *Lentinula edodes* species (TONINI, 2004). In a study performed at the Rural Federal University of Rio de Janeiro (UFRRJ), the use of the *Euterpe edulis Martis* stem, which is thrown away during heart of palm harvesting, was investigated as a fibrous raw material alternative for producing cellulosic pulp by the kraft method (ANDRADE et al, 2000).

The demand for easily-renewable raw materials for the manufacture of consumer goods is a growing trend around the world as may be seen in patents like (PI 1102323-6 A2, 2011– nanocrystalline cellulose; PI1100439-8, 2011; CN103012822, 2013 and CN102720089, 2012– cellulose membrane). 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; THOMAS, 2008). In line with this trend is the proposal to obtain cellulose from King Palm leaf sheaths, thereby taking advantage of and recovering an abundant by-product which results from the industrial processing of canned heart of palm. It is expected a 27 to 30% yield in the pulping of the dried waste.

To carry out such a project, the leaf sheaths resulting from the processing of heart of palm, which are often left in the field or used as animal feed, must undergo cellulose extraction using specific equipment. Normally, the most industrially-used pulping of lignocellulosic raw materials employs alkaline reagents for transforming the fresh fiber into cellulose pulp (SIXTA, 2006). Pulping with soda and anthraquinone, as an additive, is suitable and common for pulping processes in plants that are not subject to seasonality (HOLTON, 1977; ANTUNES et al, 2000).

A variety of references describe the treatment of plant waste with alkaline pulping using soda and an additive for delignification. The literature review cite pulping with soda from rice straw (RODRÍGUEZ et al, 2010), alkaline pulping with soda to obtain fiber from corn husks (REDDY; YANG, 2005), the alkaline pulping of soybean husk (REDDY; YANG, 2009) and banana fiber production through alkaline extraction with soda (ELANTHIKKAL et al, 2010).

Among the studies there is the treatment of date palm petioles, using alkaline pulping to obtain and characterize fibers as replacements for wood fiber

(KHRISTOVA et al, 2004; KHIARI et al, 2009). In the alkaline chemical pulping method for King Palm leaf sheaths, which is the object of study in this research, soda (NaOH) was used as a delignifcation reagent and anthraquinone (AQ) as a process additive. An experimental design was proposed with three controllable factors – AQ concentration (0.33% - 1,17%), period of time in the reactor (110 - 160 minutes) at pulping temperature (127°C) and NaOH concentration (10% - 34%). The response variables analyzed were the kappa number, which represents the residual lignin content in the pulped material (ABNT, 2005), water absorption capacity, absorption speed and apparent density of the pulp. Would these parameters be also effective to the production of cellulose pulp for sanitary absorbent purposes?

This study objective is to examine the feasibility of cellulose pulping from King Palm leaf sheaths in order to obtain a fiber with absorbent capacity and low residual lignin content, as well as, to demonstrate the influence of the controllable process factors on the response variables analyzed. The practical contribution, in the industrial engineering perspective, is to propose the reuse of plant waste from agribusiness in order to produce cellulose pulp for sanitary and absorbent purposes. The academic contribution is to comprehend how the pulping parameters' levels may influence the physical-chemical characteristics of this cellulose pulp, and to provide useful information for re-planning of experiments in search for optimization extraction.

2. EXPERIMENTAL

This study characteristic is the applied research, with a quantitative approach, since it uses numerical analyses and statistical techniques. The aim of this study is explanatory in nature, since it seeks to identify the factors that determine or contribute to the occurrence of phenomena, through using an experimental method (GIL, 1991).

2.1. Raw Material

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.

It is important to mention that previous studies done by FEPAGRO (State Foundation for Agricultural Research) 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 (Patent 0606063-3 of 2008). Inulin, a natural sweetener, is present in the filtered liquor following the acid hydrolysis of the palm leaf sheaths. Thus, the pulping of the leaf sheaths, proposed in this paper, was developed in two ways: from fresh leaf sheath waste and after undergoing acid hydrolysis for previous inulin extraction.

The fresh crushed raw material and the hydrolyzed material was dried in an oven at 68°C, mixed, homogenized and set aside in a container to be used during the alkaline extraction tests through a design of experiments (MONTGOMERY, 2001).

The acid hydrolysis process of the leaf sheaths entailed attacking the fiber with an acid solution diluted in water. In the acid hydrolysis reaction (inulin production process), temperature and pressure were kept constant at 127°C and 1.5 atm which represent the maximum parameters permitted for operating, respectively, in a vertical autoclave, BIOENG brand, model A50, number 274. Table 1 contains the acid hydrolysis conditions as per Patent 0606063-3 (2008).

Table 1: Process conditions for the acid hydrolysis of King Palm waste (Patent 0606063- 3 of 2008)

Solution-material ratio	10:1
Acid concentration % (v/v)	3
Pressure (atm)	1.5
Temperature (ºC)	127
Time in the autoclave at the stipulated temperature (min)	30

To increase the efficiency of the attack on the fiber, the material was ground in order to increase the contact surface with the acid solution. After the material underwent hydrolysis, it was neutralized in an aqueous medium with soda (NaOH) until reaching a pH close to 7.0. It was 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. The granulation of the raw material and the hydrolyzed material were determined by passing it through a Tyler Mesh 2 sieve.

2.2. Pulping and Pulp Characterization

The treatments were performed in the Pathology Laboratory Plant located at the State Foundation for Agricultural Research (FEPAGRO). Pulping was performed by using a vertical autoclave, BIOENG brand, model A50, number 274. The autoclave has a 20-liter capacity and contains a basket which is capable of supporting two two-liter beakers. The equipment is used for bench-level experiments, since despite its small size it can reproduce industrially-applicable process parameters, also it has the required instruments for measurement and control parameters as pressure and temperature. Temperature and pressure were kept constant at 127°C and 1.5 atm, respectively, which represent the maximum parameters permitted for operating the equipment.

The controllable factors as AQ concentration, reaction time and soda concentration used according to industrial parameters and those available in the literature were 0,33-1,17% (on dried raw material), 110-160 min and 10-34%, respectively (SMOOK, 1982; KHRISTOVA et al, 2004; VASCONCELOS, 2005; SIXTA, 2006; KHIARI et al, 2009; RODRÍGUEZ et al, 2010). A 50 g of dry material was used and the liquor-material ratio was 10:1.

The alkaline treatments occurred on different days since it requires around four hours to process but efforts were made to maintain the same environmental conditions, thereby minimizing experimental error. During the mounting of the experiment, efforts were made to completely cover the plant material with the alkaline solution before pulping.

Once the experiment began the time established for each point was measured from the time the system reached a temperature of 127°C with a maximum deviation of ± 5°C from the stipulated temperature. At the end of the cooking time, the next step was opening the equipment to remove the beakers and cool them before open, 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.

All the samples were dried after collection and sent for laboratory analysis, which was done once for each sample. The analysis of the response variables was conducted in an independent laboratory belonging to the National Network of Industrial knowledge, which is a private institution that works to improve the industry development. Kappa number, absorption capacity (g), absorption speed (g/s) and apparent pulp density (g/cm3) were the response variables analyzed and they were determined according to ABNT NBR ISO 302 (2005), ABNT NBR 15004 (2003) and ABNT NBR NM - ISO 534 (2006), respectively. Table 2 shows the controllable factors, intervals used and their respective levels analyzed in the experiments.

Table 2: Points and controllable factors, in their high and low levels, and center point for the Second Order Central Composite Design (SOCD)

Factors	**CONTROLLABLE FACTORS**	**Real Levels**		**Center Level**	**Star Level**	
		Low Level	**High Level**			
		(-1)	(+1)	(0)	(-1.68)	(+1.68)
X_1	AQ concentration (%)	0.5	1.0	0.75	0.33	1.17
X_2	Time (min)	120	150	135	110	160
X_3	Soda concentration (%)	15	29	22	10	34

Whereas Table 3 presents the points investigated in laboratory trials.

Table 3: Trials investigated for extracting cellulose from King Palm leaf sheaths according to the Experimental Design

	CONTROLLABLE PROCESS FACTORS AND CODED FACTORS					
POINT	**AQ Concentration (%)**		**Time (min)**		**NaOH concentration (%)**	
1	0.5	-1	120	-1	15	-1
2	1	1	120	-1	15	-1
3	0.5	-1	150	1	15	-1
4	1	1	150	1	15	-1
5	0.5	-1	120	-1	29	1
6	1	1	120	-1	29	1
7	0.5	-1	150	1	29	1
8	1	1	150	1	29	1
9	0.33	-1.68	135	0	22	0
10	1.17	1.68	135	0	22	0
11	0.75	0	110	-1.68	22	0
12	0.75	0	160	1.68	22	0
13	0.75	0	135	0	10	-1.68
14	0.75	0	135	0	34	1.68
15	0.75	0	135	0	22	0
16	0.75	0	135	0	22	0

3. RESULTS AND DISCUSSION

The experiment designed for alkaline extraction was a Second Order Composite Design (SOCD) as show in Table 3. The values of the star points (alpha) were calculated in order to confer the orthogonality condition to the project.

Absorbent materials such as paper for sanitary purposes must display certain important characteristics to ensure the efficiency of their function, in other words, absorption and retention of liquids functions. According to Jordão and Neves (1989), the most important absorption properties of absorbent 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) stresses that the morphology of the fibers also affects the pulp’s absorption capacity, as does the quantity of resins. 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.

According to Foelkel (2010), who studied the behavior of pulps for absorbent purposes, the specifications for parameters of water absorption capacity, absorption time and density are as follows in Table 4.

Table 4: Parameters for absorbent pulps

Parameter	**Values used in the market**	**Specifications**
Absorption capacity of water [g of water/g dry pulp]	8 to 14	Nominal is better
Absorption time [s]	8 to 2.5	Nominal is better
Absorption speed [g/s]*	3.2 to 20	Nominal is better
Density of the pulp sheet [g/cm3]	0.4 to 0.6	Nominal is better

* Considering the maximum and minimum values for absorption capacity and time

In order to determine these parameters for the pulp obtained from the process waste (leaf sheaths), experiments were performed from November to February 2010. An amount of 16 tests were performed with fresh material and another16 with hydrolyzed material, which generated 32 different samples.

Only one test was performed for each experimental condition, since these are cumbersome and takes a lot of time. Thus, the results presented are preliminary study data, indicative of the factors that influence the response variables analyzed. To increase the degree of reliability of the results, it would be necessary to perform repetitions of the tests. The results obtained for the response variables for each treatment, without and with acid hydrolysis of the raw material, are listed in Table 5.

Table 5: Response variables of the Experimental Design for extracting cellulose from King Palm leaf sheaths with soda and additive before and after acid hydrolysis

	RESPONSE VARIABLES With hydrolyzed material				**RESPONSE VARIABLES With raw material**			
POINT	**Kappa No.**	**Absorption Capacity [g]**	**Absorption Speed [g/s]**	**Apparent Density [g/cm^3]**	**Kappa No.**	**Absorption Capacity [g]**	**Absorption Speed [g/s]**	**Apparent Density [g/cm^3]**
1	66.12	3.23	0.38	1.19	51.38	3.52	0.71	1.17
2	47.61	2.93	1.04	1.17	41.27	2.17	0.17	1.34
3	50.23	3.3	0.58	1.39	46.18	2.58	0.16	1.36
4	61.5	4.05	0.8	1.34	66.50	2	0.51	1.27
5	43.73	3.48	1.47	1.12	42.49	2.49	0.14	1.3
6	35.28	4.12	0.71	1.14	41.16	2.44	0.28	1.23
7	43.27	3.73	1	1.21	61.68	3.82	0.6	1.07
8	49.23	3.32	1.36	1.06	60.04	4.83	0.91	1.21
9	38.76	4.53	1.45	1.12	51.87	2.92	0.53	1.36
10	54.86	4.53	0.74	1.14	51.10	2.36	0.16	1.29
11	25.17	3.86	0.17	1.27	48.40	3.43	0.45	1.28
12	22.41	4.13	0.37	1.45	50.48	1.8	0.02	1.24
13	60.89	3.14	0.64	1.2	51.43	2.75	0.65	1.24
14	43.38	3.41	0.52	1.3	62.39	2.04	0.47	1.15
15	39.51	2.83	1.24	1.49	60.45	2.34	0.55	1.2
16	39.39	3.31	1.36	1.3	62.00	2.98	0.6	1.22

All the results were treated with Minitab 15 statistical software in order to statistically evaluate, to the 15% significance level, the statistically significant linear and quadratic effects were noted for the response variables. The value was set according to data presented in the literature. In experiments with different vegetable sources, researchers have defined statistical significant levels ranging from 10 to 20%, for creating regression models and optimization using response surface methodology as it may be seen in Rezayati-Charani and Mohammadi-rovshandeh (2005), Rezayati-Charani et al, (2006), Cho and Zoh (2007), Jiménez et al, (2009) and Zeng et al, (2011).

Once the laboratory results were obtained, a modeling was done through multiple regression, using only the significant effects indicated by the statistical analysis. With multiple regression, it is possible to estimate a mathematical equation in which the value of the dependent variable can be predicted by the values of the independent variables. For models proposition, the experimental data was adjusted by a second-order polynomial through a multiple linear regression (Equation. 1):

$$Y = A_0 + \sum_{i=1}^{n} A_i X_i + \sum_{i=1}^{n} A_{ii} X_i^2 + \sum_{i=1}^{n}\sum_{j=1}^{n} A_{ij} X_i X_j$$

(Equation 1)

Table 6 shows the equations generated by the software for the response variables of the experiments that only had the alkaline pulping stage. The models predicted by the regression were done from the analysis of the p-value of the controllable factors that were less than or equal to 0.15.

Table 6: Proposed models for the response variables of the experiments with alkaline extraction

Variable (Y)	***Equation***	***R^2 - coefficient of determination***	***p - value***
Kappa number	$Y= 61.298 + 4.51X_2 – 4.342 X_2^2$	69.40%	X_2: 0.059; X_2^2: 0.114
Absorption capacity	$Y= 2.6177 + 0.6038 X_2X_3$	54.30%	X_2X_3: 0.090
Absorption speed [g/s]	$Y= 0.5686 + 0.1625 X_2X_3$	60.60%	X_2X_3: 0.113
Apparent density [g/cm³]	$Y= 1.2119 - 0.0352 X_3 – 0.0462 X_2X_3$	61.90%	X_3: 0.139; X_2X_3: 0.137

For the kappa number response variable, the positive linear influence of the time factor can be seen in the model, as well as the negative quadratic effect of this factor. The AQ and NaOH concentration factors were not statistically significant for the model, or their interactions.

For both the absorption capacity and absorption speed of the pulp, the positive interaction between the time and NaOH concentration factors proved to be significant for the models. The time factor, however, was not significant at the 15% level. For the apparent density of the pulp, the model also showed that the interaction between the time and NaOH concentration factors was significant, albeit negative. Apart from that, for this response variable the model demonstrates the linear and negative influence for the NaOH concentration factor.

However, the R2 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 in this model 69.4% of the response variable is explained by the controllable variable and 30.6% can be explained by external factors that do not appear in the model. The other models reveal an even 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. Apart from that, experimental conditions and process variabilities can lead to skewed results, which could be analyzed if the experiments were repeated.

Table 6 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 pulping stage.

The regression analysis for the kappa number showed that the NaOH concentration is a significant factor in the model. It influences in a linear way, causing the kappa number to decrease if there is any increase in the NaOH concentration. This can be seen, since the index of the factor in the equation is negative. In the regression model, the positive quadratic effects of the AQ and NaOH concentrations can also be seen, as well as the positive interaction between the AQ concentration and time factors.

The R2 value of the generated model is close to 1, indicating that the adjustment is consistent with the sample data. 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.

The regression analysis for absorption capacity showed that the positive quadratic effect of the AQ concentration is significant in the model. However, the model did not demonstrate statistical significance for the time and NaOH concentration factors.

In the regression analysis for the absorption speed variable, the statistical significance was noted for the negative quadratic effect of the time factor. Finally, to build the model for the apparent density variable, the regression analysis showed the linear and positive influence of the time factor and negative quadratic effects for both the AQ and NaOH concentration factors.

Based on the generated regression equations, which highlight the significant coefficients for the model, Minitab 15 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 are presented in Appendices A and B, with the effects of the factors, via graphs: time versus NaOH concentration, AQ concentration versus NaOH concentration and AQ concentration versus time.

For the tests that only entailed alkaline pulping 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, for the absorption speed variable the value of 7 was assigned. The apparent density variable had a proposed value of 4, while the kappa number variable had the lowest relative importance with a value of 1. Table 6 and Table 7 show the proposed models.

Table 7: Proposed models for the response variables of the experiments with alkaline extraction after acid hydrolysis

Variable (Y)	***Equation***	***R^2 - coefficient of determination***	***p - value***
Kappa number	$Y = 38.618 - 6.107X_3 + 4.61 X_1^2 + 6.493 X_3^2 + 5.524 X_1X_2$	81.60%	X_3: 0.034; X_1^2: 0.141; X_3^2: 0.054; X_1X_2: 0.108
Absorption capacity	$Y = 3.1149 + 0.4077X_1^2$	67.30%	X_1^2: 0.042
Absorption speed [g/s]	$Y = 1.2702 – 0.2923X_2^2$	59.30%	X_2^2: 0.080
Apparent density [g/cm^3]	$Y = 1.3984 + 0.05 X_2 - 0.1019X_1^2 – 0.0595 X_3^2$	74.50%	X_2: 0.116; X_1^2: 0.022; X_3^2: 0.122

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. Table 8 lists the specified upper and lower limits for the response variables, as well as the target value.

Table 8: Specifications for the response variables from the experiments with alkaline extraction after acid hydrolysis after optimization

Response variable	**Lower limit**	**Target value**	**Upper limit**
Kappa number	5	8	10
Absorption capacity of water [g of water/g of dry pulp]	8	10	12
Absorption speed [g/s]	3.2	12	20
Density of the pulp sheet [g/cm3]	0.4	0.55	0.6

Table 9 lists the values and experimental points for the optimized values of each response variable through multivariate optimization.

Table 9: 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

However, 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.

Comparing the pulping conditions, it is possible to observe that even considering optimized values, the raw material without acid treatment revealed better results in relation to kappa number and the absorption speed. However, these values didn't meet the required values for absorption purposes. The hydrolyzed material has presented better values in the relation to apparent density and similar value to absorption capacity.

In view of the above, it would be recommended to replicate the experiments in order to confirm these values to investigate deeper the effects of the acid treatment. 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. According to data collected in the literature, it is suggested for future works to investigate temperature ranges from 150 to 180°C, with pulping time between 90 and 180 minutes, NaOH concentration within the investigated interval from 10 to 34% and AQ concentration from 0 to 1% in relation to dry matter.

4. CONCLUSION

Poor performance is definitely not a bad result! In the field of experiments! To know in advance what does not work is half of the way for optimization task. In light of the problem of waste generation in agribusiness and the environmental impact caused by its accumulation in the environment, the proposal for its recovery constitutes an important economic and environmental alternative and has been concern of industrial engineering researchers. The goal of this study was to verify the feasibility of cellulose pulping from King Palm leaf sheaths to obtain 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.

Bearing in mind the possibility of obtaining cellulose pulp with these desired traits, the proposal was made to extract cellulose in an alkaline medium from the leaf sheaths of the King Palm. This species of palm generates raw materials for the canned heart of palm processing industry, which in turn generates waste with potential to be reused for value-added products. Thus, pulping conditions for the waste were tested via design of experiments.

For the design of experiments, Response Surface Methodology (RSM) was used, with an experimental design along the lines of SOCD (Second Order Composite Design), in which the controllable factors were coded so that it would be possible to compare the effects of the different controllable factors. 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, despite do not meet the quality standards required for absorbent pulps is very promising. The 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, it would be advisable to replicate the experiments, in order to confirm the values above, and 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.

Further experiments using higher temperatures may result better outcomes. In case temperature remains unchanged, it is recommended the use of 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. As a final contribution, this first attempt raises the discussion around the role that industrial engineering professionals and researchers may play in the agribusiness waste recovery and recycling. This information is very useful for further experiments for this or similar plant wastes.

5. ACKNOWLEDGEMENTS

We would like to thank the National Council for Scientific and Technological Development (CNPq) for providing a research scholarship. We'd also like to express our thanks to the laboratory that made it possible to carry out this study by providing materials and equipment (FEPAGRO), as well as the vegetable waste processing

company for supplying raw material in order to perform the experiments.

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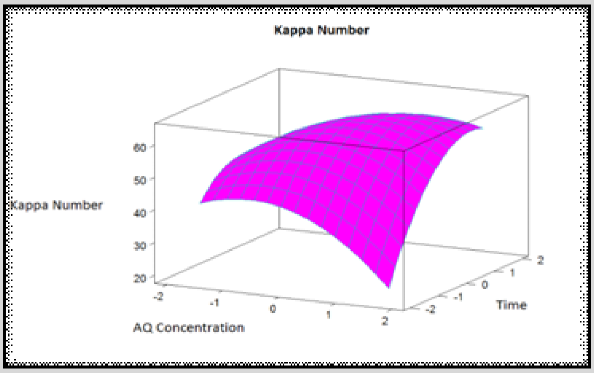
APPENDIX A

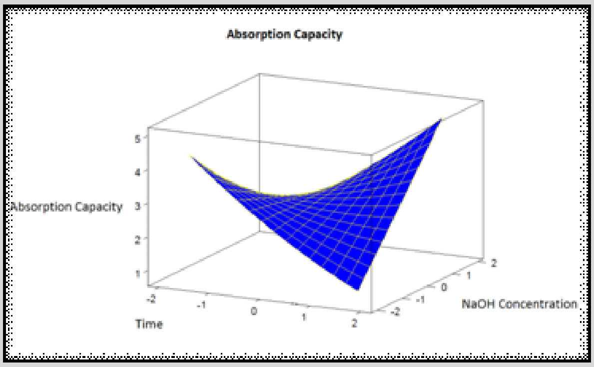
Response Surface graphs for extraction

Effects of the controllable factors on the response variables

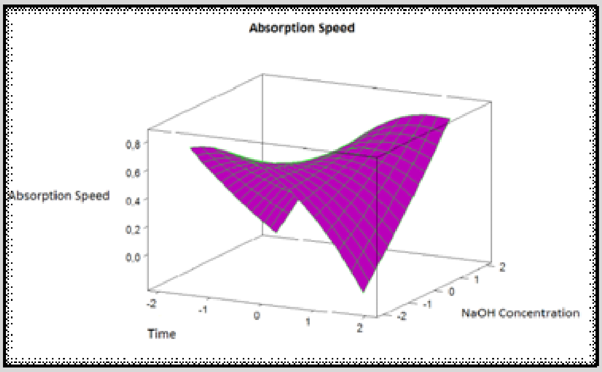
Kappa Number



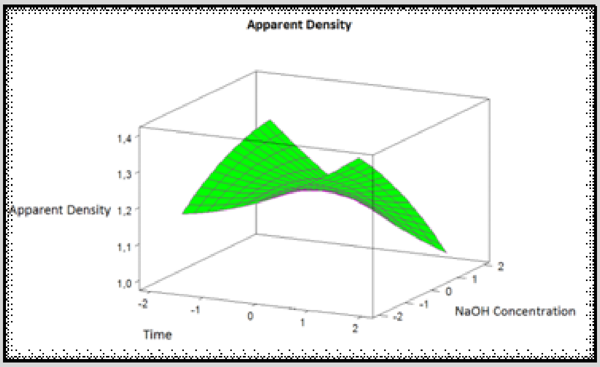
Absorption Capacity



Absorption Speed



Apparent Density



APPENDIX B

Response Surface graphs for extraction followed by hydrolysis