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Production of Writing and Printing Papers from Woody Petioles of Oil Palm Fronds (Elaeis Guineensis): Determination of Optimum Pulping Conditions using Soda-AQ Process

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ABSTRACT: *Comparative assessment of physico-chemical characteristics, anatomic properties and economic relevance of Oil Palm Fronds (woody petioles) to the pulp and paper industry was investigated. Sample preparation was conducted in accordance with TAPPI Standard T12 – OS – 75, which specifies that samples be grinded to a fine particle size to permeate 0.4mm screen and retained on a 0.6mm screen. The Moisture content, Lignin, Extractives, Alpha cellulose, Ash content and fiber dimensions were investigated. The pulping investigation had four (4) factors at three (3) different levels each: Factor 1, cooking temperature (150, 160 and 170°C) ; Factor 2, cooking time (60, 90 and 120minutes);Factor 3, liquor concentration (10%, 15% and 20% NaOH charge); and Factor 4, anthraquinone concentration (0.00%, 0.25% and 0.50%). The experimental design had 3×3×3×3 i.e., 81 pulping scenarios. Pulping Operation was subsequently carried out by the Soda-AQ Process which resulted in the production of pulp and paper samples. Pulp Yields was in the range of 29% to 47.99% calculated on oven dry (O.D) basis. The resultant pulps obtained from the cooking operation had very good feel, and exhibited fairly bright color, with slightly slow tendency to felt, thereby making drainage and consequent paper making time short. Over-all parameter achieved asserts that oil palm fronds (woody petioles) have a promising future (when used in blend with long fiber pulp) in substituting wood in the pulp, paper and fiber- board industry.*

KEYWORDS: Oil palm frond (Woody petiole), Lignin, Tappi standards, kappa number, Pulp screened yield, Soda-AQ, Oven dried (O.D.)

INTRODUCTION

As the importance of paper extends from the home as toiletries, government and schools/academic institutions as writing and printing materials, and in the industries as wrapping/packing materials, it becomes necessary to study by research to discover ways of converting waste materials to pulp and paper in order to meet the growing requirement as well as reduce the cost of paper materials in Nigeria, hence, the extensive physico-chemical analysis on oil palm wastes **(Chibudike, 2011)**.

The Oil Palm is a major source of vegetable oil, ranking next to coconut in importance. Palm Oil become known to the western world when Portuguese explorers sailed south and eastward around the hump of Africa. According to the Commonwealth Economic Committee (1956), a total of 845,000 m. tons of palm kernels and 664,000 m. tons of oil was produced in the world in 1955, with West Africa supplying the bulk. About 50% of the kernels, 421,000 m. tons, came from Nigeria alone. The chemical constituents of Oil palm wastes are carbon, oxygen, hydrogen, ash (largely silica). They are composed of cellulose; other carbohydrates present are starch, sugar, and lignin, which act as an adhesive for cellulose **(FAO. 2009)**

Oil palm trees are chiefly grown in the Southern part of Nigeria, and other parts of West Africa. Where oil palms are majorly processed, palm bunches and palm fronds constitute wastes (bye products). These residues are woody petioles (leaf stems) often used for fencing,

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the trunks of the trees for building, and the high quality animal folder are used by local craftsmen who plait the leaves into thatch and the fronds into brooms and baskets. The discarded bunches of the oil palm are used as substitute for firewood while the sap of the tree serves as good source of palm wine. The residue from the oil–extraction process constitutes useful domestic needs and also provide valuable source of income for many small holders in a variety of ways **(F.A.O., 2009)**. However, the percentage of these residue usefully utilized are quite low, the rest constitute waste and nuisance to the environment which end up being burned for no beneficial purpose. Most often, these waste materials pose disposal problem which constitute nuisance to the environments where they are processed. They often have negative value as resources are employed to burn the materials on site or transport for disposal elsewhere. The common method of disposal by burning causes a great deal of pollution and is a menace to the environment **(Chibudike, 2019)**.

Paper is defined as "thin sheets of compressed vegetable cellulose fibre". It is used for writing and printing, for wrapping and packing and for many other varieties of special purpose, from filtering precipitates from solution and to make building material. In modern society, paper is a basic material. The development of well-developed machineries to produce it has been largely responsible for increasing literacy, and raising educational levels throughout the world. Wood is composed of cellulose; other carbohydrates present are starch, sugar and lignin, which act as an adhesive for cellulose (some other adhesive used are gum, mucilage, lattice and resins**.** Paper sources are primarily from wood, which is composed of cellulose fibres that exist as small tubes oriented along the length of the trees. In plants fibres, woody cells formed at the active area of growth between the bark of the wood constitute the cambial layer. The fibres help in water transportation. These fibres are the most important part of the tree used in papermaking. At some time in the process of industrialization, urban requirements for wood would begin to put heavy and increasing demands on the forest. The use of non-wood fibrous plants for the production of pulp and paper should be the attainment of the highest economic use of these materials. It is believed that waste utilization can assist in reviving Nigerian Economy (**Steward, 2001 and 2003).**

Experimental

Materials

The Oil Palm Fronds (Agro-biomass) used in this experimental work was obtained during post-harvest treatment in Oke Ira, Ebute-Metta (West) Local Government Area of Lagos State, Nigeria.

Raw material characterization

Prior to chemical characterization and pulping, the raw material was washed, cleaned, sorted to remove foreign matters and air-dried, then stored to less than 60% relative humidity and aerated from time to time, to avoid decay. Following drying at ambient temperature, the raw material was cold-ground in a Wiley mill, to avoid altering its composition, permeating 0.25mm and retained on a 0.40mm sieve to keep size fractions between 0.25 and 0.40 mm using No. 25 and 40 of the Tyler series in accordance with TAPPI Standard T12–oS–75. Particles larger than 0.40 mm are inefficiently attacked by the chemical reagents, whereas those below 0.25 mm can cause filtering problems. The sample was characterized by analyzing its content of moisture, hot water solubility, klason lignin, α-cellulose, 1% NaOH solubility, total extractives and ash. Standard procedures were used for the analyses of these parameters and these procedures are outlined in Table 1.

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Table 1 Standards used in the Chemical Characterization

Agro-biomass Characterization	Standards
Sample preparation	TAPPI Standard Test Method T 12 oS-75
Moisture	TAPPI Standard Test Method T 264 om-88
Hot water solubility	TAPPI Standard Test Method T 207 cm-99
Total Extractives	TAPPI Standard Test Method T 204 cm-97
Acid insoluble (klason) lignin	TAPPI Standard Test Method T 222 om-02
Alpha (α)-cellulose	TAPPI Standard Test Method T 203 os-74
1% NaOH solubility	TAPPI Standard Test Method T4 os-59
Ash	TAPPI Standard Test Method 211 om -02
Holocellulose	TAPPI standard Test method T-249, 2004
Kappa No.	TAPPI Standard Test Method T236 om-06
Viscosity	TAPPI Standard Test Method T230 om-08
Brightness	TAPPI Standard Test Method T452 om-08

Determination of Fiber Morphology

Small slivers were obtained and macerated with 10 ml of 67% HNO_3 and boiled in a water bath (100 ± 2ºC) for 10 min **(Ogbonnaya et al., 1997)**. The slivers were then washed, placed in small flasks with 50 ml distilled water and the fiber bundles were separated into individual fibers using a small mixer with a plastic end to avoid fiber breaking. The macerated fibers suspension were finally placed on a slide (standard, 7.5 cm × 2.5 cm) by means of a medicine dropper and stained with1:1 aniline sulfate–glycerin mixture to enhance cell-wall visibility (cell walls retain a characteristic yellowish color) and easier measurement. About twenty (20) fibers were measured per each sample at a magnification of X 101 on a Reichort visopam projection microscope and fiber diameter, lumen diameter, cell wall thickness and cross-sections were obtained. All samples were measured in a swollen condition.

Outline of the Production Process

Figure 1 illustrates the process of making paper from Oil Palm Fronds (woody petioles). The sample was characterized chemically and morphologically and converted into brown pulp at a delignification degree of 18.2 kappa from Soda-AQ Process. The resulting pulps was fully bleached by the D1-Ep-D2 sequence and characterized for its beatability, drainability and physical-mechanical properties.

Description of the Pulp and Paper-making Process

The sample was shredded prior to chemical characterization and pulping, a portion of the shredded sample was washed, cleaned, sorted to remove foreign matters and air-dried, then stored to less than 60% relative humidity and aerated from time to time, to avoid decay. Following drying at ambient temperature, the raw material was cold-ground in a Wiley mill, to avoid altering its composition, permeating 0.25mm (because samples below 0.25 mm can cause filtering problems) and retained on a 0.40mm sieve (because particles larger than 0.40 mm are inefficiently attacked by the chemical reagents) to keep size fractions between 0.25 and 0.40 mm using No. 25 and 40 of the Tyler series in accordance with TAPPI Standard T12 – oS – 75. This portion of the sample was characterized by analyzing its content of moisture, hot water solubility, klason lignin, α-cellulose, total extractives and ash. Standard procedures were used for the analyses of these parameters and these procedures are outlined in table 1. The second portion of the shredded sample was subjected to a thorough cleaning process, 2kg of air-dry sample was loaded into a 15 L capacity batch reactor (digester) with eight (8) liter cooking liquor at liquor-sample ratio of 4:1. The digester is furnished with an outer electrical heating jacket. The lid of the digester was firmly bolted to prevent leakage, the digester was switched on and the time of rise of temperature and pressure was noted at intervals of five (5) minutes. The content of the digester was stirred while in operation by rotating the vessel via a motor connected through a rotary axle to a control unit, including measurement and control instruments of pressure and temperature, to facilitate attainment of the working temperature (5ºC/min). The pulping temperatures gradually rose to the maximum cooking temperature of 150ºC within a period of 61minutes and allowed to be steady at this temperature for minimum of 29minutes. The digester was switched off after maximum cooking period of 90

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minutes from start of operation and allowed to cool below 60°C before the content were blown down. The digester's initial temperature, pressure and starting time were all noted, and the various changes in these parameters were also recorded. The resultant pulp was subjected to thorough washing with plenty of water. When it was observed that subsequent washing resulted in no further change in color, the pulp was transferred into the valley beater for processing into a more refined pulp before the bleaching operation.

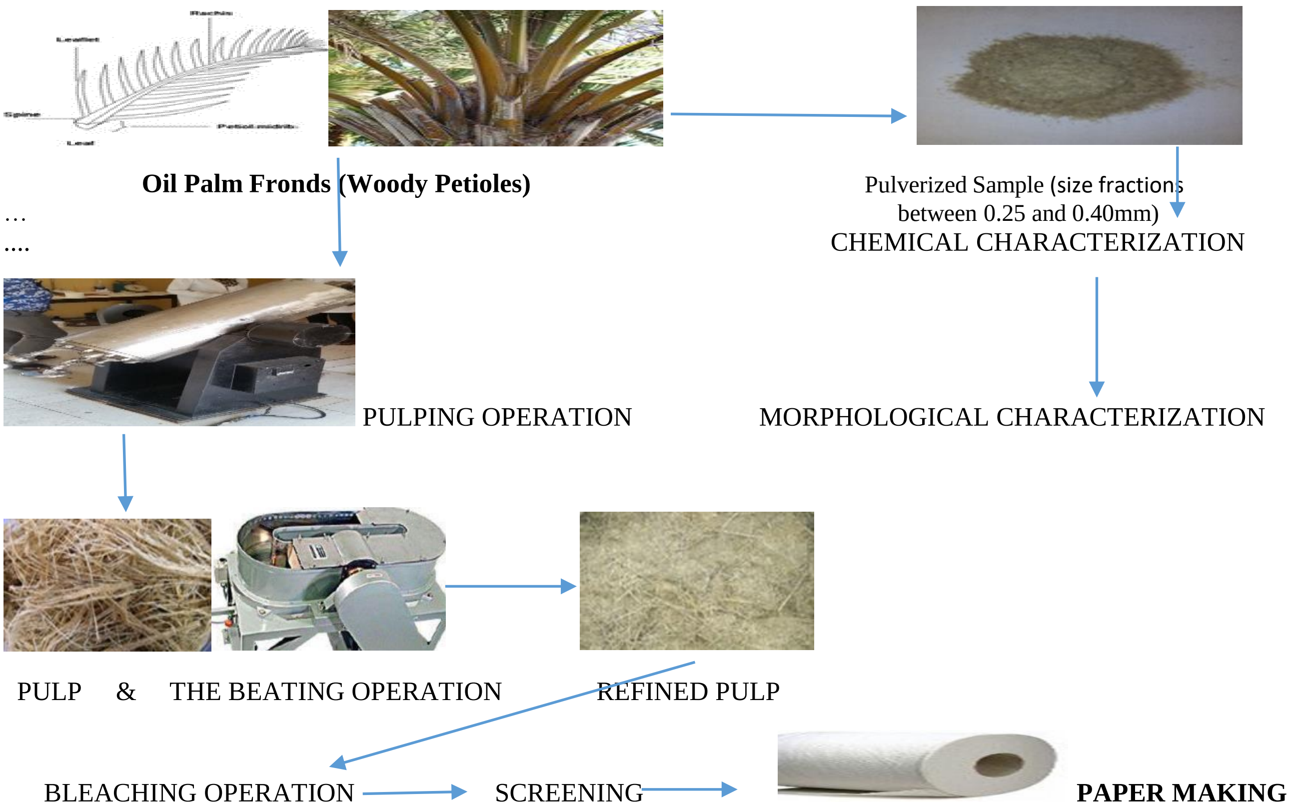

Figure 1: Steps in Oil Palm Fronds (Woody Petioles) fractionation and conversion to paper

Table 2: Pulping Conditions for the Sample (Oil Palm Woody Petioles) Investigated

Conditions of Pulping Operation	Parameters
Air dry weight of Oil Palm Woody Petioles (kg) (A.D)	2
Liquor charge (%)	10, 15 and 20
pH of white liquor	6
pH of black/spent liquor	3
Liquor/biomass ratio	4:1
Maximum cooking temperature (°C)	150, 160 and 170
Time to reach maximum temperature (minutes)	41
Time at maximum temperature (minutes)	19, 49 and 79
Over-all cooking time (minutes)	60, 90, 120
Blow-down temperature (°C)	60

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RESULTS AND DISCUSSION

Analyses of the Chemical Properties of Oil Palm Woody Petioles

Chemical analyses of Oil Palm Woody Petioles was conducted. Table 3 illustrates the results of the characteristics obtained. The moisture content and hot water solubility of Oil Palm Woody Petiole is quite low, while the extractive content is also low compared to other agricultural residues studied in literature, which means that Oil Palm Woody Petioles contain less substances like waxes, fats, resins, phytosterols, non-volatile hydrocarbons, low molecular weight carbohydrates, salts and other water-soluble substances.

Table 3: Chemical Characterization of Oil Palm Woody Petioles

Parameters	Oil Palm Woody Petioles (Oil Palm Frond)
Source of Sample (Geographical Location)	Southern Nigeria
Age of Maturity (years)	6-8
Moisture content (%)	9.09
Hot Water Solubility (%) @ 80-95ºC for 0.5hr	6.50
Extractives (%)	7.11
Ash (%)	3.10
Alpha-cellulose (%)	40.39
Lignin (%)	11.09
Holocellulose (%)	63.75
Pulp yield (%)	41.79

A higher content of extractives would be converted into pitch, which would adversely affect the runnability of process equipment and the quality of furnished paper because of shadow marking. Oil Palm Woody Petioles has low content of extractives hence would require a moderately low dose of pulping liquor to neutralize acidic extractive, which would have little or no effect on the pulp yield and might create less digester corrosion caused by extractives. The higher the lignin content, the greater the stiffness of fibers. Lignin contents in Oil Palm Woody Petioles is quite low and almost similar with that of the softwood. As observed in Table 3, Klason lignin contents in Oil Palm Woody Petioles is (11.09%). In practice, this means that these materials would need milder pulping conditions (lower temperatures and chemical charges) in order to reach a satisfactory kappa number and it would also undergo bleaching more easily and with the utilization of fewer chemicals. The average fiber dimensions of Oil Palm Woody Petioles investigated in this research study is shown in Tables 4. Despite the decrease in the value of lignin, the extractive content, and ash in addition to decrease in chemical consumption and cooking time, pulp yield was surprisingly low. Papers made from this type of fibers might show reduced water absorbency.

Analyses of the Morphological Properties of Oil Palm Woody Petioles

The morphological parameters of Oil Palm Woody Petioles investigated are presented in table 4. The Fiber length of Oil Palm Woody Petioles is 1.0254, the Fiber diameter is 85.13µm, fiber lumen width is 4.22µm while the Fiber cell wall thickness is 6.11µm. Softwood fiber is between 3-5 mm long and about 39 to 41 µm wide, meaning that Woody Petioles length and width are about 79.56% to 88.70% and 69.22% to 86.43% lower, respectively, than those of softwood.

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Table 4 Morphological properties of Oil Palm Woody Petioles (Palm Frond)

Plant Materials	Fibre length, (L), (mm)	Fibre diameter, (D), (µm)	Fibre Lumen, (d), diameter (µm)	Fibre wall/Cell wall thickness, (w), (µm)
Woody Petioles	1.0254	85.13	4.22	6.11

Table 5: Biometry/Morphological Indices of Fiber Dimensions

Plant materials	Derived Values			
	Slenderness ratio, L/D	Flexibility coefficient, (d/D)×100	Runkel ratio, 2w/d	Rigidity Coefficient, 2w/D
Woody Petioles	12.0451	4.96	2.90	0.14

Table 6. Best 20 Pulping Scenarios showing Independent Variables and Dependent Variables for the Pulping Process from 81 Experimental Runs

Experimental Run	Factor 1	Factor 2	Factor 3	Factor 4	Response 1
	A:Anthraquinone (% o.d. biomass)	B: Soda Charge (%)	C:Temperature (°C)	D: Cooking Time (Minutes)	Pulp Screened Yield %
5	0.25	15	150	60	41.79
29	0.25	10	150	90	42.45
30	0.50	10	150	90	43.78
31	0.00	15	150	90	42.88
32	**0.25**	**15**	**150**	**90**	**47.99**
33	0.50	15	150	90	46.76
34	0.00	20	150	90	44.44
35	0.25	20	150	90	45.45
36	0.50	20	150	90	46.66
37	0.00	10	160	90	44.77
38	0.25	10	160	90	43.89
39	0.50	10	160	90	42.94
41	0.25	15	160	90	45.96
42	0.50	15	160	90	44.98
44	0.25	20	160	90	43.88
45	0.50	20	160	90	44.55
50	0.25	15	170	90	40.77
53	0.25	20	170	90	43.77
59	0.25	15	150	120	43.79
60	0.50	15	150	120	42.76

AQ= anthraquinone charge; TY = total yield; SY: screened yield

Table 7. Model Summary Statistics

Source	Std. Dev.	R^2	Adjusted R^2	Predicted R^2	PRESS	
Linear	1.91	0.2872	0.1732	-0.0109	129.56	
2FI	2.00	0.4077	0.0960	-0.6048	205.68	
Quadratic	**1.41**	**0.7676**	**0.5507**	**-0.0591**	**135.75**	**Suggested**
Cubic	1.46	0.9504	0.5206		*	Aliased

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We focus on the model maximizing the **Adjusted R^2** and the **Predicted R^2**. A negative **Predicted R^2** implies that the overall mean may be a better predictor of your response than the current model. In some cases, a higher order model may also predict better. **Adeq Precision** measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 6.111 indicates an adequate signal. This model can be used to navigate the design space.

Table 8. ANOVA for Response Surface Quadratic Model [Partial sum of squares]

Source	**Sum of Squares**	**df**	**Mean Square**	**F-value**	**p-value**	
Model	98.38	14	7.03	3.54	0.0103	significant
A-Anthraquinone	1.94	1	1.94	0.9770	0.3386	
B-Soda Charge	0.6730	1	0.6730	0.3389	0.5691	
C-Temperature	1.74	1	1.74	0.8785	0.3635	
D-Cooking Time	0.0013	1	0.0013	0.0006	0.9800	
AB	0.4016	1	0.4016	0.2022	0.6594	
AC	1.75	1	1.75	0.8813	0.3627	
AD	1.05	1	1.05	0.5281	0.4786	
BC	4.95	1	4.95	2.49	0.1351	
BD	1.04	1	1.04	0.5258	0.4795	
CD	2.39	1	2.39	1.20	0.2898	
A^2	8.18	1	8.18	4.12	0.0605	
B^2	3.69	1	3.69	1.86	0.1927	
C^2	0.1995	1	0.1995	0.1005	0.7556	
D^2	36.19	1	36.19	18.22	0.0007	
Residual	29.79	15	1.99			
Cor Total	128.17	29				

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The **Model F-value** of 3.54 implies the model is significant. There is only a 1.03% chance that an F-value this large could occur due to noise. **P-values** less than 0.0500 indicate model terms are significant. In this case D^2 is a significant model term. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model. The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal the VIFs are 1; VIFs greater than 1 indicate multi-colinearity, the higher the VIF the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerabl

Final Equation in Terms of Coded Factors

$$\text{Pulp Screened Yield} = +45.41 + 0.5283A + 0.3096 - 0.5509C - 0.0191D + 0.2343AB - 0.5762AC + 0.5425AD - 1.06BC + 0.7324BD - 0.6852CD - 1.28A^2 - 0.8034B^2 - 0.2288C^2 - 2.88D^2$$

Final Equation in Terms of Actual Factors

$$\text{Pulp Screened Yield} = -124.09024 + 39.93275\,\text{Anthraquinone} + 3.92412\,\text{Soda Charge} + 1.25748\,\text{Temperature} + 0.848500\,\text{Cooking Time} + 0.187480\,\text{Anthraquinone} * \text{Soda Charge} - 0.230464\,\text{Anthraquinone} * \text{Temperature} + 0.072332\,\text{Anthraquinone} * \text{Cooking Time} - 0.021153\,\text{Soda Charge} * \text{Temperature} + 0.004883\,\text{Soda Charge} * \text{Cooking Time} - 0.002284\,\text{Temperature} * \text{Cooking Time} - 20.53441\,\text{Anthraquinone}^2 - 0.032136\,\text{Soda Charge}^2 - 0.002288\,\text{Temperature}^2 - 0.003195\,\text{Cooking Time}$$

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients. The equation in terms of actual factors can be used to make predictions about the response for given levels of each factor. Here, the levels should be specified in the original units for each factor. This equation should not be used to determine the relative impact of each factor because the coefficients are scaled to accommodate the units of each factor and the intercept is not at the center of the design space.

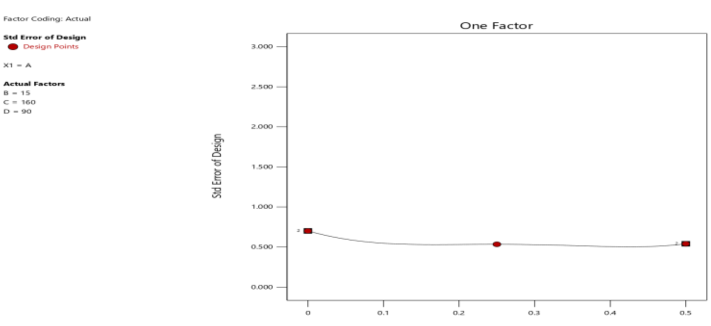

Figure 2: One factor interraction of independent variable

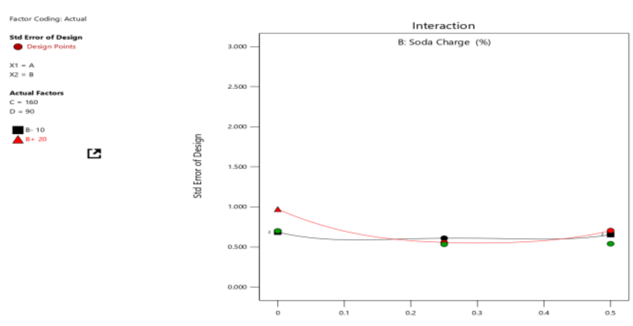

Figure 3: Multiple factor interraction of independent variables

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Table 9: Pearson's r showing the polynomial components of all independent variables

	Intercept	A-Anthraquinone	B-Soda Charge	C-Temperature	D-Cooking Time	AB	AC	AD	BC	BD	CD	A^2	B^2	C^2	D^2
Intercept															
A-Anthraquinone		1.000	0.170	0.209	0.012	-0.118	-0.456	-0.362	-0.080	-0.227	-0.038	0.190	-0.061	0.049	0.209
B-Soda Charge		0.170	1.000	0.000	0.000	0.229	-0.069	-0.167	-0.530	-0.500	0.089	-0.100	0.000	0.189	0.000
C-Temperature		0.209	0.000	1.000	0.014	0.008	0.385	-0.032	0.183	0.129	-0.117	0.072	-0.013	-0.395	0.003
D-Cooking Time		0.012	0.000	0.014	1.000	-0.144	0.015	0.506	0.111	0.000	-0.410	-0.187	-0.153	0.139	-0.376
AB		-0.118	0.229	0.008	-0.144	1.000	-0.093	0.010	-0.108	-0.306	0.087	0.110	0.157	-0.104	0.104
AC		-0.456	-0.069	0.385	0.015	-0.093	1.000	0.189	0.098	0.139	-0.007	0.033	-0.156	0.039	-0.039
AD		-0.362	-0.167	-0.032	0.506	0.010	0.189	1.000	0.118	0.334	0.008	-0.040	-0.179	0.080	-0.080
BC		-0.080	-0.530	0.183	0.111	-0.108	0.098	0.118	1.000	0.177	-0.126	-0.141	0.000	0.000	-0.134
BD		-0.227	-0.500	0.129	0.000	-0.306	0.139	0.334	0.177	1.000	-0.178	0.000	0.000	-0.189	0.000
CD		-0.038	0.089	-0.117	-0.410	0.087	-0.007	0.008	-0.126	-0.178	1.000	0.085	-0.009	0.099	0.171
A^2		0.190	-0.100	0.072	-0.187	0.110	0.033	-0.040	-0.141	0.000	0.085	1.000	-0.029	-0.045	0.196
B^2		-0.061	0.000	-0.013	-0.153	0.157	-0.156	-0.179	0.000	0.000	-0.009	-0.029	1.000	-0.296	-0.259
C^2		0.049	0.189	-0.395	0.139	-0.104	0.039	0.080	0.000	-0.189	0.099	-0.045	-0.296	1.000	0.148
D^2		0.209	0.000	0.003	-0.376	0.104	-0.039	-0.080	-0.134	0.000	0.171	0.196	-0.259	0.148	1.000

Graphical Optimization

Influence of morphological properties on Biometry (Slenderness, Flexibility, Runkel and Rigidity coefficient) of the Oil Palm Woody Petioles fiber investigated are shown in Tables 5. The Slenderness ratio of Oil Palm Woody Petioles is 12.0451 while the Flexibility coefficient is 4.96. Generally, there are four different types of fibers which are classified under flexibility ratio **(Bektas et al., 1990)**: (1) High elastic fibers having elasticity coefficient greater than 75; (2) Elastic fibers having elasticity ratio between 50 to75; (3) Rigid fibers having elasticity ratio between30 to 50; (4) High rigid fibers having elasticity ratio less than 30. According to this classification, flexibility coefficient of Oil Palm Woody Petiole is in uniformity with hardwoods. When Runkel proportion is greater than 1, it indicates that a fiber has thick wall and cellulose obtained from this type of fiber is less suitable for paper production; when it is equal to 1, it specifies that a cell wall has medium thickness and cellulose obtained from this type of fiber is suitable for paper production. When the rate is less than 1, it points out that a cell wall is thin and cellulose obtained from this fiber is the most suitable for production of paper

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(Eroglu et al., 1980; Xu et al., 2006). Runkel value of Oil Palm Woody Petioles is greater than 1 and according to the Runkel classification, it specifies that the fiber has thick wall and cellulose obtained from this type of fiber is not very suitable for paper production except when used in blend with certain long fiber pulp plants like kenaf to boost its potential for producing certain types of paper materials. The low content of lignin in Oil Palm Woody Petioles (11.09%) made the fibers appear less tough and less stiffer compared to other fibers like pineapple leaves, wheat straws, corn stalk/sheets, coconut fruit bunch, elephant and lemon grass and other fibrous agro-wastes investigated in other studies furnishing rigidity coefficient following the order: (Highest) Bagasse (0.59)>Coconut (0.58)>EFB(0.57)> Pineapple leaves(0.56) > Corn(0.50)> Lemon grass (0.44)>kenaf(0.39) >Elephant grass (0.34) > Wheat(0.17)> Oil Palm Woody Petioles(0.14)>Rice(0.06)>(lowest) **(Chibudike et al., 2011)**. This is probably because lignin provides compressive strength to plant tissue and individual fibers and stiffens the cell walls, to protect carbohydrates from chemical and physical damages. Detailed research work on these agro-wastes is not included in this report.

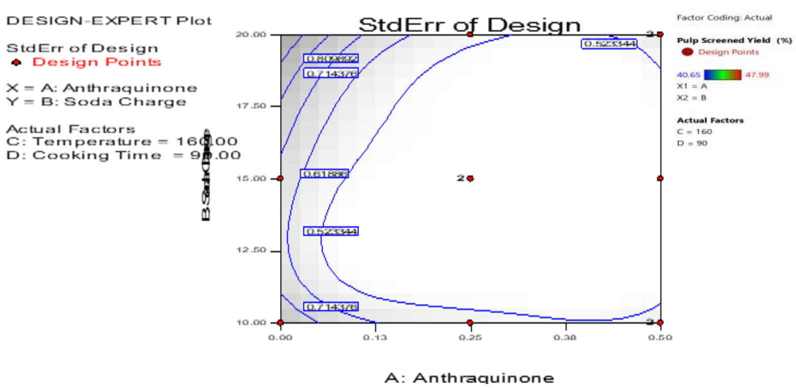

Figure 4: Standard Error of Design showing the effect of AQ

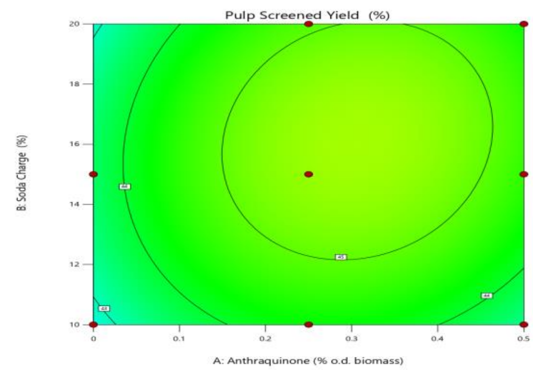

Figure 5: Model Graph of Pulp Screened Yield

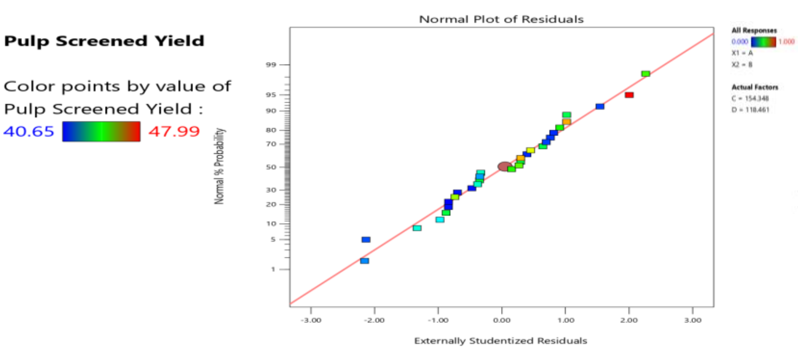

Figure 6: Normal Plot of Residuals

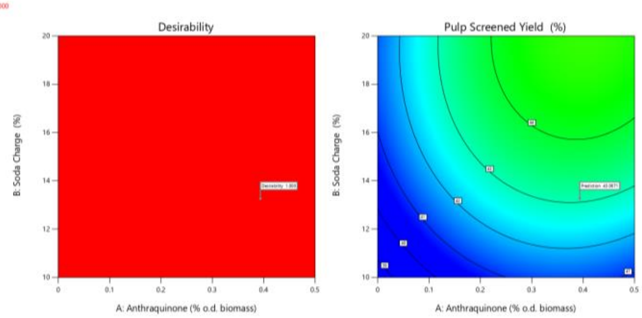

Figure 7: Desirability Plot of Numerical

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Table 10. Optimization Solution Found for the Resonse Surface Mean Model (10 Solutions found. Number of Starting Points 10)

Solutions Number	Anthraquinone	*Soda Charge *	Temperature*	Cooking Time *	Desirability	
1	0.35	16.01	163.39	73.00	1.000	Selected
2	0.27	15.01	166.02	115.78	1.000	
3	0.15	19.43	156.22	68.50	1.000	
4	0.34	10.30	161.51	70.55	1.000	
5	0.43	12.30	162.11	75.69	1.000	
6	0.44	19.33	159.81	110.93	1.000	
7	0.37	15.28	156.01	90.53	1.000	
8	0.43	17.66	152.47	87.37	1.000	
9	0.33	16.96	157.69	73.06	1.000	
10	0.34	19.50	151.99	89.21	1.000	

*Has no effect on optimization results.

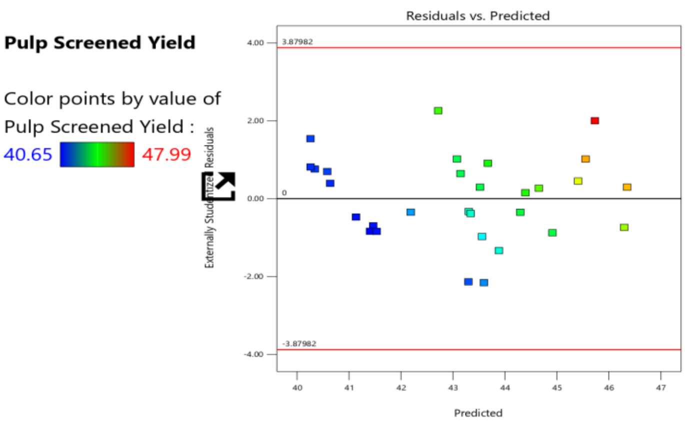

Figure 8: Scattered plot of Residual vs Predicted Values

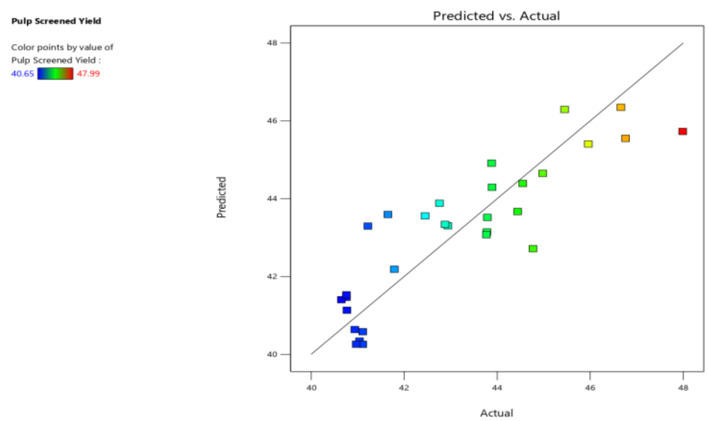

Figure 9: Scattered plot of Predicted vs Actual Values

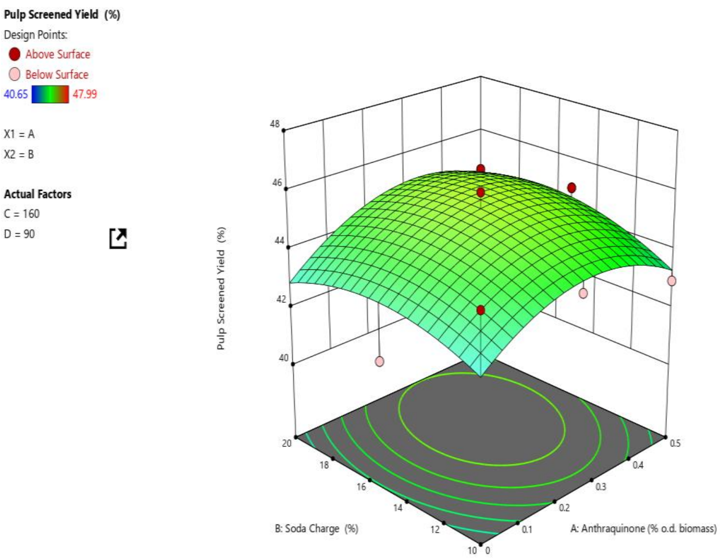

Figure 10: 3D Model Graph showing Optimum Design Points

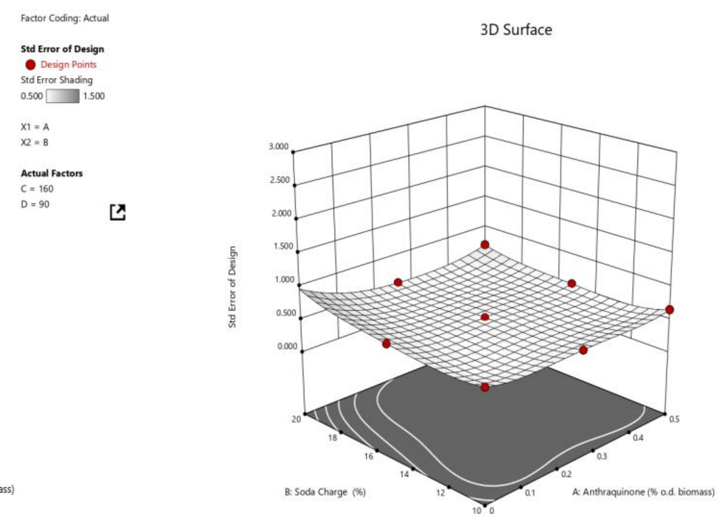

Figure 11: 3D Surface Model Graph showing various Design Points

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At the end of the pulping operation, the pH of the cooking liquor dropped from 6.0 to 3.0 indicating that the anthraquinone performs certain functions besides that of Sodium Hydroxide increasing the hydroxide content of the liquor, thereby increasing the rate of lignin removal. The black liquor obtained after the digestion process was not discarded, but rather employed in further chemical analysis. The black liquor was distinctly alkaline, but not caustic owing to the fact that a large part of the alkali was present in the form of neutral compounds, hence the need for chemical recovery.

The Acid-insoluble lignin in the Oil Palm Woody Petioles was determined to be 11.09%. This necessitated the use of a mild liquor ranging from 10 to 20% for the cooking operation. Ash content of the Oil Palm Woody Petiole recorded 3.1% which is a measure of inorganic mineral present in the sample. These values afford more effective reuse of spent chemical after the pulping operation. The fibre length of the Oil Palm Woody Petiole recorded 1.0254mm which processes the tendency to contribute to tensile strength of its corresponding furnished paper samples. Alpha cellulose recorded a value of 40.39% which indicates the amount of un-degraded higher molecular weight cellulose present. The pulp yield is quite low i.e. 41.79%. This does not really exert a very good beneficial effect. Temperature coefficient of delignification was determined to be approximately 2.0, indicating that an increase in cooking temperature by 5% might result in an increase in the rate of lignin removal.

All analytical tests were carried out in duplicate. Statistical analysis was performed using the Design Expert software. Data were analysed by the analysis of variance (ANOVA), and p-value lower than 0.05 was considered significant in surface response analysis. The optimal values of the operation parameters were estimated by the three-dimensional response surface analysis of the independent variables (Cooking time, Liquor charge and Liquor/Biomass Ratio respectively) and the dependent variable (Pulp Yield =Y%). A negative "Pred R-Squared" implies that the overall mean is a better predictor of the response than the current model. The residuals are represented graphically by means of a residual plot as shown in figure 6. This normal probability plot indicates whether the residuals follow a normal distribution, thus follow the straight line. Here, the scatter had a definite pattern along the straight line which indicates that a transformation of the response may provide a better analysis.

Hence, the optimum pulping condition according to the selected optimization solution found is summarized as 73 minutes cooking time, 16.01% soda-charge, 163.39 ºC cooking temperature and 0.35% anthraquinone charge, oven dried weight of biomass.

CONCLUSION AND RECOMMENDATION

Pulping is a chemical-technological process for the production of cellulose fibers for paper-making from woods and other plant materials. Paper strength depends on the cellulose content of a raw plant material. Cellulose content was at a satisfactory level (above 40%) for the fiber utilized in the present study. Overall, Oil Palm Fronds fibers (Woody Petioles) appear to be suitable for producing paper products due to lower lignin and extractive components as well as higher in cellulose content, though short in fiber length. Literature studies about softwoods revealed that elasticity coefficient was found within the range 50-70. Examining this information given, and comparing it with data generated in this research study, it seems Oil Palm Woody Petiole is similar to other softwood fibers. Depending on all the investigation carried out, it is possible to conclude that Oil Palm Woody Petioles when used in blend with long fiber pulp plants would be suitable for good brightness paper production. Considering the summary of analyses of data generated in this research work in comparison with literature and previous studies on other agro-base fiber residues, Oil Palm Woody Petiole when used in blend with other long fibrous pulp plants like kenaf can be very suitable to furnish good paper based materials like fiber plate, rigid cardboard, cardboard, tissue, corrugating medium, printing and writing paper.

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Agricultural wastes, annual plants and non-wood materials have attained such importance in the world cellulose economy, that to ignore their relevance in the pulp and paper industry would result in a complete lack of balance. In a world where virgin pulp sources are scarce, and environmental concerns require reduction in cutting down green forest, agricultural residues could become a good source of fiber in the tropical regions of the world where they are grown. The search for local long fiber pulp material which can be easily propagated remains one of the most important key desirderatum for the eventual resuscitation of the present mom bund paper industries of Nigeria. One important way of stemming the tide of imports is to find a good substitute to fine pulp for the use of the Nigeria paper companies when they eventually start producing. Besides being an innovation and new entry into the pulp map, Oil Palm Woody Petioles can become the best gift of FIIRO into the future pulp market of the tropical world.

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