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## Research Article

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# Optimization of extraction techniques for processing and utilization of *Cyperus Dichrostachys A. Rich* Plant as fiber

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**Abstract:** The Textile industry is an important contributor to the GDP of countries worldwide. Both natural and synthetic fibers are the main raw materials for this sector. Environmental concerns, depletion of non-renewable resources, the high price of oil and limited oil reserves with consumer demand is driving research into cheap, biodegradable, sustainable, renewable and abundantly available green materials. Natural fibers are of the good substitute sources for swapping synthetic fibers and reinforcing polymer matrices because of their contributions in maintaining of ecology, nature of disposal, low energy requirement for processing and sustainability. The current research emphasizes on evaluating and determining the best extraction methods to process and treat *Cyperus Dichrostachys A. Rich* plant in order to make the fiber suitable for variety of applications. *Cyperus Dichrostachys A. Rich* plant was treated with two conditions (cold and warm conditions) using statistically planned tests. Process conditions were optimised using central composite design methodology with the experimental design. Under optimised conditions, the strength and fiber yield of CDA fibers were significantly compared. The strength and fiber yield of the fiber was at maximized with optimized conditions and use for valorisation applications.

**Key words:** *Cyperus Dichrostachys A. Rich*, Lignocellulosic fiber, Natural fiber

## 1. Introduction

At present, most manufacturing and industrial sectors are fascinated in evolving sustainable development and environmental friendly products ([Wang and LV JN, 2007](#), [Debnath, 2020](#), [Wang et al., 2007](#)). They are in a paradigm shift to sustainability in all aspects of fashion ([Radhakrishnan, 2020](#)), eco-friendly polymer composite manufacturing principles ([Balli et al., 2019](#)), eco-friendly building materials production and utilization ([Nowotna et al., 2019](#), [Pleitner et al., 2019](#)), organic production systems with the main consumable natural fibers like cotton ([Günaydin et al., 2019](#)). The main concern is maintaining natural environment by following friendly production and manufacturing concepts that do not devour any dangerous provisions with no poisonous releases to the natural atmosphere at any circumstances.

The utilization of natural fibers in different applications becomes more issue that is critical. There are varied applications of plant fibers in composite industries, engineering and manufacturing applications ([Siakeng et al., 2019](#), [Garcia Filho and Monteiro, 2019](#), [Battezzatore et al., 2019](#), [Sanjay et al., 2019](#)), in fiber-reinforced polymer composites engineering applications ([Naveen et al., 2019](#), [Mochane et al., 2019](#), [Islam and Rahman, 2019](#)).

Moreover, they are becoming common in *structural applications* ([Feng and Malingam, 2019](#)) especially in rope making ([Jahangir Ali Fathima Benazir, 2010](#)), and automotive applications like shock absorbers, windshields ([Balakrishnan et al., 2019](#)), doors, ceilings ([Vijay, 2016](#), [Selvakumar and Meenakshisundaram, 2019](#)). They are also vital sources for reinforcement materials with polymer matrix for small-scale applications ([Rajini, 2019](#)).

The varied application of plant fibers is the fact that they have exceptional characteristics of flexibility, eco-friendly nature, low cost, renewability, and accessibility, less harmful effect to human beings, bio degradability, ease of extraction, fabrication and manufacturing, high strength to weight ratio with simple and harmless implementation process as compared with synthetic fibers.

Moreover, natural resources have played foremost role in the economic activities with substantial sustainability to the growth of the world in progress through socio-economic development ([Satyanarayana et al., 2007](#), [Kim et al., 2019](#), [Verma et al., 2019](#), [Tomczak et al., 2007](#)). The maximum utilization of such resources done with new developments and products in advance with preventing environmental pollution and generating employment opportunities followed by improvement of people's living standards. One of the most abundant foundations of such resources

has been lignocellulosic materials, have been utilized since 6000 BC available from many plant parts ([Satyanarayana et al., 2007](#)).

Natural fibers are generally extracted with a simple and economical method known as retting process after which they are exposed to different chemical treatment approaches. Normally the makeup of the lignocellulosic fibers consists of cellulose, hemi-cellulose, and lignin. The suitable natural fibers extraction represents a significant test happening during the processing of plant fibers. The most common methods to separate the plant fibers were dew retting and water retting processes. Depending on the fiber category, the methods need nearly 15 to 30 days for the removal of waxes, pectin substance, hemicellulose and lignin. Alternative methods just like mechanical extraction and chemical treatments have been introduced to reduce long processing time. In retting process, the existence of the bacteria and moisture in the plants allows to break down large parts from cellular tissues and its adhesive substances that surround the fibers, enabling the separation of individual fibers from the plant. The reaction time must be carefully evaluated when using dew or water retting because excessive retting can cause difficulties for the separation of individual fibers or may weaken the fiber strength.

On the other hand, the mechanical extraction process of fibers can produce high-quality fibers with shorter retting time, however in respect to dew or water retting process the technique is more expensive.

Retting is widely used extraction methods of fibers from the plant parts. It is a process of controlled degradation of the plant to allow the fiber to be separated from the woody or non woody core and thereby improving the ease of extraction of the fibers through biological activity of microorganism, bacteria or fungi from the environment to degrade the pectic polysaccharides from the bon fiber tissue and, thereby, separate the fiber bundles. The fiber separation and extraction process has a major impact on fibre yield and final fibre quality. It influences the structure, chemical composition and properties of the fibres. Most scholars divide retting procedures as biological, mechanical, chemical and physical fibre separation process as shown **Figure 1**.

Biological retting may be either natural or artificial retting. Natural retting comprises dew or field retting and cold water retting. Dew or field retting is commonly applied retting process with appropriate moisture and temperature ranges. Then, the crops should remain on the fields until the microorganisms have separated the fibres from the cortex and xylem then the stalk is dried and baled.

Moreover, Cold water retting by using anaerobic bacteria that breakdown the pectin of plant straw bundles submerged in huge water tanks, ponds, hamlets or rivers and vats. The process takes between 7 and 14 days and depends on the water type, temperature of the retting water and any bacterial inoculum. Even though the process produces high quality fibres, environmental pollution is high due to unacceptable organic fermentation waste waters.

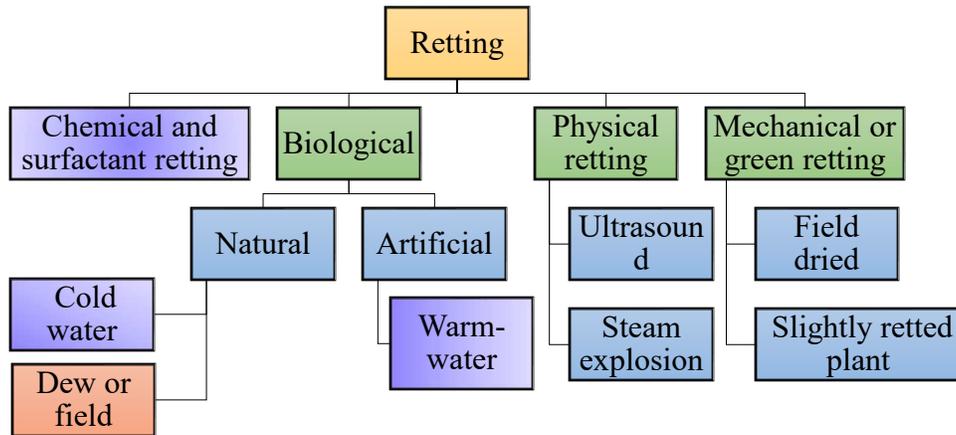


Figure 1: Summary of classification of extraction methods

Source: (Morrison Iii et al., 2000, Parikh et al., 2002, Abdul Khalil et al., 2008)

Artificial retting involves warm-water or canal retting and produces homogeneous and clean fibres of high quality in 3–5 days. Plant bundles are soaked in warm water tanks. After sufficient retting, the bast fibres are separated from the woody parts. The sheaves or hurds are loosened and extracted from the raw fibres in a breaking or scotching process.

Mechanical or green retting is much simpler and more cost-effective alternative to separate the bast fibre from the plant straw. The raw material for this procedure is either field dried or slightly retted plant straw. The bast fibers are separated from the woody part by mechanical means. Weather-dependent variations of fiber quality are eliminated. However, the produced green fibers are much coarser and less fine as compared to dew or water retted fibers (Thomas et al., 2011).

Physical retting includes ultrasound and steam explosion method. In ultrasound retting, the stems obtained after the harvest are broken and washed. Slightly crushed stems are immersed in hot water bath that contains small amounts of alkali and surfactants and then exposed to high-intense ultrasound. This continuous process separates the hurds from the fibre. The steam explosion method represents another suitable alternative to the traditional field-retting procedure. Under pressure and increased temperature, steam and additives penetrates the fibre interspaces of the bast

fibre bundles. The subsequent sudden relaxation of the steam leads to an effective breaking up of the bast fibre composite, which results in an extensive decomposition into fine fibres ([Morrison Iii et al., 2000](#), [Abdul Khalil et al., 2008](#)).

Chemical and surfactant retting ([Parikh et al., 2002](#)) is a retting process in which the fiber Plants submerged in heated tanks containing water solutions of sulfuric acid, chlorinated lime, sodium or potassium hydroxide and soda ash to dissolve the pectin component. The use of surface-active agents in retting allows the simple removal of unwanted non-cellulosic components adhering to the fibres by dispersion and emulsion-forming process.

Reddy et al has identified a new fiber from nappier grass that the fiber has good physical properties of length 142mm, diameter 0.255mm and aspect ratio 556.8 with chemical compositions of  $\alpha$ -Cellulose, (45.66%), Hemicellulose (33.67%) and Lignin (20.60%). It has also found that the Maximum stress of (75 mpa), Young's modulus (6.8 gpa) and Elongation at break (2.8 %) tensile properties make it feasible for utilizing the fibers as reinforcement for composites ([Reddy et al., 2012](#))

Different scholars have developed various natural fibers for different applications. Reddy et al has identified a new fiber from nappier grass and show that the fiber has good physical properties of length 142mm, diameter 0.255mm and aspect ratio 556.8 with chemical compositions of  $\alpha$ -Cellulose (45.66%), Hemicellulose (33.67%) and Lignin (20.60%). It has also found that the Maximum stress of (75 mpa), Young's modulus (6.8 GPa) and Elongation at break (2.8 %) tensile properties make the fiber feasible for utilization as reinforcement for composites (Reddy et al., 2012).

Ethiopia can nurture a diversity of naturally fiber-forming plants. One of the most common plant that is currently grown commercially and could be of interest for handcrafting, packaging and traditional equipment is *Cyperus Dichrostachus A. Rich*(CDA)([Reid et al., 2014](#), [Bayeh, 2013](#), [Chekole, 2011](#), [Larridon et al., 2011a](#)) plant. It is just a tough plant, traditionally used for weaving of huts, mats, and baskets in Ethiopia and different countries in Africa ([Debnath, 2020](#), [Larridon et al., 2011b](#)). It has a decorative nature used to weave baskets, sleeping and sitting mats, rolled twines, more traditionally cereal and crop baskets, food baskets, crop and powder filters, covering equipment for cooking devices and other home apparatus.

Yet, apart from traditional craftworks, there are no research works done on this plant regarding the characterization and modification of its intrinsic properties. Therefore, adaptation of the fiber into

valuable goods was the anxiety of this research work with technologies employed to transform into beneficial products with modified extraction technique of the fiber. Most of these distinctive extraction features were determined throughout the progress of each experimental tests and successive fiber drawing out or extraction stages. The paper investigates the optimum extraction techniques of CDA fiber with detailed scientific justifications.

## 2. MATERIALS AND METHODS

### 2.1. Materials

**CDA plants:** Well matured CDA grasses of non woody stems as shown in **Figure 2** that have been used for this study were collected from Chiss Abay around the Blue Nile River, Bahir Dar, Ethiopia.



Figure 2: Collected CDA non woody stems

**Equipment:** In this research, plastic bottles, oven dryer and conditioning chamber from EiTEX laboratory were utilized throughout the course of the study.

### 2.2. Methods

#### 2.2.1. Experimental design

Response surface methodology by central composite design (CCD) employing the multivariate approach was used to design the experiment as well as to do the analysis of the results. Because it enables the development of concept using fewer experiments, without wastage of a large amount

of time and resource the experimental number of CCD is as per  $k^2 + 2k + CP$  where  $C_p$  is the replicate number of centre points and  $k$  is the factor number. It is more advantageous to decide the ideal condition of the experimental parameters ([Bezerra et al., 2008](#), [Gunst, 1996](#)). It leads to the construction of reliable response surfaces that are characterized by high adherence to the experimental data describing the reality being studied ([Myers and Montgomery, 1995](#)). Each run in the course of subsequent experimental design was triplicate and the average values were used. In order to establish better fiber extraction, DOE was applied with a pre-determined set of factors. Following completion of the treatment according to the developed model design, CCD optimized the obtained significant variables on the efficiency of extraction procedure.

### 2.3. Extraction methods of CDAF plant

#### 2.3.1. Preparation

CDA stalks were collected and treated using different extraction techniques. The mature CDA stems were stratified and individualized into thinner and flat strips. Then each stem was separated and extracted with mechanical, cold water, and Hot water extraction mechanisms ([NagarajaGanesh, 2016](#)). All samples were striped manually with mechanical action to speed up the extraction time of the bast and core parts of the non-woody stem. All the samples were kept at ambient conditions for a minimum of 3 weeks before the extraction process. The whole extraction process was carried out by combining mechanical and retting techniques followed by stripping, washing, squeezing and sun drying.

Table 1: factors considered in the extraction process

Method	Considered factors	Condition	Reference
Cold Water	(NaOH, MLR, Time)	Room temperature	Morrison Iii et al., 2000, Parikh et al., 2002, Abdul Khalil et al., 2008, NagarajaGanesh, 2016
Worm/hot/ Water	(NaOH, MLR, Time and Temperature	Room temperature up to 78°C	

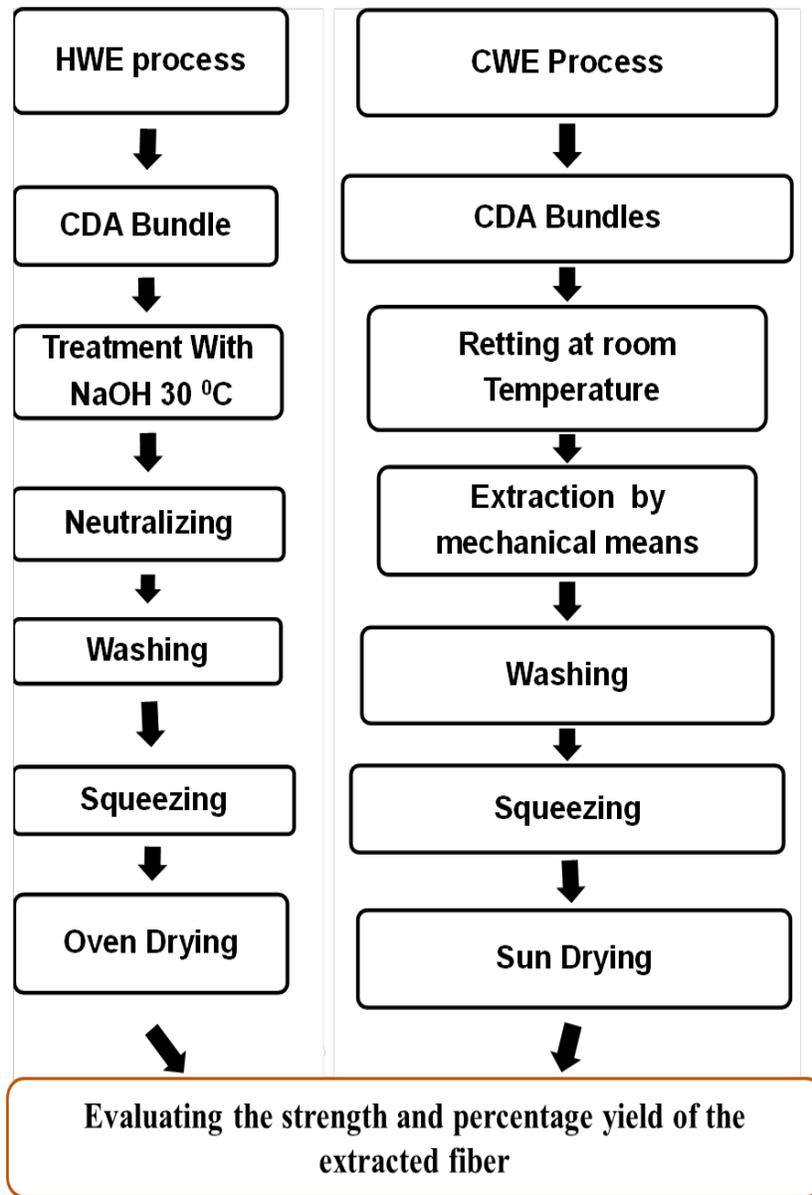


Figure 3: The general extraction process of CDA fibers

### **2.3.2. Cold water extraction method**

The non-woody stems were cut into 5 cm and placed into 20 plastic beakers in cold water at room temperature in aerobic environment with different NaOH concentration and time. The non-woody stems underwent soaking in water for a minimum of 15 days and a maximum of 40 days to undergo microbial and chemical degradation through retting. Thereafter, the stalks were dried in natural sunlight to remove moisture content after completion of the retting operation. Then the extracted fibers were set in a controlled laboratory environment for 24hrs before characterization ([Indran, 2015](#)).

### **2.3.3. Hot water extraction method**

Known weight of CDA plant samples were treated in NaOH solution with different concentrations. For the optimization process the following combinations were used; MLR (6-17), NaOH concentration, (1.7-5 %), time (1.5-4 hr.), and temperature (30-60 °C). After extraction, the extracted fiber was squeezed and dried using oven drying. Then the extracted fiber was stored in sealed plastic bags, in a controlled laboratory environment for further characterization.

### **2.3.4. Statistical analysis**

The data has statistically evaluated using DOE software, Version 11. Analysis of Variance (ANOVA) was used to examine the effects of extraction methods on the properties of CDA fibers both one factor effect and interaction effects with 3D surface were used. Least Significant Difference (LSD) method was used for further evaluation of the effect of extraction methods. LSD ranks the means and calculates the minimum value to be significantly different with each other at  $p \leq 0.05$ . From the ANOVA table, fit summary, signal-to-noise ratio value, Predicted  $R^2$  value and diagnostic plots, the significance of the models was checked, and four model equations were developed. Further the one factor analysis and interaction effect of each factor in each model was detailed and discussed with required scientific justifications. Finally, the optimized factors were determined and rework of extraction at the optimized extraction technique was done. The characterization of the new fiber was done for only the optimized extraction technique.

## **2.4. Analysis techniques of the response variables**

### **2.4.1. Fiber Yield**

The yield of the extracted fiber was measured by the percentage of the ratio between the final mass of the fibers after extraction process (Mf) and mass of the sample before extraction; process (Mi), which can be calculated using equation 2.1.

$$\text{fiber yield (\%)} = \left( \frac{M_f}{M_i} \right) \times 100$$

Equation 1: Determination of fiber yield percentage

### **2.4.2. Tensile strength**

The strength of each sample was tested with a minimum of 20 tests from each sample and the average value was calculated. Similarly the tensile properties of CDAFs were determined using an FAVIMAT+single fiber testing machine adopting ASTM D 3379 standard. All the tests were carried out at room temperature and 65±2% relative humidity. To ensure the repeatability of the results a minimum of 20 specimens were taken to perform the tensile tests with gauge length of 20mm and testing speed of 20mm/min over 210 cN load cell and pretension of 0.20cN/tex. Then the tenacity was calculated by using the formula given here:

$$\text{Fiber Tenacity} = \frac{\text{max tensile force (cN)}}{\text{linear density (Tex)}}$$

Equation 2: Estimation of CDA fiber tenacity

## **3. RESULTS AND DISCUSSION**

It is worth mentioning here that unlike any other bast fibers where the fiber extracted only from the outer layer of the plant, the whole non-woody stem of CDA plant was transformed into fiber. As it is clearly seen in the following sections all the response variables were dependent on the extraction parameters.

### **3.1. Cold water extraction (CWE)**

#### **3.1.1. Analysis of Variance for Yield**

As shown in the ANOVA Table 4, the Model was significant with F-value of 5.75; P-value of 0.0058, which was less than 0.0500; difference between Predicted R-squared and Adjusted R-squared values was -0.7181 which was less than 0.2 and signal-to-noise ratio value of 8.3726. All

those values proved that the model was fitting the data and can reliably be used to interpolation of the factor space, navigate the design space, and entailed that the model has a strong enough signal to be used for optimization. In this case; time, the combination effect of time and NaOH concentration and quadratic value of concentration were significant model terms on the fiber yield.



Table 2: ANOVA table for yield percentage of CWF

Source	Sum of Squares	Df	Mean Square	F-value	P-value	
Model	5953.60	9	661.51	5.75	0.0058	Significant
A-NaOH	28.56	1	28.56	0.2482	0.6291	
B-Time	3033.38	1	3033.38	26.36	0.0004	
C-MLR	51.53	1	51.53	0.4479	0.5185	
AB	840.30	1	840.30	7.30	0.0222	
AC	173.63	1	173.63	1.51	0.2474	
BC	23.02	1	23.02	0.2000	0.6642	
A <sup>2</sup>	1405.76	1	1405.76	12.22	0.0058	
B <sup>2</sup>	546.77	1	546.77	4.75	0.0543	
C <sup>2</sup>	64.35	1	64.35	0.5592	0.4718	
Lack of Fit	915.02	5	183.00	3.88	0.0814	Not significant
Predicted R <sup>2</sup>	0.6923		Adjusted R <sup>2</sup>	-0.0258	Adeq. Prec	8.3726

### 3.1.2. Effect of NaOH and time on yield

As shown in (Figure 4 a), the linear effect of changing the level of NaOH concentration has a quadratic relationship up to a maximum yield percentage of 51.25 % at the concentration level of 5%. Beyond this range the yield has decreased in quadratic function as NaOH concentration has increased. The reason was due to the quadratic effect of the chemical that increased the degradation rate of the fiber facilitated by time increments.

Figure 4 b strictly showed that fiber yield has a negative linear relationship with time. This was due to the increased amount of microorganisms that the cellulosic part was consumed as the duration of extraction time increased and reduced the fiber yield (Vignesh et al., 2016). The design model for final equation in terms of actual factors was given as

$$Yield (\%) = 59.34219 + 2.19606 X_2 + 0.273300 X_1 X_2 - 0.395319 X_1^2$$

Equation 3: Model equation of the actual factors for Yield (%) of CWF

Where,  $X_1 = \text{NaOH concentration}$ ,  $X_2 = \text{Time}$ ,

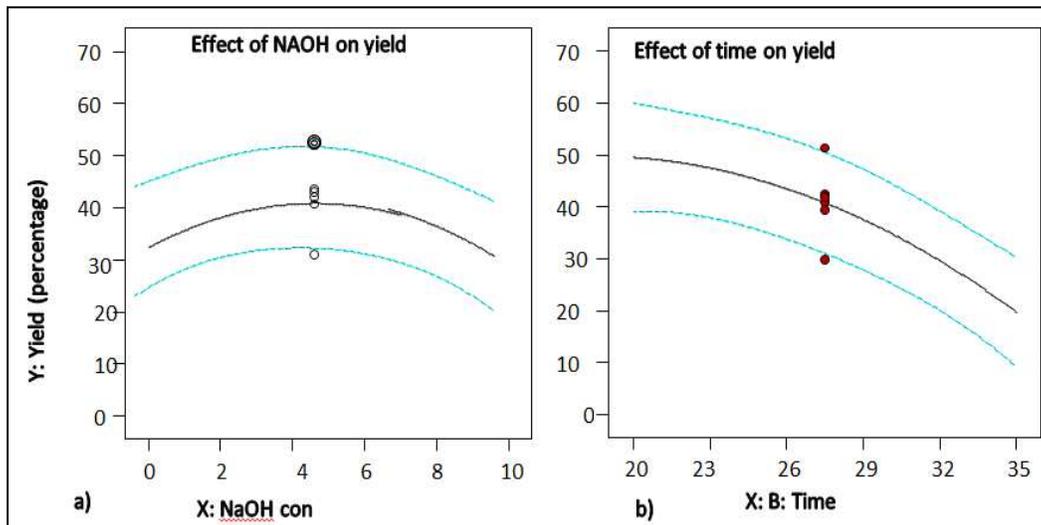


Figure 4: One factor effect analysis for fiber yield in CWE

### 3.1.3. Interaction effect of NaOH and time on yield

The interaction effect of time and NaOH concentration on fiber yield showed a quadratic effect up to a certain level. **Figure 5** established that maximum fiber yield of 52.88% was attained at 2.5 % NaOH concentration and 23 days of treatment. Beyond this range, their interactions show a reduction on fiber yield.

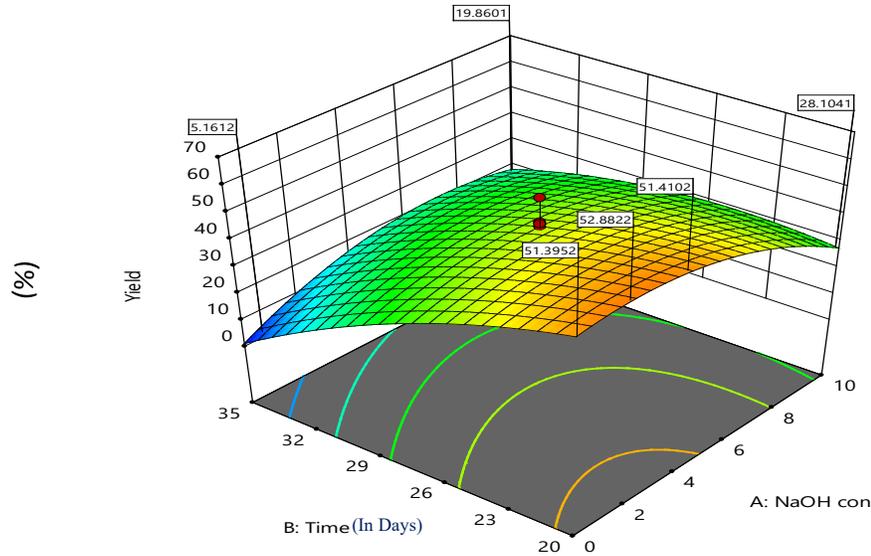


Figure 5: The interaction effect of NaOH and time on yield in CWE

### 3.1.4. Analysis of Variance for strength

As shown from the ANOVA table (5), F-value 17.39 entails that the model was significant with only a 0.01% probability that F-value enlarged occurrence due to noise levels. Similarly, it clearly states that model terms A, C, AC, A<sup>2</sup>, B<sup>2</sup> P-are the significant model terms since their p-values were less than 0.0500.

Lack of Fit F-value 0.91 point out that the Lack of fit was not significant relative to the pure error. There was a 54.22% chance that a "Lack of fit F-value" this large was occurred due to noise. Non-significant lack of fit was worthy need the model to fit.

Table 3: ANOVA table for strength in CWE

Source	Sum of Squares	Df	Mean Square	F-value	P-value	
Model	2.58	9	0.2871	17.39	<0.0001	Significant
A-NaOH	0.3710	1	0.3710	22.47	0.0008	
B-Time	0.0532	1	0.0532	3.22	0.1029	
C-MLR	0.9849	1	0.9849	59.66	<0.0001	
AB	0.0295	1	0.0295	1.79	0.2108	
AC	0.4598	1	0.4598	27.85	0.0004	
BC	0.0784	1	0.0784	4.75	0.0543	
A <sup>2</sup>	0.3557	1	0.3557	21.55	0.0009	
B <sup>2</sup>	0.1905	1	0.1905	11.54	0.0068	
C <sup>2</sup>	0.0047	1	0.0047	0.2869	0.6039	
Residual	0.1651	10	0.0165			

Lack of Fit	0.0784	5	0.0157	0.9052	0.5422	Not significant
Predicted R <sup>2</sup>	0.8859	Adjusted R <sup>2</sup>	0.7384	Adeq. Prec	13.9002	

In addition to this, the Predicted R<sup>2</sup> value was in reasonable agreement with the Adjusted R<sup>2</sup> of 0.8859; i.e. the difference was less than 0.2 proved that the model was fitting the data and can reliably be used to interpolate. The adequate Precision value 13.9002 was greater than 4 suggested that the model has a strong enough signal to noise ratio to be used for optimization for navigation of the design space.

The model equation was developed using the coefficient of estimate analysis and the model was developed using quadratic model. Therefore, the model equation for actual factors was designed as:

$$\text{strength(CN)} = -0.123874 - 0.085621X_1 - 0.074547X_3 + 0.008718X_1X_3 + 0.006288X_1^2 - 0.002044X_2^2$$

Equation 4: Model equation of actual factors for strength of CWF,

Where X1=NaOH concentration, X2=Extraction Time, X3= MLR value

### 3.1.5. Effect of NaOH, Time and MLR on strength

From the one factor analysis, NaOH concentration has quadratic effect followed by linear relationship (**Figure 6 a**). That was because as NaOH concentration increased, the strength increased before degradation point. The reason was that NaOH removed most of the impurities like lignin, pectin and other waste materials that increased the cellulose surface energy to resist applied force on the fibre.

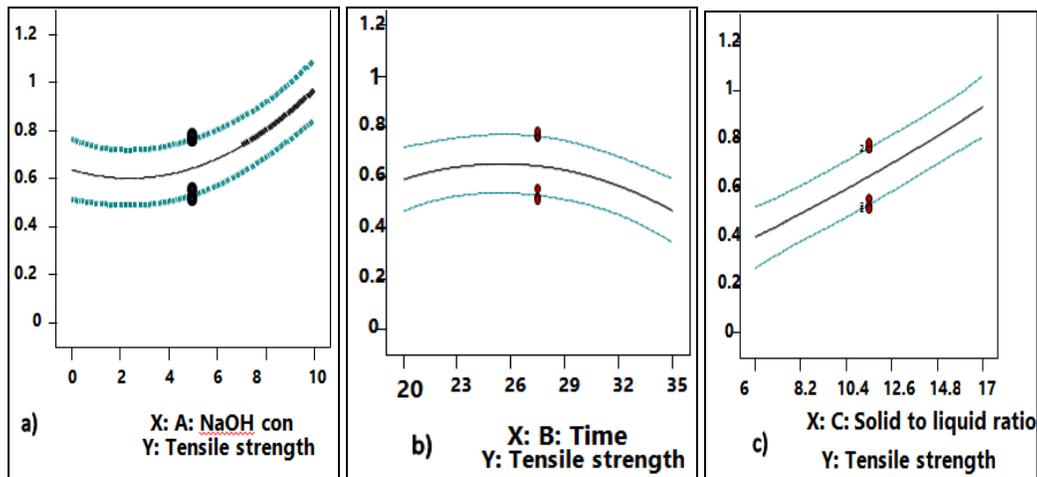


Figure 6: One factor effect analysis for fiber strength in CWE

On the contrary, as the duration of extraction increased, the strength increased to a certain level (up to 27 days, but beyond this range strength of the fibre decreased dramatically due to the microbial effect of feeding on the fibre has increased (Figure 6 b). MLR value has a linear relation with strength of fiber (Figure 6 c).

### 3.1.6. Interaction effect of factors on the fiber strength

For the extraction process with three control variables, the interaction terms significantly affect the strength of the fibre were NaOH concentration and MLR value on strength. The interaction effect of NaOH and MLR was direct relation effect that as NaOH concentration and MLR value increased, the strength of the fibre increased linearly (Figure 7).

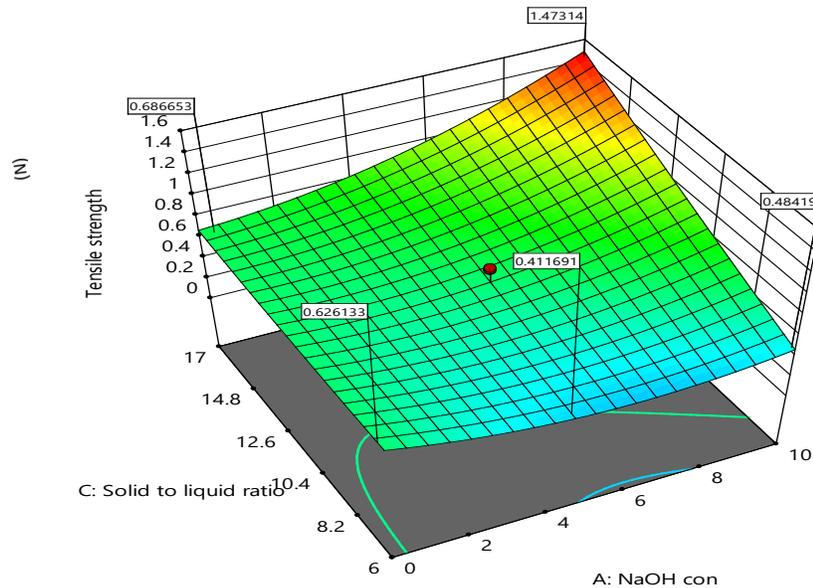


Figure 7: The interaction effect of factors on strength in CWE

From the design evaluation, ANOVA statistics, and diagnostics graphs, the models provide a good estimate of the true response surface. The ultimate goals are in the range for factors and maximize for both responses. The optimal solutions for the process was developed after considering all of the criteria applied to find the optimal settings and look through all the given solutions to see which ones best meet the specified criteria. The optimal solution has a maximized desirability value of 0.861 with the factor settings in the highest desirability scores, which indicate there is an island of acceptable outcomes. Therefore, the model is optimal process with 3.8 % NaOH concentration and 17:1 MLR value soaked for about 20 days with a maximal yield of 45% fibre having an optimized tensile strength of 90.58 CN (177.61 CN/Tex).

## 3.2. Hot Water Extraction (HWE)

### 3.2.1. Analysis of Variance for Yield

Table 6 showed that the model F-value of 3.13 implied that the model was significant. There was only a 1.80% chance that the F-value large occurred due to noise levels. Similarly, P-values less than 0.0500 indicated model terms A, D, CD, C<sup>2</sup>, D<sup>2</sup> were significant model terms and the lack of fit F-value of 0.75 suggested the lack of fit was not significant relative to the pure error. There was only 67.51% chance that a "Lack of Fit F-value" this large occurrence due to noise since non-significant lack of fit model was good for the model to fit the expected criteria.

From the fit statistics analysis, negative predicted R<sup>2</sup> inferred that the overall mean of the model was a better predictor of the response variables and adequate precision ratio of 7.063 indicated an adequate signal so that the model can be used to navigate the design space as well.

Table 4: ANOVA Table for Yield percentage in HWE

Source	Sum of Squares	Df	Mean Square	F-value	P-value	
Model	2146.62	14	153.33	3.13	0.0180	Significant
A-NaOH con	565.27	1	565.27	11.52	0.0040	
B-Time	1.02	1	1.02	0.0208	0.8874	
C-MLR value	0.1631	1	0.1631	0.0033	0.9548	
D-Temperature	463.46	1	463.46	9.45	0.0077	
AB	1.21	1	1.21	0.0247	0.8773	
AC	108.16	1	108.16	2.21	0.1583	
AD	29.74	1	29.74	0.6064	0.4483	
BC	20.25	1	20.25	0.4128	0.5302	
BD	32.96	1	32.96	0.6719	0.4252	
CD	251.67	1	251.67	5.13	0.0387	
A <sup>2</sup>	134.86	1	134.86	2.75	0.1181	
B <sup>2</sup>	29.05	1	29.05	0.5923	0.4535	
C <sup>2</sup>	341.83	1	341.83	6.97	0.0186	
D <sup>2</sup>	390.67	1	390.67	7.96	0.0129	
Lack of Fit	441.08	10	44.11	0.7484	0.6751	Not significant
Predicted R <sup>2</sup>	-0.219	Adjusted R <sup>2</sup>		0.384	Adeq. Prec	7.063

The actual model equation was designed for yield percentage; it was evident that from the VIFs values; indicated that there is no much multi-nonlinearity, shows less factors correlation. The equation can make predictions about the yield response for given levels of each factor for the design space.

The model equation of actual factors was developed:

$$Y (\text{yield } \%) = 105.15 + 1.64X_1 - 2.24X_4 + 0.048X_3X_4 - 0.115X_3^2 + 0.02X_4^2$$

Equation 5: model equation of actual factors for fiber yield in HWE

Where  $X_1$ =NaOH concentration,  $X_3$ =MLR value,  $X_4$ =Temperature

### 3.2.2. Single Factor Effect Analysis on Yield percentage

The effect of one factor on percentage yield for hot extraction method is shown in **Figure 8**. NaOH concentration and temperature of extraction have inverse relationship with fiber yield this may be due to the fact that both NaOH and temperature contribute for the complete removal of all the impurities in the fiber, finally reduce the field fiber yield.

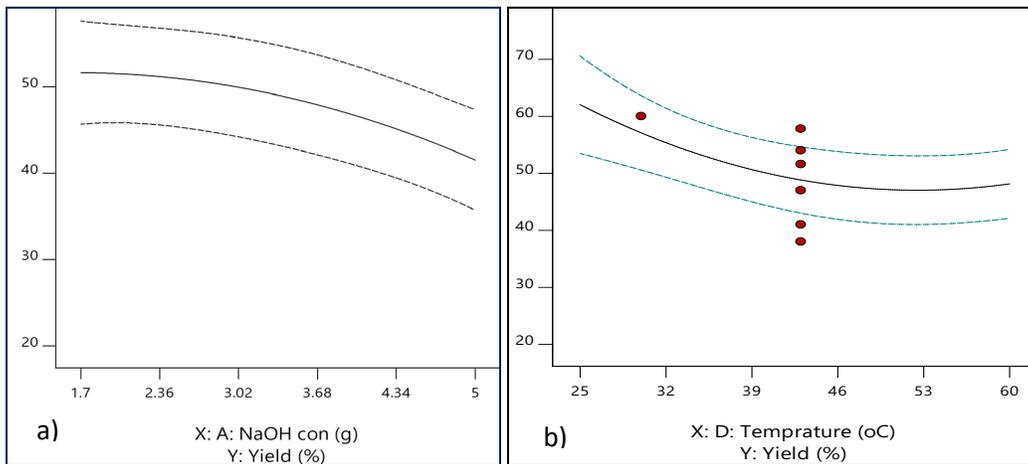


Figure 8 : Single factor analysis graph for yield in HWE

When there was an interaction between factors, the factor's effect depends upon the settings of other factors, so the one-factor plots can be very misleading when there are interactions between factors in response to two-way interactions. The interaction effect of all parameters on fiber yield was analyzed and it was shown in **Figure 9**.

For the significant terms; variables NaOH and temperature have inverse relationship with percentage yield of the fiber due to the fact that both NaOH and temperature contributed to the complete extraction and removal of all the impurities in the fiber, finally reduced the yield percentage.

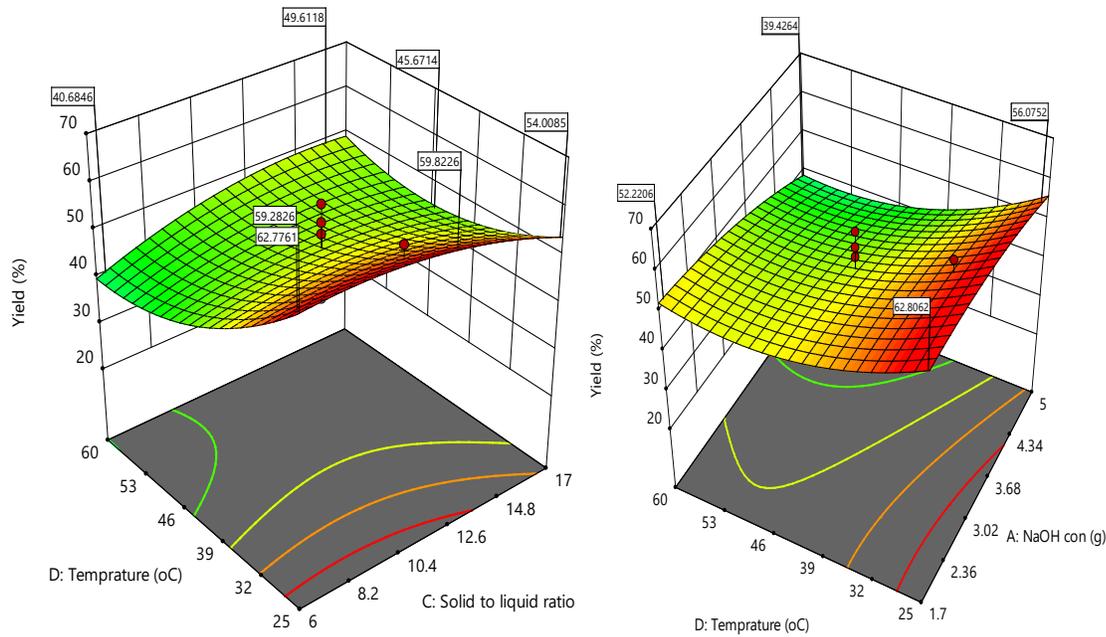


Figure 9: Interaction effect analysis graph for yield in HWE

### 3.2.2.1. Analysis of Variance for Strength

(As shown in table 7), the Model F-value of 18.20 advocated that the model was significant with 0.01% chance F-value large occurrence due to noise. A, B, C, D, AB, and AC were significant model terms since they have a p-value of 0.05. In addition, the lack of fit F-value of 4.14 supposed that there was a 6.28% chance that a lack of fit F-value large occurs due to noise. Furthermore, from the fit statistics, the difference between predicted  $R^2$  Adjusted  $R^2$  was less than 0.2 that designated an adequate signal to navigate the design space as well.

Table 5: ANOVA table for tensile strength in HWE

Source	Sum of Squares	Df	Mean Square	F-value	P-value	
Model	21849.21	10	2184.92	18.20	< 0.0001	Significant
A-NaOH con	7723.72	1	7723.72	64.34	< 0.0001	
B-Time	6723.32	1	6723.32	56.01	< 0.0001	
C-MLR	788.14	1	788.14	6.57	0.0191	
D-Temperature	1546.20	1	1546.20	12.88	0.0020	
AB	757.90	1	757.90	6.31	0.0212	
AC	3574.84	1	3574.84	29.78	< 0.0001	
AD	245.72	1	245.72	2.05	0.1687	
BC	0.0650	1	0.0650	0.0005	0.9817	
BD	65.44	1	65.44	0.5452	0.4693	

CD	478.63	1	478.63	3.99	0.0604	
Lack of Fit	2099.43	14	149.96	4.14	0.0628	Not significant
Predicted R <sup>2</sup>	0.7230		Adjusted R <sup>2</sup>	0.8557	Adeq. Pre	16.002

Then the actual model equation for the actual factors on strength was predicted using the coefficient of estimate analysis (equation 6)

$$Strength(cN) = 181.61 - 27.71X_1 + 2.104 - 57X_2 - 1.68059X_3 - 0.03X_4 - 3.34X_1X_2 + 1.65X_1X_3$$

Equation 6: Model equation for actual factors of strength of HWEF

Where, X<sub>1</sub>=NaOH concentration, X<sub>2</sub>=Time, X<sub>3</sub>=MLR, X<sub>4</sub>=Temperature

### 3.2.3. Single Factor Effect Analysis on Strength of HWEF

In this experimental method, all the four factors have a significant effect on the strength property of the extracted fiber. Except MLR, all the other three factors affect the strength of the extracted fiber negatively. Therefore, it was not possible to conclude the effect of model terms on the strength property using one factor at a time method.

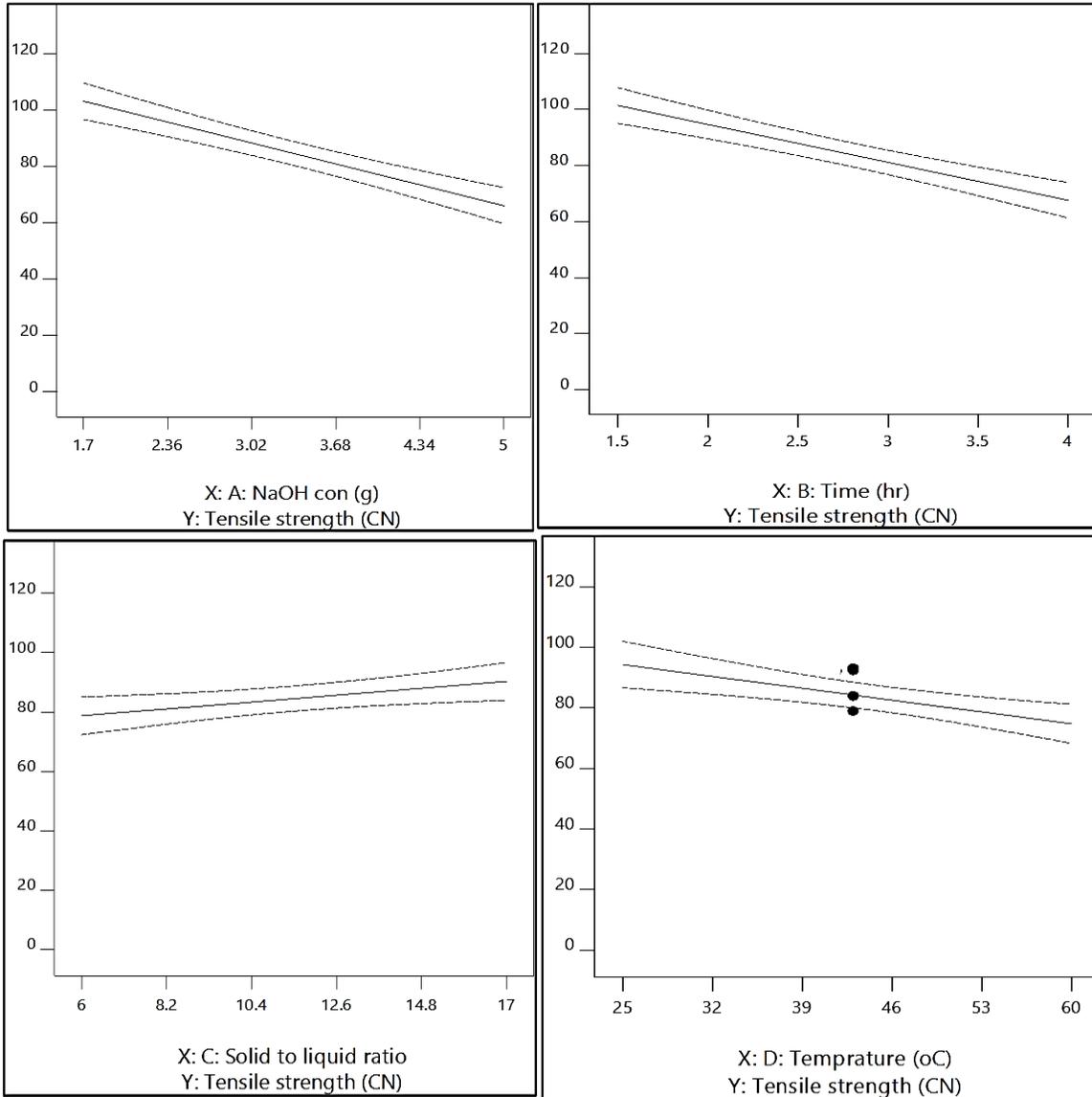


Figure 10: One factor analysis graph for strength in HWE

### 3.2.4. Interaction effect analysis of strength in HWEF

The interaction of NaOH concentration and time has a negative linear effect on the strength of the fiber. From **Figure 11**, it can be concluded that as the concentration increased the tensile strength of the extracted fiber was lowered. This was due to the fact that as time and temperature increases, the degradation rate of the fiber was increased; this in turn reduced the strength of the fiber dramatically. On the other hand, the interaction effect of NaOH and MLR was inversely related effect that as NaOH concentration and MLR value increased, the strength of the fiber decreased

linearly. Even though MLR value has positive impact on strength of the fiber, the NaOH concentration affected this scenario and their interactions reduced the strength of the fiber.

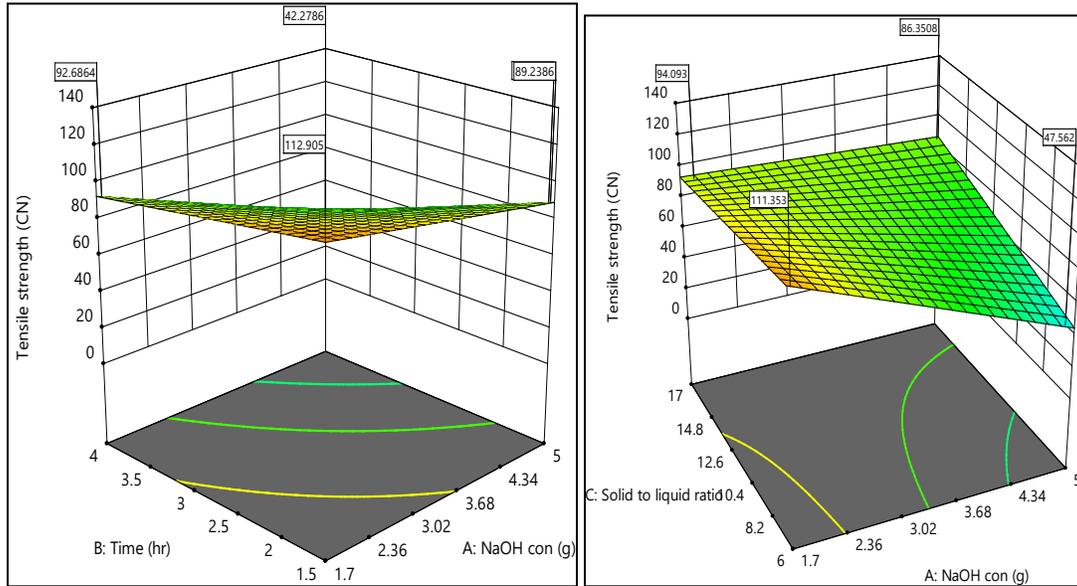


Figure 11: Interaction effect graph of factors for strength in HWE

Generally, from the design evaluation, ANOVA statistics, fit summary and diagnostics graphs, the models provided a good estimate of the true response surface. The extraction process was optimized using design expert software. Since the goals of optimization was to maximize economic benefit by minimizing processing cost, the process variables (concentration, MLR, time and temperature) need to be set within the range and the two response variables, yield and tensile strength were set to maximum levels. Therefore, the model was optimal process with 1.7% NaOH concentration and 6:1 MLR value treated at 30°C for 90 minutes with a maximal yield of 59.6% fiber having an optimized tensile strength of 247.68 cN/Tex. From the two extraction techniques, the hot water extraction was found the better optimized process in both strength (247.68 cN/Tex) and yield (59.6%) than that of cold-water extraction.

#### 4. Conclusions

The need for recyclable, renewable and sustainable materials has brought about the amplified consumption of natural fibers for various applications. This study was mainly focused on the optimization of extraction techniques of the lignocellulosic fiber derived from *Cyperus Dichrostachys A.Rich* plant. On the progress of the study, the new natural lignocellulosic fiber was successfully extracted using the well optimized extraction techniques and successfully demonstrated to be an alternative source for producing the fiber. The fiber was extracted with cold

extraction and hot extraction techniques at different NaOH concentrations. The influence of alkali treatment with correlated factors and extraction techniques on the properties of the fiber was studied well.

The elimination of lignin, hemicellulose and pectic substances from the fiber strands on hot water extraction was proved by repeated tests. The tensile property of the fiber was increased for hot extraction due to improved fiber structure at for 1.7% NaOH treatment that gave optimized strength and yield.

#### **Conflict of Interests/Competing Interests**

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**Financial interests:** The authors declare they have no financial interests.

**Non-financial interests:** none.

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