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Analysis of Predicted Kappa Number and Viscosity in Oxygen Delignification of Manihot Esculenta Crantz

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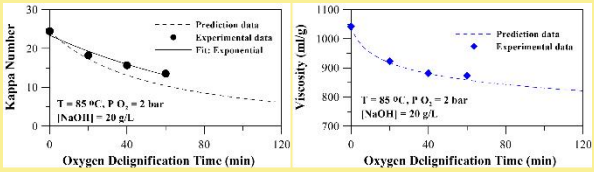
***Abstract*—** The oxygen delignification of cassava stem waste (*Manihot esculenta crantz*) kraft pulps was examined under various conditions. Two important parameters related to the quality of the resulting pulp, which is Kappa number and viscosity, are affected by certain operational conditions. Therefore, it is crucial to measure and analyze these parameters beforehand. This study aimed to predict the Kappa number and viscosity under different conditions and to identify optimal conditions for producing high-quality pulp. The prediction was made using a kinetic model of oxygen delignification reaction based on experimental data. The analysis revealed that the optimal conditions were a pressure of 2 bar, an alkali charge of 2%, a temperature of 80°C, and a reaction time of 53 minutes, resulting in a pulp viscosity of 878.73 ml/g. Additionally, the experimental data matched closely to the prediction data obtained from the model.



***Keywords*—** Kappa Number, Manihot Esculenta Crantz, Oxygen Delignification, Pulp, Viscosity

I. INTRODUCTION

As the industry of pulp and paper is growing rapidly, the demand of raw material for pulp production also increases markedly. Wood fibers are the common raw material for pulp. However, the use of non-wood fibers has increased due to the shortage of wood fibers, such as long plantation cycle. In the other hand, some non-wood fibers have shorter plantation cycle and are used in papermaking for special purpose. Some studies have reported the pulp production from non-wood fibers as raw material such as: wheat straw [1], banana pseudo stem [2], elephant grass and switchgrass [3], rapeseed straw [4], Abaca fiber [5], pineapple leave fiber [6], rice straws, corn stalks, cotton stalks, and bagasse [7], etc. The current usage of those non-wood plant fibers would perform a main role in increasing pulp and papermaking raw materials.

Cassava stem of Manihot Esculenta Crantz is one of the sources of non-wood fibers. It is a cultivable plant and resistant to extreme weather, which can be planted from the beginning of the rainy season to the dry season. After being planted for 2-7 months, this cassava will not decompose even though there is no rain for 3 consecutive months. Manihot fibers are suitable as a raw material for textiles, pulp, and paper due to their composition, which includes 20-35% cellulose, relatively low ranging between 10-14% hemicellulose, and 12-17% lignin [8].

In pulp production, the raw materials, either wood or non-wood, are cooked in pulp reactor together with active hydroxide and hydrogen sulfide ions at elevated temperature. This cooking process is known as the kraft process. The kraft process is carried out to decompose the lignin polymer chain which binds the fibers, cellulose and hemicellulose. Since kraft process undergoes in quite harsh condition, therefore, during the kraft cooking process, not only the lignin part is degraded, but cellulose and hemicellulose could also be degraded [9]. The resulting kraft-pulp is still brown therefore the resulting pulp needs to undergo the next processes, such as oxygen

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chemical bleaching and delignification (OD), to enhance the pulp's brightness. The oxygen delignification (OD) process applies pressure of oxygen and concentration of alkaline to further degrade the remaining lignin content in the unbleached pulp. After OD process, pulp results are in a low kappa number, which shows lower lignin content, affecting a reduced need for active chemicals during pulp bleaching. This decrease in lignin content reduces the requirement for chlorine dioxide and other chemicals in the bleaching process to achieve the desired brightness.

A. Oxygen Delignification of Kraft Pulp

Oxygen delignification is a process that utilizes oxygen and an alkaline solution to eliminate a significant amount of lignin remaining in the pulp after the cooking phase. It operates at elevated temperatures and pressures and can handle both high and medium pulp consistencies, either in a single stage or multiple stages. Serving as a versatile intermediary step between cooking and final bleaching, oxygen delignification is most commonly applied in medium consistency conditions in industrial settings. However, its effectiveness is limited, as excessive delignification can lead to significant cellulose degradation beyond a certain threshold. Thus, the less selectivity is a notable disadvantage of oxygen delignification.

The Kappa number is the most used index or parameter for measuring residual lignin present in pulp. A low Kappa number can negatively impact pulp strength due to the dissolution of carbohydrates, which decreases pulp yield. A high kappa number increases the bleaching chemicals cost. The standard value (SNI) of Kappa number for pulp is 18 ± 2. Considering the long process of kraft pulp extended with oxygen delignification with some important variables affecting the value of viscosity and Kappa number, a kinetic model would be very useful for forecasting the viscosity and Kappa number at different operating condition. To verify the prediction results, experimental data were also produced. In addition, the experimental data was used to determine the constants of the kinetic model of reaction. Therefore, for this paper, the Kappa number and viscosity of kraft pulped Manihot Esculenta Crantz in oxygen delignification obtained experimentally and predictably were analyzed.

B. Kinetic of Oxygen Delignification

During OD, both lignin and carbohydrate degradation occur at the same time. Consequently, the study of the kinetics of oxygen delignification involves the examination of both delignification and carbohydrate degradation kinetics. The kinetics of this process are influenced by factors such as oxygen pressure, temperature, alkali concentration, and the interaction between oxygen, alkali, and the fibers.

The degree of delignification is typically calculated by measuring the Kappa number of the pulp, that indicates the remaining lignin content. The degradation of carbohydrate is evaluated by measuring the reduction in intrinsic viscosity [η].

1) Kinetic model of Kappa number change.

The widely used kinetic model for delignification reactions is described in the following equation [10]:

$$-\frac{dK}{dt} = k[OH^-]^m[O_2]^n K^q \quad (1)$$

where (K) is the Kappa number; [OH^-] is the sodium hydroxide concentration and [O_2] is the oxygen concentration. The constant parameters m, n, and q in equation (1) are determined empirically from the experimental data. The reaction rate coefficient (k) depends on the temperature and is shown by the Arrhenius equation as follow:

$$k = Aexp\left(-\frac{E_A}{RT}\right) \quad (2)$$

where (E_A) is the activation energy, A is frequency factor, (R) is the gas constant and (T) is the absolute temperature.

2) Kinetic model of viscosity reduction.

Similar empirical power law models, as shown in equation (1), have also been proposed for viscosity reduction corresponding to the cellulose degradation.

$$-\frac{d\eta}{dt} = k[OH^-]^x[O_2]^\beta \eta^\lambda \quad (3)$$

where (η) is intrinsic viscosity; [OH^-] is the sodium hydroxide concentration and [O_2] is the oxygen concentration. The constants x, β, λ in equation (3) are determined empirically based on the experimental data. The reaction rate constant (k) is temperature-dependent and follows the Arrhenius equation as described in equation (4):

$$k = A_m exp\left(-\frac{E_m}{RT}\right) \quad (4)$$

where (E_A) is the activation energy, A_m is frequency factor, (R) is the gas constant and (T) is the absolute temperature.

II. METHOD

A. Materials

The part of cassava or Manihot Esculenta Crantz used in this research was the stem. The stems were cut and screened (SCAN-CM 40:94) for the desired size of chips. The chips were then dried to obtain 25 wt.% water content. Sodium sulfide and sodium hydroxide were obtained from Merck. The standard concentration of NaOH and Na_2S for Kraft pulping were set to 97.0 g/L and 32.4 g/L, respectively. The Manihot chips were digested in Kraft delignification process before oxygen delignification.

B. Sample Preparation

Before the oxygen delignification reaction, the Manihot chips were digested by conventional Kraft pulping process. The procedure of Kraft pulping followed the method used in our previous research [11]. The raw material of cooking process with the thickness from 3 to 5 mm were performed in rotating digester chambers, with a ratio of chips to cooking liquid is 300 g/L.

The method of oxygen delignification was referred to our previous studies [5, 11]. In order to find the kinetic model of oxygen delignification reaction, experiments were carried out at different variables of alkali charges, oxygen

pressures, and temperatures. The detailed operating condition is shown in table 2. The dried pulp used was 4 grams with consistency of 10%. Every condition or variable was run for several reaction times from 0 to 60 minutes, as detailed in table 1.

Prediction data for the Kappa number and viscosity were acquired under different operating conditions of oxygen delignification. The experimental conditions included reaction times varying from 0 to 120 minutes, pressures between 2 and 5 bars at a fixed temperature of 85°C, and NaOH concentrations of 2%. Additionally, temperatures ranged from 70 to 100°C at oxygen pressure of 2 bar and NaOH concentration of 2%, while NaOH concentrations varied from 1% to 5% at constant temperature of 85°C and oxygen pressure of 2 bar. These conditions are detailed in Table 2. The predictive data were generated using kinetic models for Kappa number and viscosity reduction, which were based on the experimental results.

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TABLE 1.
EXPERIMENT VARIABLES IN OXYGEN DELIGNIFICATION

Constant Variable	Changing Variable
Material: *Manihot esculenta crantz*	Pressures: 2, 3, and 4 bar
	Temperatures: 75, 85, 95, and 100°C
Process: Kraft pulping	Concentrations of NaOH: 1%, 2%, and 4%
	Process duration times: 0, 20, 40, and 60 min

C. Characterization and Analysis

Kappa number of pulp and pulp viscosity were determined corresponding with the procedures of TAPPI T 236 cm-85 and TAPPI T-230 om-89. Degree of Polymerization (DP) of pulp was assessed by using equation described as follows:

$$DP^{0.905} = 0.75[\eta] \quad (5)$$

The analysis of forecasted data for Kappa number and viscosity was made by observing the reduction of Kappa number and viscosity at different reaction conditions. The forecasted data were correlated with the experimental data to determine the deviation between the two sets. The optimal operating conditions were then selected based on the standard values of Kappa number and viscosity for qualified pulp. A reduction in Kappa number implies the level of lignin removal from the fibers or wood, while a decrease in viscosity demonstrates the extent of carbohydrate degradation and provides an indication of the pulp yield achieved after the delignification process.

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III. RESULTS AND DISCUSSION

The main control variable in a Kraft pulp process is the Kappa number, which represents the amount of remaining lignin content in the pulp. Kappa number reveals the degree of delignification. Low Kappa number shows high lignin removal, leading to higher degree of delignification. Carbohydrate degradation is observed by quantifying the decrease in intrinsic viscosity [η], which represents to the degree of polymerization. The significant reduction in intrinsic viscosity shows a higher degree of cellulose degradation.

A. Analysis of Predicted Kappa Number

Experimental data were used to determine kinetic model parameters (q, m, n, EA, and A) for the oxygen delignification reaction. These parameters were subsequently incorporated into Equations (1) and (2) to develop a kinetic model describing Kappa number reduction.

TABLE 2.
VARIABLES FOR PREDICTION DATA IN OXYGEN DELIGNIFICATION

Parameters	Conditions
Pressures (bar)	2, 3, 4, 5
Temperatures (°C)	70, 75, 80, 85, 90, 95, 100
NaOH Concentrations (%)	1, 2, 3, 4, 5
Heating times (min)	0, 20, 40, 60, 80, 100, 120

Kinetic of oxygen delignification is influenced by several factors such as temperature, hydroxy ion concentration, and oxygen pressure, as shown in equation (1). A constant alkali (sodium hydroxide) concentration and oxygen solubility were assumed to constant during oxygen delignification process thus the reduction rate of Kappa number can be expressed as common kinetic model, as displayed in the equation (6) [12]:

$$-\frac{dK}{dt} = k_q K^q \quad (6)$$

The reaction rate constant (k_q) was correlated to the alkali concentration [OH^-, g/L], temperature (T, K), activation energy (E_A, kJ/mole) and oxygen solubility (O_2, g/L), as depicted in equation (7) [13].

$$k_q = A_q exp\left(-\frac{E_A}{RT}\right)(OH^-)^m(O_2)^n \quad (7)$$

By integrating equation (6) under constant oxygen pressure and initial alkali concentration, the value of reaction rate order (q) can be estimated. The q, reaction rate order, was selected based on the maximum value of the obtained correlation coefficient (R^2) from a plot of Kappa number versus time. The other parameters were determined by a similar procedure as method of obtaining reaction rate order (q). The obtained q value was 1.7. Therefore, the kinetic model equation of Kappa number reduction for *Manihot esculenta crantz* chips can be displayed below:

$$-\frac{dK}{dt} = 148.41 exp\left[\frac{-34.24}{RT}\right][OH^-]^{0.839}[O_2]^{0.725}K^{1.7} \quad (8)$$

This equation was then used for obtaining the predicted Kappa number data. The change of Kappa number was observed at different operating conditions of oxygen delignification.

Figure 1 illustrates the reduction in Kappa number of Manihot esculenta crantz chips as a function of reaction time under varying NaOH concentrations, either for prediction or experimental data. The same trend was found

for the condition of various oxygen pressures and temperatures, as displayed in Figure 2 and 3, respectively. These figures indicate that the predicted data of Kappa number closely match the experimental Kappa number (dotted). It is also observed that the Kappa number decreases as NaOH concentration, oxygen pressure, and temperature increase. Under the highest conditions of temperature, oxygen pressure, and alkali concentration, the Kappa number reaches its lowest value. Low Kappa number means high lignin removal. Some lignin removal is expected after oxygen delignification; however, the lowest value of Kappa number does not represent the best condition.

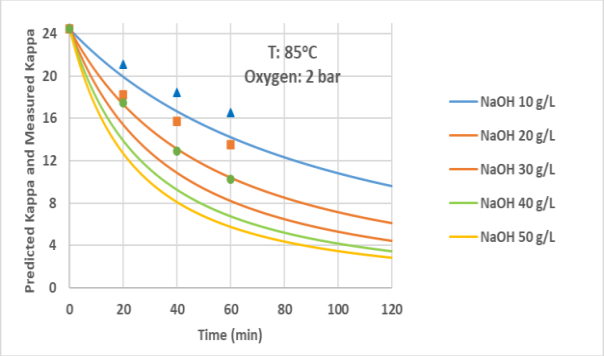

Figure 1. The prediction and experimental data of Kappa number versus oxygen delignification time of *Manihot esculenta Crantz* that were obtained under various NaOH concentrations, with a constant temperature and oxygen pressure.

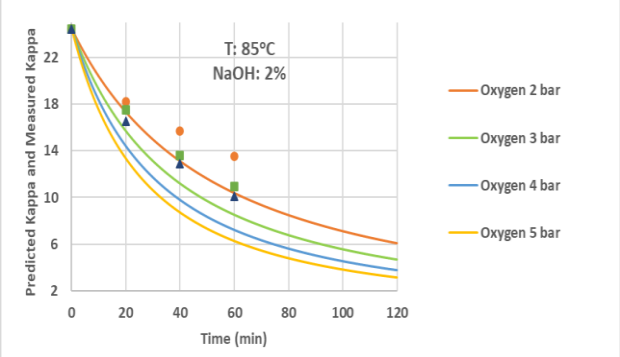

Figure 2. The prediction and experimental data of Kappa number versus oxygen delignification time of *Manihot esculenta Crantz* that were obtained under various oxygen pressures, with fixed temperature and NaOH concentration.

The reduction in the Kappa number is linked to a decrease in viscosity, which signifies carbohydrate degradation. This degradation, in turn, affects both the yield and the strength of the pulp. Low Kappa number shows the decrease of intrinsic viscosity which represents high degree of carbohydrate degradation, leading to the low yield and strength of the pulp. Therefore, the optimum condition was chosen according to the standard value of Kappa number and intrinsic viscosity for the qualified pulp. Table 3 shows the R-square value between the prediction and experimental data for Kappa number at various variables. The R-square value for all variables is close to 1 showing that the experimental data align closely with the predicted data. Therefore, the kinetic model can be used to predict the Kappa number at other variables.

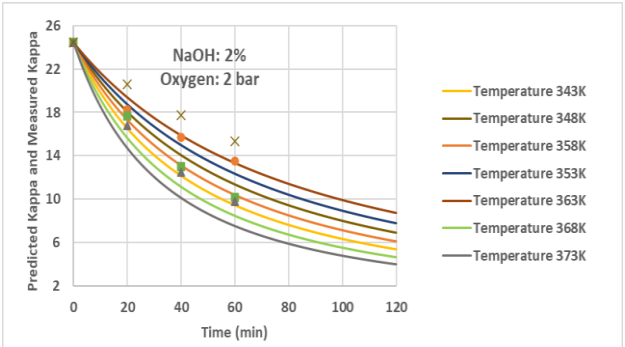

Figure 3. The prediction and experimental data of the Kappa number versus oxygen delignification time for *Manihot esculenta Crantz* that were obtained under various temperatures, with a constant oxygen pressure and NaOH concentration.

TABLE 3.
THE R-SQUARE VALUE BETWEEN THE PREDICTION AND EXPERIMENTAL DATA FOR KAPPA NUMBER

[NaOH] (g/L)	R^2	O_2 Pressure (Bar)	R^2	Temp. (K)	R^2
10	0.9996	2	0.9945	348	0.9930
20	0.9945	3	0.9983	358	0.9945
40	0.9876	4	0.9976	368	0.9935
				373	0.9963

B. Analysis of Predicted Viscosity Degradation

Lignin degradation occurs during oxygen delignification. At the same time, carbohydrate degradation also occurs. Therefore, besides the kinetic of delignification, the kinetic of carbohydrate degradation should be included in a kinetic study of oxygen delignification. Since carbohydrate degradation is represented by viscosity reduction, thus the kinetic equation of viscosity reduction was developed to describe carbohydrate degradation. Several parameters, including λ, x, β, E_m, and A_m, as displayed in equation (3) and (4) should be determined to get the kinetic model of viscosity degradation. The general equations describing the rate of carbohydrate degradation in terms of viscosity are presented in equations (9) and (10) [14].

$$-\frac{d\eta}{dt} = k_\lambda \eta^\lambda \tag{9}$$

and

$$k_\lambda = A_\lambda exp\left(-\frac{E_A}{RT}\right)(OH^-)^x(O_2)^\beta \tag{10}$$

where, η is intrinsic viscosity (ml/g), $[OH^-]$ is NaOH concentration (g/L), $[O_2]$ is oxygen concentration (g/L), and t is time (min).

By integrating equation (9) at constant oxygen partial pressure and initial alkali concentration, the reaction rate order (λ) can be estimated. The λ, reaction rate order, was selected based on the maximum value of correlation coefficient (R^2) obtained from a plot of intrinsic viscosity as function of time. The other parameters were determined by similar procedure as method of obtaining reaction rate

order (λ). The obtained λ value was 16. The viscosity degradation kinetic model for Manihot esculenta crantz chips is shown below:

$$-\frac{d\eta}{dt} = 2.467x10^{-40} exp\left[\frac{-54.29}{RT}\right][OH^-]^{0.3556}[O_2]^{1.087}\eta^{16} \quad (11)$$

This equation was then used for determining the predicted viscosity. The change of viscosity was observed at different operating condition of oxygen delignification.

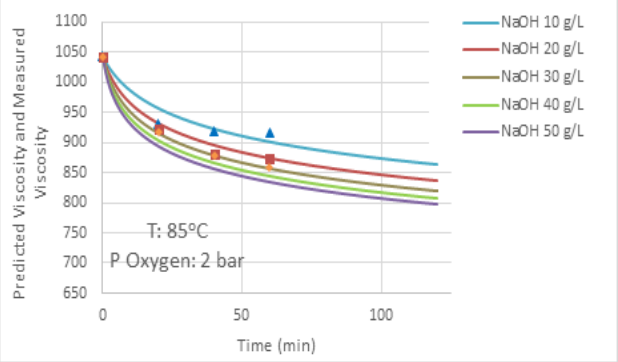

Figure 4. The prediction and experimental data of viscosity versus oxygen delignification time of *Manihot esculenta crantz* under various concentrations of NaOH at fixed temperature and oxygen pressure.

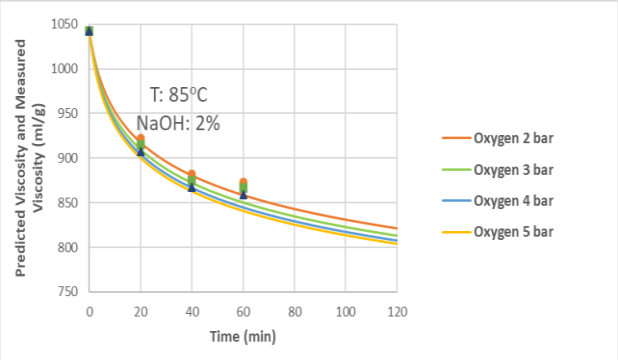

Figure 5. The prediction and experimental data of viscosity versus oxygen delignification time of *Manihot esculenta crantz* under various oxygen pressures at fixed temperature and NaOH concentration.

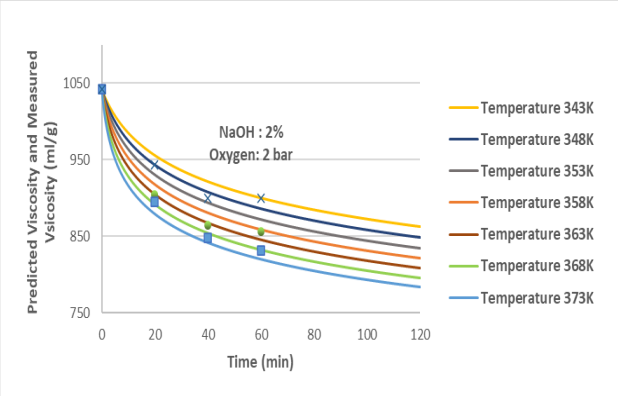

Figure 6. The prediction and experimental data of viscosity versus oxygen delignification time of *Manihot esculenta crantz* under various temperatures at fixed oxygen pressure and NaOH concentration.

Figure 4 shows the decrease in intrinsic viscosity with reaction time of *Manihot esculenta crantz* chips at various NaOH concentrations, either for prediction or experimental data. The same trend was found for condition of various oxygen pressures and temperatures, as displayed in figure 5 and figure 6, respectively. These figures also show that all the prediction data of intrinsic viscosity are close enough to the value of experimental viscosity (dotted). Intrinsic viscosity also decreased with increasing NaOH concentration, oxygen pressure, and temperature. At the highest condition of oxygen pressure, temperature, and alkali concentration, the viscosity reached the lowest value.

The drop in viscosity represents carbohydrate degradation which affects the yield and physical properties such strength of the pulp. The intrinsic viscosity of the resulting pulp should be above the standard viscosity value of the good pulp. Table 4 shows the R-square value between the prediction and experimental data for viscosity at various variables. The R-square value for all variables is close to 1 showing that the experimental data are suited well to the prediction data. Therefore, the kinetic model can be used to predict the viscosity at other variables.

TABLE 4.
THE R-SQUARE VALUE BETWEEN THE PREDICTION AND EXPERIMENTAL DATA FOR VISCOSITY

[NaOH] (g/L)	R^2	O_2 Pressure (Bar)	R^2	Temp. (K)	R^2
10	0.9959	2	0.9960	348	0.9837
20	0.9960	3	0.9975	358	0.9960
40	0.9960	4	0.9375	368	0.9971
				373	0.9971

C. *Degree of Polymerization*

The intrinsic viscosity [η] of the pulp can be employed to calculate approximately the degree of polymerization (DP) of the cellulose in pulp [15]. The longer reaction time will produce the lower viscosity of pulp. The bond between fibers in cellulose is degraded by the liquor which results in lowering fiber strength and increasing pulp solubility. In oxygen delignification reaction, lignin removal and carbohydrate degradation arise simultaneously. Thus, making a forecast is to predict Kappa number and viscosity is useful because we do not need to try all the operating condition experimentally. Equation (5) from reference [16] establishes the correlation between cellulose degree of polymerization within the pulp and its intrinsic viscosity.

The delignification reaction should be terminated when the delignification has achieved 50%. Table 5 presents the time required to achieve 50% lignin reduction and the corresponding viscosity for different NaOH concentrations, with a constant oxygen pressure of 2 bar and a temperature of 85°C. Table 6 provides the same data for varying oxygen pressures, while maintaining a constant alkali concentration of 2% and temperature at 85°C. Table 7 shows the time and viscosity values at 50% lignin reduction for various temperatures, with the alkali

concentration held at 2% and oxygen pressure at 2 bar. Carbohydrate degradation occurs when the lignin content decreases more than 50% [13]. Medium consistency pulp typically requires approximately 50 to 60 minutes for delignification [13]. The minimum viscosity value for pulp, according to standard processing guidelines, is 846 ml/g [17,18]. Based on this standard value and the data presented in Tables 5, 6, and 7, the optimal operating conditions for oxygen delignification of *Manihot esculenta* Crantz are a temperature of 80°C, an oxygen pressure of 2 bar, and a NaOH concentration of 2%. Under these conditions, a reaction time of 53 minutes results in a viscosity of 878.73 ml/g.

TABLE 5.
REACTION TIME AND VISCOSITY AT 50% LIGNIN REDUCTION FOR VARIOUS CONCENTRATIONS OF NAOH

Alkali Charge (%)	**1**	**2**	**3**	**4**	**5**
Reaction time (min)	81.3	45.7	32.6	25.7	21.3
Viscosity (ml/g)	871.09	873.19	874.47	875.274	876.08

TABLE 6.
REACTION TIME AND VISCOSITY AT 50% LIGNIN REDUCTION FOR VARIOUS OXYGEN PRESSURE

Oxygen Pressure (bar)	**2**	**3**	**4**	**5**
Reaction time (min)	45.7	34.1	27.7	23.6
Viscosity (ml/g)	873.19	881.23	886.92	891.27

TABLE 7.
REACTION TIME AND VISCOSITY AT 50% LIGNIN REDUCTION FOR VARIOUS TEMPERATURES

Temperature (°C)	**70**	**75**	**80**	**85**	**90**	**95**	**100**
Reaction time (min)	70.9	61.4	53	45.7	39.5	34	29.1
Viscosity (ml/g)	890.82	884.57	878.73	873.19	867.76	862.78	858.25

IV. CONCLUSION

The analysis of Kappa number and viscosity after oxygen delignification of Manihot esculenta crantz chips was completed, and the optimal operating conditions for an efficient and effective process. A reduction in Kappa number signifies a decrease in lignin content, while a decline in viscosity indicates carbohydrate degradation. The predicted Kappa number and viscosity values closely aligned with the experimental data. The optimal conditions for oxygen delignification of Manihot esculenta crantz were achieved at a pressure of 2 bar, a temperature of 80°C, and 2% NaOH for 53 minutes, resulting in a viscosity of 878.73 ml/g. These prediction results provide valuable guidance for the experimental implementation of oxygen delignification, especially in selecting process conditions.

V. ACKNOWLEDEGMENT

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