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Improving Reaction Selectivity with NaOH Charges and Reaction Time in the Medium Consistency Oxygen Delignification Process

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Abstract. As more delignification is targeted, fiber degradation becomes a main issue in the pulping process. Sodium hydroxide (NaOH) is highly related to pulp quality in the medium consistency oxygen delignification process. Accordingly, the purpose of this research was to study the effect of NaOH charges and reaction time on reaction selectivity during the pulping process through medium consistency oxygen delignification. This research used *Eucalyptus pelita* and *Acacia mangium* pulp with a kappa number (KaNo) of 17–18. The medium consistency oxygen delignification process condition included a temperature of 80°C and oxygen pressure of 1 bar, while the reaction times were 20, 40, 60, 80, and 100 min. The NaOH charges were 5, 10, 15, 20, and 25 kg/t of pulp. The analysis parameters used were KaNo and viscosity. The variation in reaction time did not show a significant change in KaNo. The increase in reaction time and NaOH charge variations, meanwhile, can reduce pulp viscosity. Higher NaOH values significantly increased the delignification degree, and the higher NaOH charges and reaction times together lowered the degree of polymerization (DP). The variation in reaction time indicated that with a longer reaction time, the lower the DP, and with a higher NaOH charge and longer reaction time, the lower the reaction selectivity.

Keywords: Delignification degree, Fiber degradation, Medium consistency oxygen, Polymerization degree, Reaction selectivity

1. Introduction

The degradation of fiber and dissolving of lignin can be predominantly found in the intermediate cooking and bleaching process. The medium consistency oxygen delignification system as intermediate process completed by using chemical treatments, including acidic and basic treatments (Hermansyah et al., 2019). The alkaline treatment is an efficient method for delignification (Harahap et al., 2019), especially using sodium hydroxide (NaOH) to influence the physical properties of the fibers. This treatment removes the hemicellulose and lignin contained in the fiber (Fatra et al., 2016). This process is also in part considered a continuation of the pulping alkaline process and, somehow, the first step in the bleaching process. The oxygen delignification in the medium consistency oxygen stage decreases the kappa number (KaNo) prior to chlorination and provides the bleaching plant with a pulp that has a considerably reduced KaNo (Bajpai, 2012). This process also removes part of the residual lignins from kraft cooking through the reaction of pulp with oxygen and NaOH under high temperatures condition (Júnior and Gomes, 2018).

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Oxygen delignification helps in part by substituting both chlorine and chlorine dioxide during bleaching and has additional technical and economic benefits (Akim et al., 2011). Carbohydrate degradation occurs in particular during the initial stage and continues toward the end of kraft cooking. The oxygen-alkali process (i.e., oxygen delignification) is known to be more selective (i.e., carbohydrate yield/delignification) than the final kraft cooking phase (Jafari et al., 2014a; Jafari et al., 2014b). The equipment used for medium consistency oxygen delignification consists of a steam medium pressure injection system, centrifugal pump, pressurized reactor, distribution reactor, and gas mixer with high turbulence (Hart and Rudie, 2012). The medium consistency oxygen with single reactor system can improve delignification selectivity and depolymerization as seen from change the KaNo and viscosity of the pulp produced. The variables involved in medium consistency oxygen delignification are reactor pressure, temperature process, pH, reaction time, and pulp consistency. The variation in this process's conditions is shown in Table 1 (Júnior and Gomes, 2018).

Table 1 Typical conditions in industrial medium consistency oxygen delignification

Operational Conditions	High Consistency (%)	Average Consistency (%)
Pulp consistency, %	25–34	10–14
Reaction time, min	30–45	50–60
Reaction temperature, °C	115–400	70–105
Alkaline charge, $kg.t^{-1}$	18–23	18–28
Oxygen load, $kg.t^{-1}$	15–24	20–24

The addition of oxygen gas to the pulp in an oxygen mixer produce maximum contact between the pulp and oxygen (Nasser, 2015). The results of this process in turn reduce the KaNo values, thus decreasing bleaching chemical consumption (Markus and Pearce, 2017). KaNo is the volume of 0.1 N potassium permanganate solution consumed by 1 g of moisture-free pulp under the acidic conditions, with the result corrected to 50% consumption of the added permanganate (TAPPI, 2006). The initial KaNo value influences the level of delignification in the same NaOH charge. The KaNo target for the cooking process are 25 to 30, and further delignification using the alkaline oxygen process recommends a KaNo of 15–20 (Jafari et al., 2014a).

The delignification in the medium consistency oxygen stage reflects on KaNo reduction. The classification and species of wood also affect delignification in the medium consistency oxygen delignification process (Hart and Rudie, 2012). The reduction in KaNo can reach up to 75% for softwood species and 45–50% for hardwoods.

Medium consistency oxygen delignification is flexible and best done between the cooking and bleaching processes (Rahmati et al., 2010). Using medium consistency oxygen delignification before bleaching is meant to reduce the amount of chlorinated organic compounds in the bleaching plant's effluent (Jablonsky et al., 2018).

The decrease in lignin content during medium consistency oxygen delignification is represented by the degree of delignification. The degree of delignification is the percentage decrease in lignin content before and after the medium consistency oxygen process. Lignin content is calculated based on KaNo, where L (%) = 0.147×KaNo (Violette, 2003), while the formula delignification degree (%) is (Wistara et al., 2015):

$$\mathrm{DD}\ (\%) = \frac{\mathrm{Lo}(\%) - \mathrm{Lt}(\%)}{\mathrm{Lo}(\%)} \times 100\% \tag{1}$$

where DD is the degree of delignification, Lo(%) is the lignin content before medium consistency oxygen delignification, and Lt(%) is the lignin content after medium consistency oxygen delignification.

The process of fiber degradation in NaOH media is initially characterized by the swelling of the cell wall. The reaction between oxygen and degradation products in the medium consistency oxygen delignification process reduces the amount and effectiveness of oxygen for the delignification reaction during the oxygen delignification (ODL) process (Zhao et al., 2018). Fiber degradation can be determined from the degree of polymerization (DP). DP is calculated from intrinsic viscosity using the relationship proposed by Inmergut, Shurtz, and Mark (Henrique et al., 2015):

$$DP^{0.905} = 0.75\ [\eta] \tag{2}$$

where the values 0.905 and 0.75 are constants characteristic of the polymer-solvent system and [η] is intrinsic viscosity ($mL.g^{-1}$); (Henrique et al., 2015).

DP indicates the level of cellulosic degradation during the cooking process. The higher the DP, the stronger the cellulose (fiber) in cellulose degradation events. The solubility of cellulose in soda also decreases as DP increases (Yamane et al., 2015). DP is calculated from the intrinsic viscosity [η] of the pulp in $mL\ g^{-1}$ and the weight fractions of hemicelluloses (H) and cellulose (G) in the pulp according to Equation 3 (Jafari et al., 2014b):

$$DP = \left[\frac{(1.65[n]-116H)^{1.111}}{G}\right] \tag{3}$$

The selectivity of the medium consistency oxygen delignification stage improved by offering a greater effect on KaNo reduction, but induced more serious cellulose degradation (Chong et al., 2013). Reaction selectivity is commonly calculated as the ratio of the change in KaNo to the change in pulp viscosity during medium consistency oxygen delignification. The reaction selectivity coefficient (α) is defined as the ratio between the change in KaNo (ΔK) and (1/DPt–1/DP0) during the delignification process as long as the medium consistency oxygen process. The (1/DPt–1/DP0) value represents the number of chain scissions per polymer cellulose unit. Reaction selectivity is defined as the reduction in KaNo divided by the number of cellulose chain scissions (1/DPt–1/DP0) (Ji, 2007):

$$\text{Reaction selectivity} = \frac{(\Delta K)}{(1/DPt-1/DP0)} \tag{4}$$

2. Methods

2.1. Materials

This research featured *Eucalyptus pelita* and *Acacia mangium* pulp samples with standard KaNo 17–18 (TAPPI, 2006). In the evaluations, pulp samples of 4 g OD (oven dry) were used. The chemicals used were lab-grade NaOH and pure oxygen gas.

2.2. Instrumentation

The equipment used for this research was a water bath with a temperature setting of 80°C, six reactor tubes with a capacity of 270 cm^3 and a valve for oxygen filling, and two rotating chambers in which to place the reactor tubes.

2.3. Procedure

The samples utilized in this research were of *Eucalyptus pelita* and *Acacia mangium* unbleached pulp with KaNo 17–18. Six reactor tubes with a 270 cm^3 capacity were used to react as much as 4 g of pulp in OD conditions.

Figure 1 (a) Test tube for the medium consistency oxygen process with a capacity of 270 cm^3; (b) Position of a pulp sample in a reaction tube; (c) Valve injecting oxygen into a test tube; (d) Position of a test tube in the rotary chamber; (e) Test tube rotation system sitting in a water bath; (f) Temperature display

The NaOH charges were 5–25 kg/t at intervals of 5 kg/t based on OD pulp sample weight. The variation in reaction time was 20−100 min at 20-min intervals. The reactor temperature during the process was 80°C, with an oxygen pressure of 1 bar. The mixing of material consistency was 10%. The pulp and NaOH stirred until evenly and tightly closed in a reactor tube. After the reactor tube was closed, oxygen gas was injected into the tube until a pressure of 1 bar was reached. Each reactor tube was then placed into the rotator at a temperature of 80°C and the rotating motor turned on. The tool's condition was controlled for temperature and reaction time until the intended reaction time was completed in accordance with the variations. The delignification process occurs due to the addition of NaOH and oxygen with the heating condition in the Medium consistency Oxygen (MCO_2) process. After the MCO_2 process completed, the reactor tube is removed from the heater and opened to transfer the pulp sample to a plastic glass, then the pulp sample is washed with 2,000 mL water. The samples were strained with Whatman filter paper No. 41 using a vacuum. Then, the pulp was ready to be analyzed for KaNo and viscosity. The KaNo parameter analysis used the TAPPI T 236 CM-94 method (TAPPI, 2006) and the viscosity parameter the TAPPI T 230 method (TAPPI, 1999).

3. Results and Discussion

The analysis parameters of the pulp quality produced after medium consistency oxygen delignification were KaNo and viscosity. The KaNo values indicated the delignification process in the pulp and the degree of delignification. The lower the KaNo value, the higher the solubility of the lignin in the medium consistency oxygen delignification process. The higher the solubility of the lignin, the higher the degree of delignification in the medium consistency oxygen delignification process.

Viscosity indicated the value of cellulose degradation or DP, where the lower the viscosity, the higher the cellulose degradation. The results of the pulp analysis as based on NaOH charge and reaction time variations in medium consistency oxygen delignification are featured in Table 2.

Table 2 Results of the pulp quality analysis in the medium consistency oxygen delignification process.

Test No.	Time (min)	NaOH Charge (kg/t)	*Eucalyptus pelita*		*Acacia mangium*	
			KaNo	Viscosity (cm^3/g)	KaNo	Viscosity (cm^3/g)
1	Pre–medium consistency oxygen delignification	-	17.69	679	18.00	808
2	20 min	5	12.54	670	11.88	785
3		10	12.05	667	11.87	768
4		15	11.13	659	11.19	727
5		20	11.04	654	11.10	720
6		25	10.90	652	11.02	709
7	40 min	5	12.19	668	11.78	771
8		10	11.60	664	11.52	754
9		15	10.83	636	10.84	712
10		20	10.70	624	10.70	699
11		25	10.46	620	10.47	683
12	60 min	5	11.62	657	11.66	757
13		10	11.36	653	11.36	717
14		15	10.68	599	10.68	657
15		20	10.27	569	10.27	624
16		25	10.20	565	10.20	613
42	80 min	5	11.46	637	11.56	741
43		10	11.13	634	11.33	694
44		15	10.53	571	10.51	586
45		20	10.04	535	10.04	549
46		25	10.03	532	9.95	493
47	100 min	5	11.20	630	11.48	734
48		10	11.00	627	11.22	667
49		15	10.24	568	10.42	573
50		20	9.84	509	9.84	525
51		25	9.90	506	9.80	460

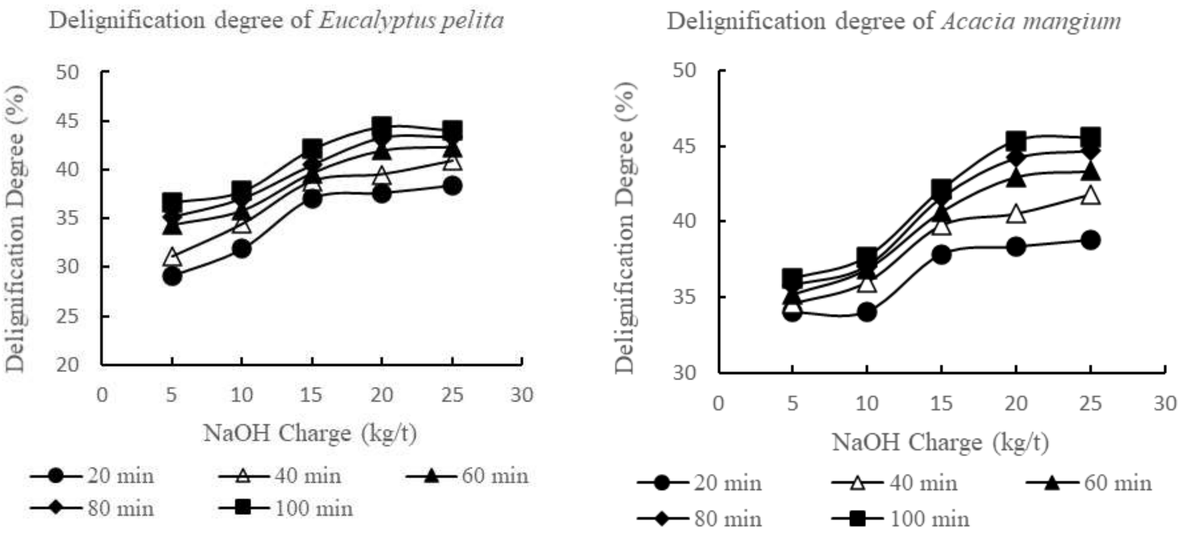
As demonstrated in Table 2, the KaNo values significantly decreased in the MCO$_2$ process. The variation in reaction time did not show a significant change in KaNo but more lignin content dissolves with an increase in NaOH charge. Meanwhile, in the NaOH charge variation, there was a tendency for the higher NaOH charges to decrease KaNo. Increasing the reaction time and NaOH charge also reduced pulp viscosity, indicating that the cellulose chain-breaking is strongly influenced by (high) NaOH charge and reaction time.

Based on KaNo and viscosity, several other parameters, namely the delignification and polymerization degrees, could be determined. Reaction selectivity was calculated based on the difference in KaNo (dK) and DP (1/DPt–1/DP0) in each treatment (Violette, 2003). The reaction selectivity value in the treatment indicated the effectiveness of the medium consistency oxygen delignification process.

3.1. The Effect of NaOH Charges on the Degree of Delignification

Delignification consists of three stages: initial delignification, bulk delignification, and residual delignification. The initial delignification stage occurs in the impregnation process, bulk delignification occurs in the cooking process at temperatures above 140°C (Tunc, 2003), and residual delignification occurs in the medium consistency oxygen delignification process.

The delignification degrees of the *Eucalyptus pelita* and *Acacia mangium* samples were calculated based on KaNo and viscosity in the medium consistency oxygen delignification process. The NaOH charges effect on the delignification degree values is shown in Figures 2a and 2b.



(a) (b)

Figure 2 The effect of NaOH charges and reaction times on delignification degree per each sample

Figures 2a and 2b show that the higher the NaOH charge in medium consistency oxygen delignification, the higher the delignification degree. This indicates that the lignin dissolved increased due to an increase in NaOH during the delignification process. Reaction time also had a significant impact on the degree of delignification, where the longer the reaction time, the higher the degree of delignification.

In *Eucalyptus pelita*, the highest degree of delignification was 5 kg/t, and at the reaction times of 20, 40, 60, 80 and 100 min, the delignification degree increased 2.3%, 2.5%, 2.0%, 2.0%, and 1.8%, respectively.

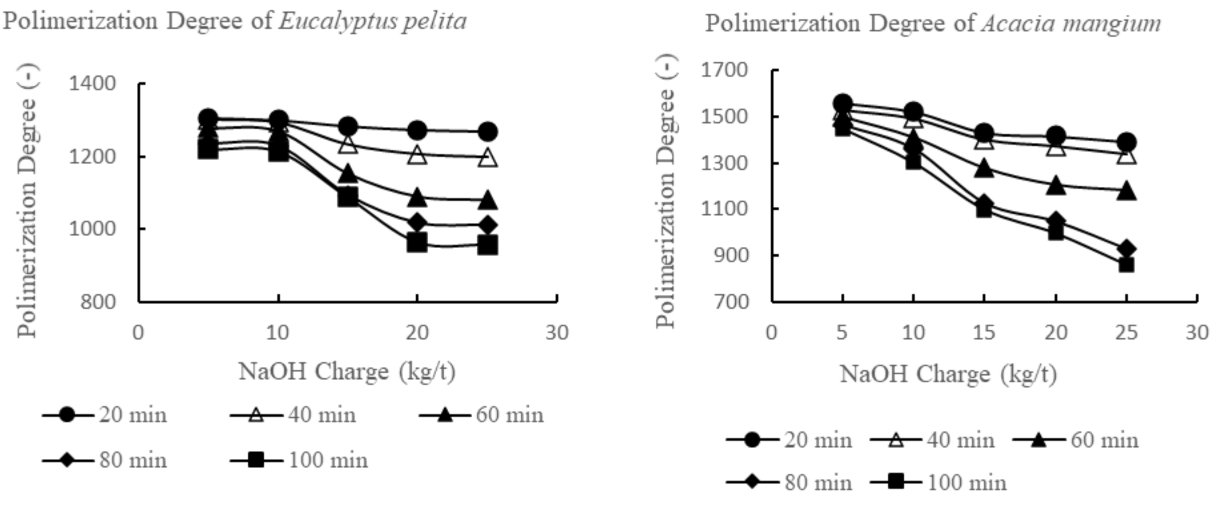
In *Acacia mangium*, the highest degree of delignification varied between 36.2–45.6% and was achieved at a reaction time of 100 min and NaOH charges of 5, 10, 15, 20, and 25 kg/t. A reaction time of 20 min produced the lowest delignification level at 34.0–38.8%. Per every 5 kg/t increase in NaOH charge and reaction times of 20, 40, 60, 80, and 100 min, the degree of delignification increased 1.2%, 1.8%, 2.0%, 2.2%, and 2.3%, respectively.

The variations in NaOH charge and reaction time show that the increase in degree of delignification was below 5%, identifying that the residual delignification stage occurs very slowly (Tunc, 2003). This is due to on the residual delignification stage was formed new lignin-carbohydrate bonds, which are stable in kraft pulping process (Septia et al., 2018).

3.2. *The Effect of NaOH Charges on Polymerization Degree*

DP indicates the degradation of cellulose. Cellulose degradation is also influenced by hydroxide ion concentration as a result of the primary degradation mechanism (Wetterling, 2012). During cellulose degradation using alkali (e.g., NaOH), the final group of cellulose is attacked by the alkali and converted to the alkali-stable m-saccharinic end group, and no further degradation takes place (Tunc, 2003).

DP is closely related to intrinsic pulp viscosity value: the higher the DP, the longer the carbon chain length contained in the cellulose. This shows a more resistant degradation event of cellulose, while the remaining carbon chain length is indicated by the DP value. The DP of *Eucalyptus pelita* and *Acacia mangium* in the medium consistency oxygen delignification process with variations in NaOH charge and reaction time can be seen in Figures 3a and 3b.



(a) (b)

Figure 3 The effect of NaOH charges on DP (-) per sample

Figures 3a and 3b show that the higher the NaOH charge and reaction time, the lower the DP for both *Eucalyptus pelita* and *Acacia mangium*. The longer the reaction time at variations of 20 min, the greater the reduction rate in DP.

For the *Eucalyptus pelita* samples, the highest DP values were 1268–1307, achieved at a reaction time of 20 min and NaOH charges of 5, 10, 15, 20, and 25 kg/t. In contrast, a reaction time of 100 min produced the lowest DP value of 957–1221. For every 5 kg/t increase in NaOH charge at reaction times of 20, 40, 60, 80, and 100 min, the decrease in DP was 9.7, 25.8, 49.6, 55.9, and 65.8, respectively.

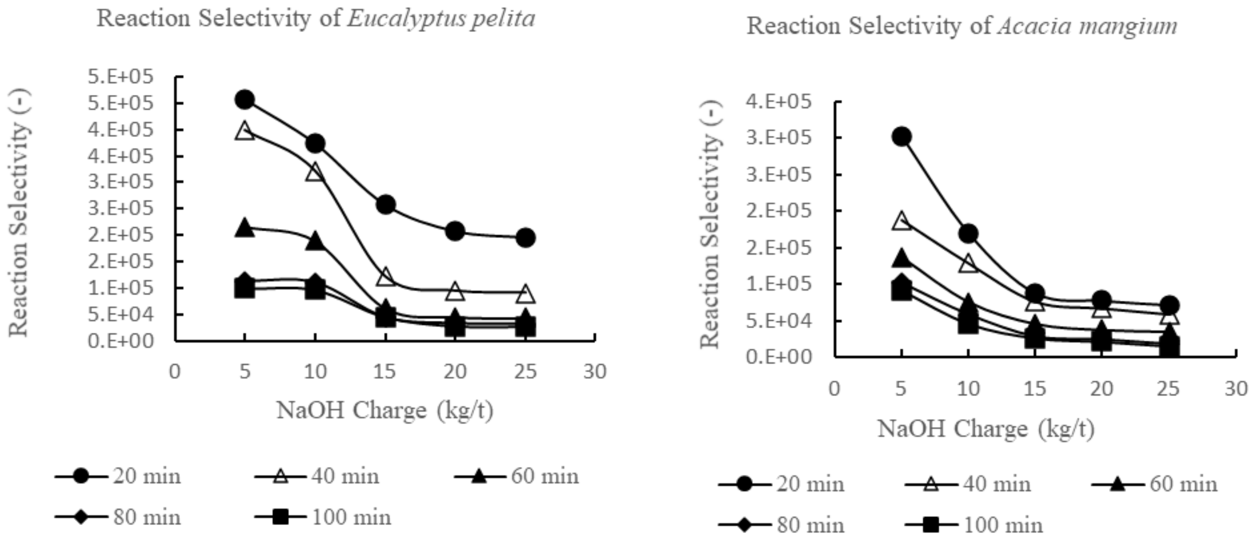
In the *Acacia mangium* samples, the highest DP values were 1392–1559, achieved at a reaction time of 20 min and NaOH charges of 5, 10, 15, 20, and 25 kg/t. Meanwhile, the reaction time of 100 min produced the lowest DP values of 861–1447. For every 5 kg/t increase in NaOH charge at reaction times of 20, 40, 60, 80, and 100 min, the DP values decreased by 41.7, 48.1, 78.2, 133.1, and 146.5, respectively.

Every 5 kg/t increase in NaOH charge per the reaction time variations indicates that the DP reduction in *Acacia mangium* is greater than in *Eucalyptus pelita*. This means that during the cellulose degradation, the cellulose end group is attacked by hydroxide ions and converted to the alkali-stable m-saccharinic more quickly in *Eucalyptus pelita*. As such, the cellulose degradation process is stopped in *Eucalyptus pelita* quickly (Violette, 2003).

3.3. *The Effect of NaOH Charges on Changes in Reaction Selectivity*

The selectivity of medium consistency oxygen delignification at a specific moment may be defined as the ratio of the rate of lignin reactions to the rate of carbohydrate reactions (Violette, 2003).

The parameters of reaction selectivity indicate the quality of the pulp produced in the medium consistency oxygen delignification process, where the higher the reaction selectivity, the more efficient the medium consistency oxygen delignification. The reaction selectivity values of *Eucalyptus pelita* and *Acacia mangium* are featured in Figures 4a and 4b.



(a) (b)

Figure 4 The effect of NaOH charges and reaction time on reaction selectivity per sample

Based on Figures 4a and 4b, the NaOH charge and reaction time in the medium consistency oxygen delignification process significantly influence selectivity, as the longer the reaction time, the higher the reaction selectivity.

In *Eucalyptus pelita*, the highest reaction selectivity was achieved at an NaOH charge of 5 kg/t. The reaction selectivity at reaction times of 20, 40, 60, 80, and 100 min were 4.58×10^5, 3.99×10^5, 2.16×10^5, 1.12×10^5, and 0.99×10^5, respectively. In contrast, the lowest reaction selectivity occurred at an NaOH charge of 25 kg/t. At the reaction times of 20, 40, 60, 80, and 100 min, the reaction selectivity values were 1.95×10^5, 9.07×10^4, 4.36×10^4, 3.27×10^4, and 2.68×10^4, respectively. Based on this variation in reaction time, the longer the reaction time, the lower the reaction selectivity. The reaction times of 20, 40, 60, 80, and 100 min produced reaction selectivity values of 1.95×10^5–4.58×10^5, 9.07×10^4–3.99×10^5, 4.36×10^4–2.16×10^5, 3.27×10^4–1.12×10^5, and 2.68×10^4–9.91×10^4, respectively.

For *Acacia mangium,* the highest reaction selectivity occurred at an NaOH charge of 5 kg/t. The reaction selectivity at reaction times of 20, 40, 60, 80, and 100 min were 3.02×10^5, 1.88×10^5, 1.36×10^5, 1.02×10^5, and 9.18×10^4, respectively. Meanwhile, the lowest reaction selectivity occurred at NaOH charge 25 kg/t. The reaction selectivity at the reaction times of 20, 40, 60, 80, and 100 min were 7.19×10^4, 5.90×10^4, 3.50×10^4, 1.77×10^4, and 1.52×10^4, respectively. Per the variation in reaction time, the longer the reaction time, the lower the reaction selectivity. At the reaction times of 20, 40, 60, 80, and 100 min, the reaction selectivity was 7.19×10^4–30.21×10^4, 5.90×10^4–18.81×10^4, 3.50×10^4–13.58×10^4, 1.77×10^4–10.17×10^4, and 1.52×10^4–9.18×10^4, respectively.

Given the variation in reaction time and NaOH charge, the reaction selectivity of *Eucalyptus pelita* is higher than that of *Acacia mangium* because the DP is smaller in the former than the latter in the medium consistency oxygen delignification process.

4. Conclusions

In medium consistency oxygen delignification, the most important analysis parameters are KaNo and viscosity. This study demonstrated that higher NaOH charges enhance the delignification rate. More lignin will dissolve with an increased NaOH charge. The longer reaction time also results in an increased delignification rate. The decrease of DP values in Eucalyptus pelita and Acacia mangium was due to higher NaOH charge and longer reaction times. Based on the variations in NaOH charge, reaction selectivity decreases according to increased NaOH charge. The 20-min reaction time variations show the highest reaction selectivity.

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