IOP Conf. Series: Materials Science and Engineering **226** (2017) 012175 doi:10.1088/1757-899X/226/1/012175

Effects of Soda-Anthraquinone Pulping Variables on the Durian Rind Pulp and Paper Characteristics: A Preliminary Test

Shaiful Rizal Masrol[1,*], Mohd Halim Irwan Ibrahim[2,3], Sharmiza Adnan[4], Mohd Rahmad Talib[3] and Lau Lee Sian[3]

[1]Faculty of Engineering Technology, Universiti Tun Hussein Onn Malaysia, 86400 Parit Raja, Batu Pahat, Johor, Malaysia
[2]Advanced Manufacturing and Materials Center (AMMC), Office of Research, Innovation, Commercialization and Consultancy Management (ORICC), Universiti Tun Hussein Onn Malaysia, 86400 Parit Raja, Batu Pahat, Johor, Malaysia
[3]Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia, 86400 Parit Raja, Batu Pahat, Johor, Malaysia
[4]Pulp and Paper Laboratory, Biomass Technology Programme, Forest Product Division, Forest Research Institute Malaysia (FRIM), 52109, Kepong, Selangor, Malaysia

Corresponding author email: srizal@uthm.edu.my

Abstract. Good combination of pulping variables is required to obtain the quality pulp and paper characteristics. Thus, in this preliminary work, naturally dried durian rind were pulped under Soda-Anthraquinone (Soda-AQ) pulping process with 18% to 22% alkali charge, 0% to 0.1% Anthraquinone (AQ) charge, 90 minutes to 150 minutes of cooking time and 150°C to 170°C to investigate the effect of pulping variables on the characteristics of the pulp and paper. Pulping condition with 0% of AQ charge was also conducted for comparison. Results indicated that the best screen yield percentage, reject yield percentage, freeness, drainage time, tear index, number of folds and optical properties were shown by the pulp produced with combination of the highest active alkali (22%), AQ charge (0.1%), cooking time (150 minutes) and cooking temperature (170°C) except apparent density, tensile index and burst index. This preliminary result shows that the optimum quality of durian rind pulp as a potential papermaking raw material pulp could be produced by selecting the good combination of pulping variables which influences the pulp and paper characteristics.

1. Introduction

In recent years, the influences of pulping variables were extensively investigated by previous studies in order to obtain the optimum quality of non-wood based pulp and paper. The common pulping variables that usually been investigated in pulping process are active alkali concentration (%), AQ charge (%), time to reach pulping temperature (minute), time at pulping temperature (minute), pulping temperature (°C) and liquor to material ratio. A study by Jimenez *et al.* [1] has investigated the influence of soda-anthraquinone pulping variables (temperature, time and soda concentration) of palm oil empty fruit bunches (EFB) paper sheets. The soda concentrations, temperatures and times used were 10%, 15% and 20% (on dried raw material); 155, 170 and 185 °C; and 30, 60 and 90 min, respectively.

Content from this work may be used under the terms of the Creative Commons Attribution 3.0 licence. Any further distribution of this work must maintain attribution to the author(s) and the title of the work, journal citation and DOI.
Published under licence by IOP Publishing Ltd

Wan Rosli *et al.* [2] conducted caustic pulping of oil-palm frond-fiber strands according to a central composite design using a two-level factorial plan involving three pulping variables (temperature: 160–180 °C, time: 1–2 h, alkali charge: 20–30% NaOH). Pulp with 35-45% yield could be obtained from oil palm frond-fiber strands. This study observed that statistically, the reaction time was not a significant factor. However, the influences of the treatment temperature and caustic charge were relatively significant to the resultant pulp properties.

Mossello *et al.* [3] prepared kenaf-based soda-anthraquinone pulp with cooking conditions (active alkali 12-15%) with a cooking time of 30-90 min and a temperature of 160ºC. The result shows reject content decreases as the active alkali concentration and time increased due to better delignification and separation of the fiber bundle. Active alkali and cooking time had a minor effect on the total yield and screened yield. The result also indicated the total yield decreased from 58.4 to 54.2% (a reduction of 4.23%) and the screened yield decreased from 56.1 to 54.1% (a reduction 3.60%). Meanwhile, Mohd Hassan *et al.* [4] investigated the effect of soda-AQ pulping conditions on the mechanical properties of Semantan bamboo (*Gigantochloa scortechinii*) paper with variables of 10 to 20% alkali charge and 150 to 170°C cooking temperature. Times to reach pulping temperature and time at pulping temperature were fixed at 90 minutes with fixed 0.1% AQ and 1:4 bamboo to liquor ratio. The results indicated that the properties show significant and highly significant different between each treatment except for temperature factor for bursting index was not significant. The study also concluded that alkaline percentage and pulping temperature have high influences in paper mechanical characteristics.

Main *et al.* [5] conducted a study to investigate the suitability of coir fibers as an alternative material in making pulp and paper. Soda-AQ pulping with various combinations of active alkali (18-22%) and cooking time (90- 150 minutes) at fixed temperature (170°C) was done. The study revealed that the pulp cooked with 22% active alkali concentration for 150 minutes gives the highest apparent density value and pulping condition of using 20% active alkali for 150 minutes gives the highest value of tear index. The burst and tensile index increases with the increment in cooking time at 18% and 20% of active alkali concentration but decreased after cooked for 150 minutes at 22% active alkali concentration. The brightness was significantly increases with increasing of active alkali and cooking time, but opacity value of the coir paper was decreased. The study manages to obtain the optimum condition for producing coir paper with the highest strength by using 22% active alkali in 120 minutes cooking time.

Soda pulping process is commonly used for non-wood based fibers. It has been known that anthraquinone (AQ) behaves as a redox catalyst during alkaline pulping, unlike hydroxide ions (OH-) in the conventional soda process [6]. Adding AQ into the soda pulp led to an increase in screen pulp yield and a reduction in rejects [6]. The delignification rate and the preserved pulp yield can be improved with the presence of anthraquinone in the alkaline pulping process [7].

Durian rind is the biggest portion of durian waste besides seeds and others. During the season of durian, the amounts of the rind disposition as waste could lead to environmental problems [8]. Durian rind chemi-mechanical (CMP) pulp and paper were successfully developed and investigated by our previous research [9,10]. In order to go deeper in durian rind pulp and paper research and development, other types of pulping methods such as soda caustic and soda-AQ pulping process were suggested. However, the optimum pulping condition needs to be obtained in order to produce the best quality of durian rind pulp and paper. Thus, the influence of pulping variables (active alkali charge. AQ charge, cooking time and cooking temperature) on durian rind pulp and paper characteristics were investigated in this preliminary work. The general findings through this preliminary study are a part of a bigger project aiming on optimization process in utilizing durian rind as an alternative non-wood based raw material for pulp and paper.

2. Experimental

2.1. Raw material preparation

Durian rinds were collected from a local durian flesh manufacturer at Batu Pahat, Johor, Malaysia. Only durian rinds from a variety of D24 were collected to control the experimental parameters. At the first part, durian rinds were cleaned from the residual aril and dirt. Then, the center divider which holding the durian flesh is removed by a knife. Then, durian rinds were sliced, spine removed, cubed and dried naturally according to Masrol *et al.* [9,10]. Finally, dried durian rind cubes were kept inside the air tight storage to prevent moisture and fungus.

2.2. Pulping process

500 g oven dry (o.d) weight of naturally dried durian rind cubes were pulped using the laboratory digester (MK system) with variables of 0 to 0.10 % anthraquinone (AQ) charge, 18 to 22% alkali charge and 150 to 170°C cooking temperature as listed in Table 1. Overall 9 pulping conditions process were conducted according to Soda-AQ pulping process except for test T1, T4 and T7 which using caustic soda pulping process in order to investigate the effect of AQ addition. The AQ concentration was set to zero for the test no T1, T4 and T7 for comparison purpose. The amount of anthraquinone (AQ) added into the liquor was obtained according to the maximum value permitted by the Food and Drug Administration (FDA) of the United States [11,12]. The constant parameters for each treatment of durian rind pulping process were durian rind oven-dry (o.d) weights to liquor ratio (1:7) and time to reach maximum pulping temperature (90 minutes).

The pulps were disintegrated in a hydro-pulper for 5 minutes and washed thoroughly with running water on a fine filter in order to remove the remaining black liquor. Then, PTI Sommerville Fractionators is used for the screening process with accordance to TAPPI T 275 standard with a slot size of 0.15 mm. Next, a spin-dry extractor is used to remove the excess water from the screened pulp followed by a dispersed process using the Hobart mixer. Finally, durian rinds pulps were sealed in the plastic bags and kept inside a cold chiller at a temperature of 6°C for further investigation.

Table 1. Pulping Conditions

Test no.	A	B	C	D
	Active Alkali (%)	A.Q (%)	time (min)	Temp (°C)
T1	18	0.00	90	150
T2	18	0.05	120	160
T3	18	0.10	150	170
T4	20	0.00	90	150
T5	20	0.05	120	160
T6	20	0.10	150	170
T7	22	0.00	90	150
T8	22	0.05	120	160
T9	22	0.10	150	170

2.3. Laboratory hand sheets preparation

Durian rind hand sheets with a basis weight of 60±3 g/m^2 were produced from unbleached and unbeaten pulp by semi-automatic sheet machine (British Handsheet Machine) according to MS ISO 5269-1:2007, IDT Pulps – Preparation of Laboratory Sheets for Physical Testing –Part 1: Conventional Sheet-Former Method. The 60 gsm hand sheet preparation were explained in our previous article by Masrol *et al.* [9,13]. The freeness test (2 sets with 1 liter of diluted pulp per set) was performed according to the TAPPI T 227 om-99: Freeness of Pulp (Canadian Standard Method). Drainage time test was performed according to TAPPI T-221: Drainage Time of Paper Pulp. Finally, the hand sheets were dried for 24 hours and conditioned for another 24 hours inside a control room at 23 ± 1 °C and 50 ± 2.0% RH according to TAPPI T 402 sp-03 Standard Conditioning and Testing Atmospheres for Paper, Board, Pulp Handsheets and Related Products and MS ISO 187: 2001, IDT for further evaluation.

2.4. Characteristics test

The durian rind pulp and paper were tested according to ISO 5270: 2012 "Pulps - Laboratory sheets - Determination of physical properties". The lists of Malaysian Standard Methods (MS ISO) test for various physical mechanical and optical properties were conducted in this study, including grammage (MS ISO 536 : 2001, IDT), thickness (MS ISO 534: 2007,IDT), brightness (MS ISO 2470-1: 2010), opacity (MS ISO 2471: 2010), Tensile (MS ISO 1924-2), Tearing (MS ISO 1974 : 1999), bursting strength (MS ISO 2758 : 2007) and folding (MS ISO 5626 : 1999).The overall characteristics test was conducted at a controlled temperature and humidity environment room as stipulated in TAPPI T 402 sp-03 Standard Conditioning and Testing Atmospheres for Paper, Board, Pulp Handsheets and Related Products and MS ISO 187: 2001, IDT. The sampling was conducted according to TAPPI T400 and MS ISO 186: 2002, IDT.

3. Results and Discussion

3.1. Effect of Soda-AQ pulping variables on pulp characteristics

Table 2 shows the screened yield increased as the AQ percentage, time and temperature were increased to the same active alkali percentage. Figure 1 shows screened yield was influenced by the active alkali charge percentage. It can be seen that durian Soda-AQ pulp shows better screened yield percentage compared to durian rind soda pulp. The effects of AQ application provide an increased pulp yield by stabilization of carbohydrates [6,14]. Adding the AQ in pulping lowered the amount of screening rejects compared to the soda pulp [15]. The highest screen yield (38.88 %) and lowest reject percentage (0.18%) were obtained by Soda-AQ pulping condition with 22% active alkali, 0.10% AQ, 150 minute time at maximum temperature, 170°C cooking temperature. A reject content less than 1% is indicative of easy and uniform cooking [16].

Table 2. Effect of pulping variables on pulp characteristics

Test No.	Active Alkali (%)	A.Q (%)	Time (min)	Temp (°C)	Screened Yield (%)	Reject Yield (%)	Freeness (mL)	Drainage Time (s)
T1	18	0.00	90	150	14.44	28.88	146.25	170.00
T2	18	0.05	120	160	17.60	19.32	187.00	79.25
T3	18	0.10	150	170	30.72	9.87	229.50	15.38
T4	20	0.00	90	150	16.00	26.84	155.25	105.50
T5	20	0.05	120	160	23.18	17.61	196.50	37.00
T6	20	0.10	150	170	37.85	1.37	300.50	10.13
T7	22	0.00	90	150	16.21	23.15	175.50	77.38
T8	22	0.05	120	160	24.51	17.04	222.00	23.63
T9	22	0.10	150	170	38.88	0.18	325.25	9.13

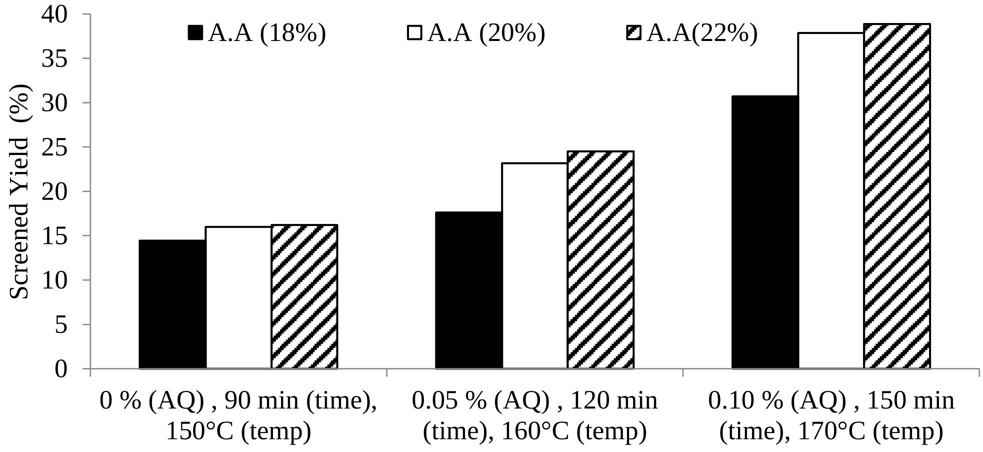

Figure 1. Pulping variables effects on durian rind soda-AQ pulp screen yield percentage

Figure 2 shows there was a significant effect of active alkali charge, AQ charge, cooking time and cooking temperature on durian rind pulp freeness and drainage time. Fast drainage time should be considered for papermaking material to reduce manufacturing cost. The best freeness and drainage time (faster) are showed by Soda-AQ pulping condition with 22% active alkali, 0.1% AQ, 150 minutes cooking time and 170°C cooking temperature. Figure 2 also shows that durian rind soda-AQ pulp shows better freeness and drainage time compared to durian rind soda pulp.

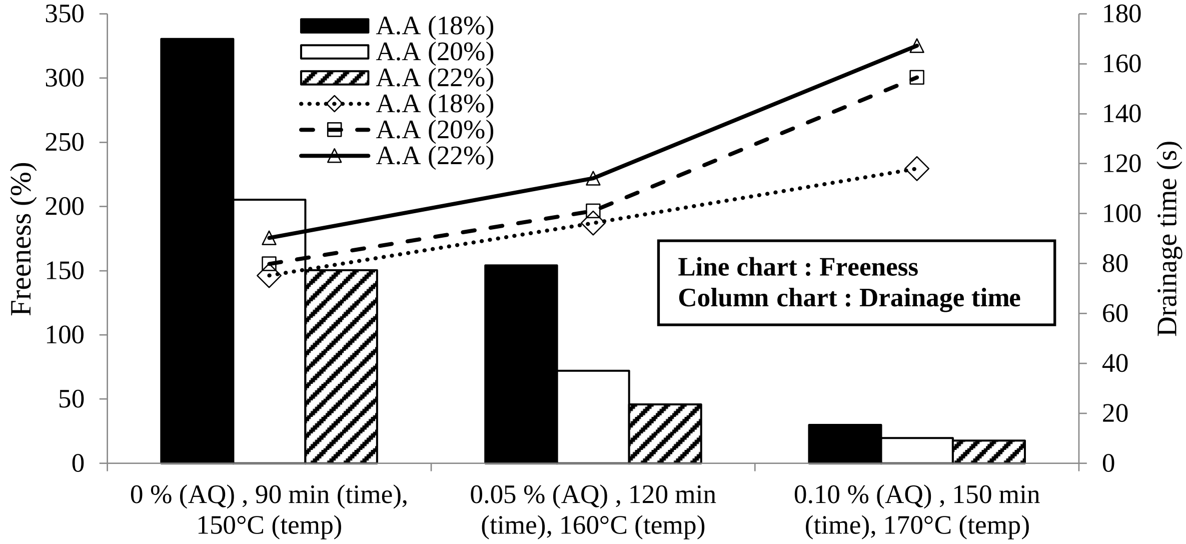

Figure 2. Pulping variables effects on durian rind soda-AQ pulp freeness and drainage time

3.2. The effect of pulping variables on optical characteristics

The obtained optical characteristics which are ISO brightness, ISO opacity, scattering coefficient and absorption coefficient for 9 conditions of durian rind pulp were summarized in Table 3. It can be seen there was an increment pattern of ISO brightness, opacity, scattering coefficient and absorption coefficient resulted from the increasing of active alkali charge, AQ charge, cooking time and cooking temperature. Figure 3 that ISO brightness was significantly influenced by active alkali charge. Alkali charge was the most influential factor in relation to the brightness, followed by the temperature, in order of importance [2]. It was also observed that durian rind Soda-AQ pulp shows better ISO brightness compared to Soda pulp. This condition is due to the addition of AQ has increased the delignification rate [6,14], hence resulting in better brightness result.

Table 3. Effect of pulping variables on optical characteristics

Test no.	Active Alkali (%)	AQ (%)	Time (min)	Temp. (°C)	ISO Brightness (%)	ISO Opacity (%)	Scattering Coefficient (m^2/kg)	Absorption Coefficient
T1	18	0.00	90	150	14.05	83.64	5.70	8.04
T2	18	0.05	120	160	16.41	91.24	7.88	9.85
T3	18	0.10	150	170	26.32	96.95	19.49	10.87
T4	20	0.00	90	150	15.40	83.98	6.59	9.53
T5	20	0.05	120	160	19.05	91.34	9.77	10.24
T6	20	0.10	150	170	31.88	97.03	25.59	11.06
T7	22	0.00	90	150	18.85	89.10	7.27	9.85
T8	22	0.05	120	160	23.91	91.40	12.77	10.28
T9	22	0.10	150	170	33.23	97.06	26.61	11.24

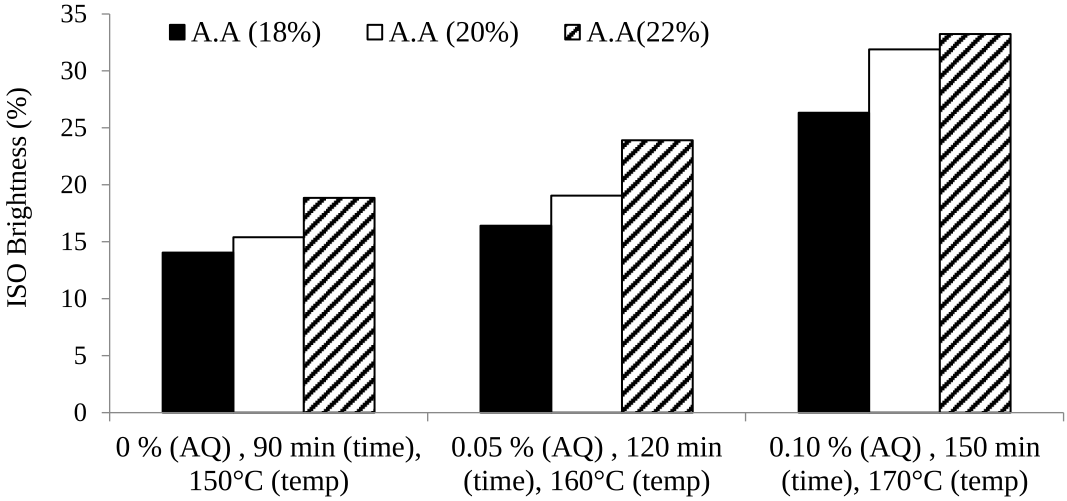

Figure 3. Pulping variables effects on durian rind soda-AQ ISO brightness

Figure 4 shows ISO opacity was increased with the effect of increasing AQ charge, cooking time and cooking temperature. It can be seen in the similar AQ charge, cooking time and cooking temperature there was no significant effect of active alkali charge on the durian rind ISO opacity. Thus, durian rind soda-AQ pulp shows better ISO opacity compared to durian rind soda pulp. Pulp produced with 0.10 % AQ shows high opacity compared to 0% and 0.05%. Opacity is a fundamental optical property and influenced by the paper thickness [5]. Thus, the decreased pattern of durian rind paper bulk density (Table 4) which indirectly effected by the increment in paper thickness resulting in higher opacity value as illustrated in Figure 4.

In this study, it can be seen that durian rind paper with higher brightness shows better of opacity which effected by the increment of active alkali charge, AQ percentage, cooking time and cooking temperature.

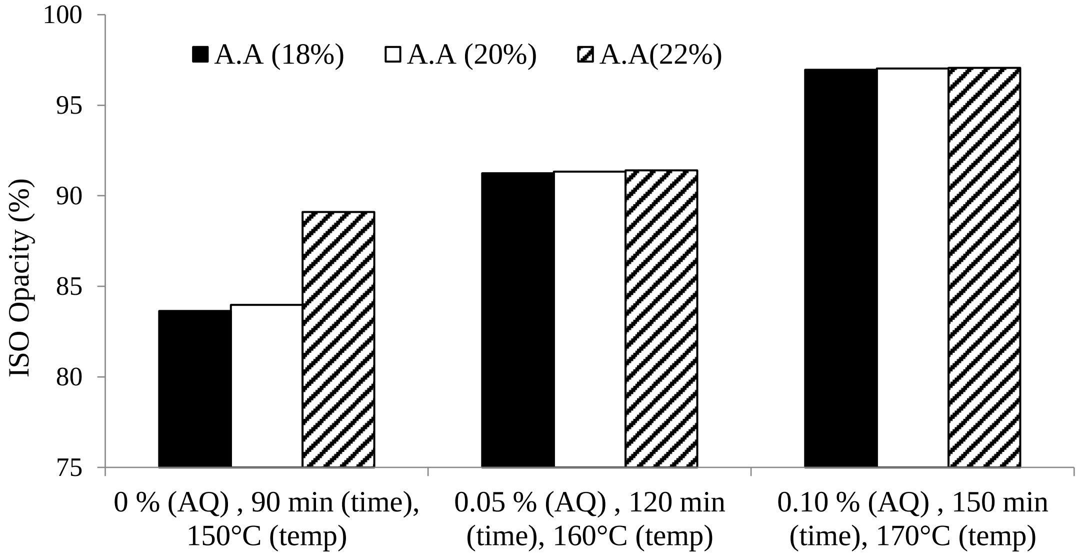

Figure 4. Pulping variables effects on durian rind soda-AQ opacity

3.3. The effect of pulping variables on physical and mechanical characteristics

Table 4 shows the influence of pulping variables on durian rind paper sheet physical and mechanical characteristics. Result in Table 4 indicated that durian rind paper bulk density was decreased with the increment of active alkali, AQ, cooking time and cooking temperature. This condition also clearly visualized by Figure 5. The highest paper bulk density was obtained by pulping condition with 18% active alkali, 0 % AQ, 90 minutes cooking time and 150 °C cooking temperature which under caustic soda pulp. It can be seen the addition of AQ with the increment of cooking time and cooking temperature resulting durian rind pulp with lower paper bulk density.

Table 4. Effect of pulping variable on physical and mechanical characteristics

Test no.	Active Alkali (%)	AQ (%)	Time (min)	Temp. (°C)	Paper bulk density (g/cm^3)	Tensile Index (N.m/g)	Tear index (mN.m^2/g)	Fold no.	Burst Index (kPa.m^2/g)
T1	18	0.00	90	150	0.930	68.53	1.56	278	3.78
T2	18	0.05	120	160	0.919	65.93	4.20	540	3.45
T3	18	0.10	150	170	0.767	52.50	9.30	879	3.24
T4	20	0.00	90	150	0.925	68.12	3.62	367	3.67
T5	20	0.05	120	160	0.836	61.96	7.32	734	3.43
T6	20	0.10	150	170	0.691	41.70	9.50	1074	3.15
T7	22	0.00	90	150	0.906	64.48	4.56	390	3.58
T8	22	0.05	120	160	0.814	58.35	8.23	836	3.41
T9	22	0.10	150	170	0.687	38.02	9.51	1101	3.08

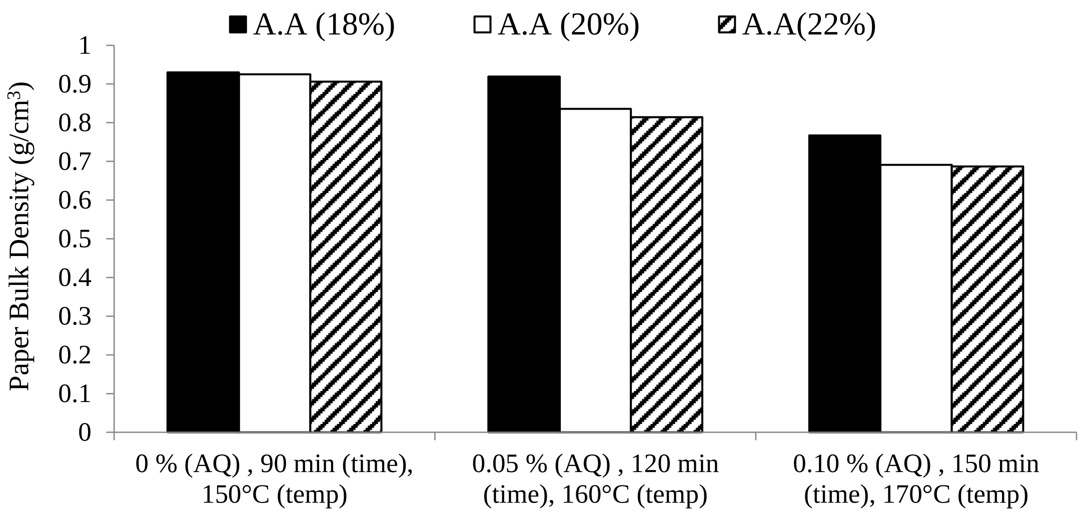

Figure 5. Pulping variables effects on durian rind soda-AQ paper bulk density

For the tensile index, result in Figure 6 illustrated a decline pattern with the increment of active alkali concentration, AQ charge, cooking time and cooking temperature. It can be seen soda caustic durian rind pulp shows better tensile index compared to the soda-AQ pulp. Figure 7 show that durian rind burst index is slightly decreased as the active alkali concentration was increased. Furthermore, there were slightly decreased of durian rind paper burst index resulted from the increasing of AQ charge, cooking time and cooking temperature. Both tensile and burst index pattern was influenced by the decreasing of

IOP Conf. Series: Materials Science and Engineering **226** (2017) 012175 doi:10.1088/1757-899X/226/1/012175

paper bulk density shown in Table 4 and Figure 5. This condition could be improved by refining or beating process that influences the pulp and paper characteristics [17].

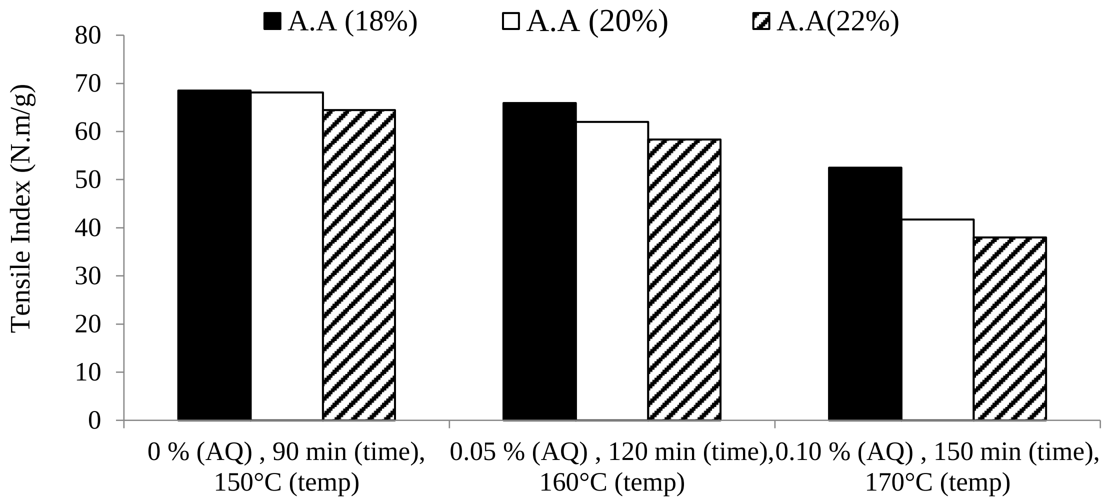

Figure 6. Pulping variables effects on durian rind soda-AQ paper tensile index

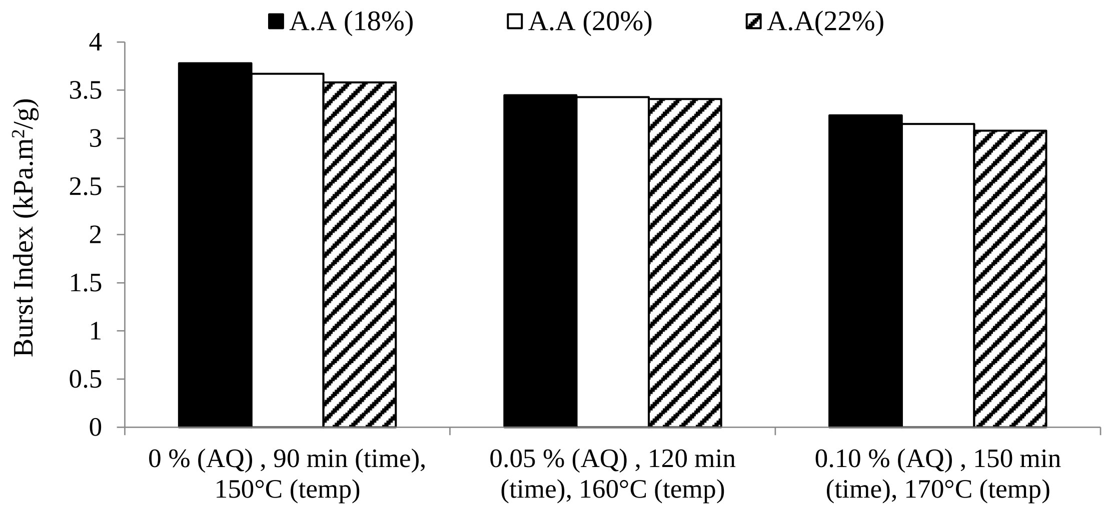

Figure 7. Pulping variables effects on durian rind soda-AQ burst index

Durian rind tear index increases with the increment of active alkali concentration, AQ charge, cooking time and cooking temperature as shown in Figure 8. However, at 0.1% AQ charge, 150 minutes cooking time and 170°C cooking temperature there were only slightly increment with the effect of active alkali concentration. This was similar to the finding by Akgül and Tozluoglu which shows the addition of AQ in soda pulping increased the tear index of the pulps [15]. In folding, Figure 9 show that folding no. increases with the increment of active alkali concentration. Meanwhile, at 0.1% AQ, 150 minutes cooking time and 170°C, there were slightly different between pulps produced with 20% active alkali compared to 22%. In the similar active alkali concentration, folding no. also showing an increment pattern as the AQ charge, cooking time and cooking temperature was increased. It can be seen, Soda-AQ durian rind pulp shows better fold characteristic compared to soda pulp.

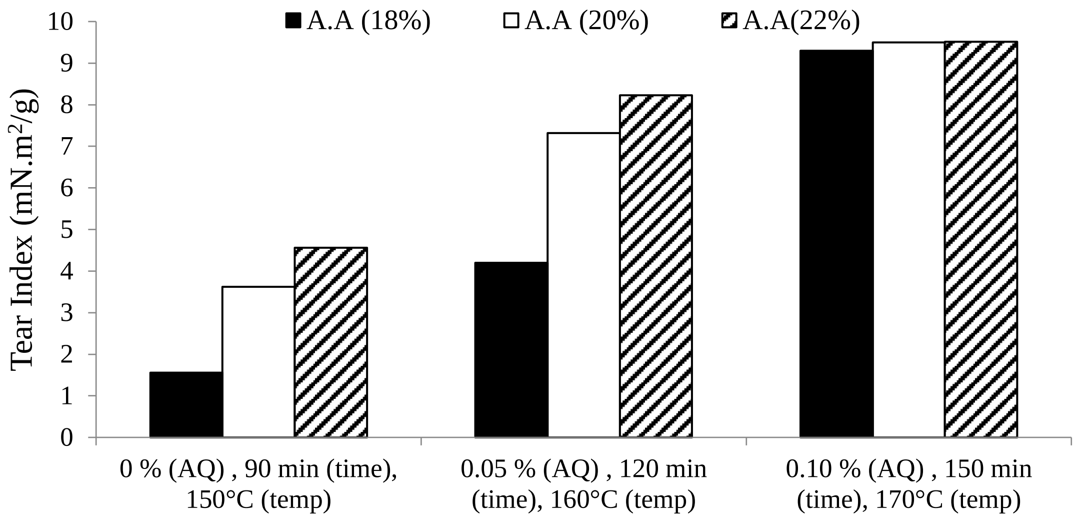

Figure 8. Pulping variables effects on durian rind Soda-AQ paper tear index

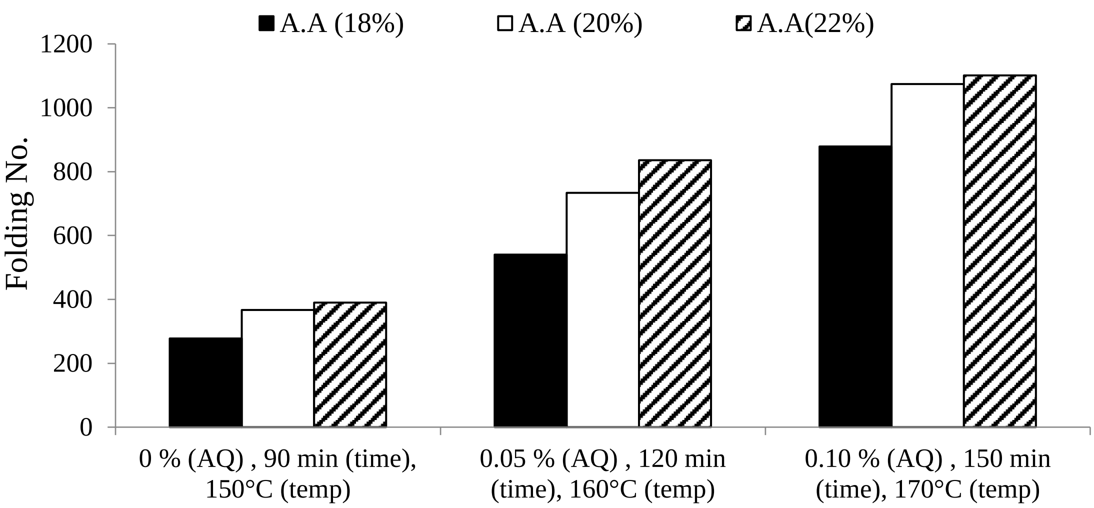

Figure 9. Pulping variables effects on durian rind soda-AQ paper folding no.

4. Conclusion

In this preliminary study, the influence of four pulping variables which are active alkali concentration, Anthraquinone (AQ) charge, cooking time and cooking temperature of durian rind soda-anthraquinone pulp was successfully investigated and generally discussed. The result indicated that all four variables had influenced the physical, optical and mechanical characteristics of durian rind pulp and paper. It has been observed that durian rind pulp produced with soda-AQ pulping process offer better quality then caustics soda pulp except for paper bulk density, tensile index and burst index. Results indicated that the best screen yield percentage, reject yield percentage, freeness, drainage time, tear index, number of folds and optical properties were shown by the pulp produced with combination of the highest active alkali (22%), AQ charge (0.1%), cooking time (150 minutes) and cooking temperature (170°C) except for apparent density, tensile index and burst index. This preliminary result shows that the optimum quality of durian rinds soda-AQ as a potential papermaking raw material pulp could be produced by selecting the good combination of pulping variables. In future, pulping optimization process and statistical analysis should be considered in order to obtain the optimum durian rinds pulp. Finally, this preliminary study offers a huge potential research focus and development area on utilizing durian rind as an alternative material for pulp and paper.

5. Acknowledgements

The authors would like to thank Universiti Tun Hussein Onn Malaysia for funding this project under UTHM Short Term Grant (STG) Vot. 1333 and Ministry of Higher Education Malaysia under SLAI scheme. Author would also like to acknowledge Pulp and Paper Laboratory, Biomass Technology Program, Forest Product Division, Forest Research Institute Malaysia for research facilities and support

References

[1] Jiménez L, Serrano L, Rodríguez A and Sánchez R 2009 *Bioresour. Technol.* **100(3)** 1262
[2] Wan Rosli W D, Law K N, Zainuddin Z and Asro R 2004 *Bioresour. Technol.* **93(3)** 233
[3] Mossello A A, Harun J, Resalati H, Ibrahim R, Md Tahir P, Fallah Shamsi S R and Mohamed A Z 2010 *Bioresources* **5(3)** 1542
[4] Mohd Hassan N H, Muhammed S and Ibrahim R 2013 *Malaysian J. Anal. Sci.* **17(1)** 75
[5] Main N M, A Talib R, Ibrahim R, Abdul Rahman R and Mohamed A Z 2014 *Agric. Agric. Sci. Procedia* **2** 304
[6] Shakhes J, Zeinaly F, Marandi M A B and Saghafi T 2011 *Bioresources* **6(4)** 4626
[7] Ferrer A, Vargas F, Jameel H and Rojas O J 2015 *Bioresources* **10(4)** 6442
[8] Wai W W, AlKarkhi A F M and Mat Easa A 2010 *Carbohydr. Polym.* **79(3)** 584
[9] Masrol S R, Ibrahim M H I and Adnan S 2015 *Procedia Manuf.* **2** 171
[10] Masrol S R, Ibrahim M H I, Adnan S, Ahmad Tajudin M S A, Abdul Raub R, Abdul Razak S N A and Md Zain S N F 2016 *MATEC Web Conf.* **51** 02007
[11] Main N M, A Talib R, Ibrahim R, Abdul Rahman R and Mohamed A Z *Bioresources* **10(4)** 6975
[12] FDA 2015 *CFR - Code of Federal Regulations Title 21; Sec 176.170: Components of paper and paperboard in contact with aqueous and fatty foods* (US Food and Drug Administration (FDA)) https://www.accessdata.fda.gov/scripts/cdrh/cfdocs/cfCFR/CFRSearch.cfm?fr=176.170
[13] Masrol S R, Ibrahim M H I, Adnan S, Amir Shah M S S, Main N M, Esa M F and Othman M H 2014 *Appl. Mech. Mater.* **660** 373
[14] Rodríguez A, Sánchez R, Eugenio M E, Yáñez R and Jiménez L 2010 *Cellul. Chem. Technol.* **44(7–8)** 239
[15] Akgül M and Tozluoglu A A 2009 *African J. Biotechnol.* **8(22)** 6127–6133
[16] Wan Rosli W D, Zainuddin Z and Lee L K 2004 *Wood Sci. Technol.* **38(3)** 191.
[17] Masrol S R, Ibrahim M H I, Adnan S, Amir Shah M S S, Main N M, Esa M F and Othman M H 2015 *Appl. Mech. Mater.* **773–774** 158