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Characteristics of Pulp and Paper Made from MD2 Pineapple Leaf Via Soda-Anthraquinone (AQ) Pulping Method

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Abstract: Increasing demand of paper has increased the interest in searching a new alternative non-wood based raw material for papermaking industry. Pineapple leaf fibre had been utilized as one of the potential papermaking materials. Therefore, the purpose of this study is to observe the potential of pineapple leaf from Malaysian MD2 variety as papermaking fibre. MD2 pineapple leaf pulp was produced according to soda-Anthraquinone (AQ) pulping process with 20% active alkali dosage, 0.1 % AQ, 8:1 liquor to fibre ratio, 90 minutes of cooking time and 170 °C cooking temperature. Handsheet with the basis weight of 120 gsm were produced from virgin soda-AQ pulp. The characteristic test of the pulp and paper were conducted in accordance with the Technical Association of the Pulp and Paper Industry (TAPPI) and Malaysian International Organization for Standardization (MS ISO) standard. The result has observed that virgin (untreated) MD2 Soda-AQ pulp recorded a value of moisture content (76.73%), screened yield percentage (17.33%), pulp drainage time (90 s), and freeness (160 ml). In physical characteristics analysis, MD2 Soda-AQ recorded a mean value of 0.522 g/cm^3 and 220.545 µm for bulk density and bulk thickness, respectively. Meanwhile, mechanical characteristics also evaluated by tensile index (40.919 N.m/g), tearing index (4.588 mN.m^2/g), burst index (2.734 kPa.m^2/g) and folding no (12.375). This study shows the potential characteristics of MD2 pineapple leaf as a non-wood based alternative material for papermaking.

Keywords: Pineapple Leaf, Non-Wood, Pulp and Paper, Soda Pulping

1. Introduction

Paper is defined as a thin sheet material which produced by processing cellulose fibres derived from plant mechanically or chemically. Paper is considered as one of the highest demand materials with consumption amounting 399 million metric tons in 2020 [1]. This is due to the strong demand for books, newspapers, packaging materials, and other printed goods. Increased demand for paper entails more deforestation to get raw materials, which may cause significant environmental and ecological damage.

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In recent years, a lot of studies and research had been conducted to find renewable source to replace the raw material needed for papermaking process which mainly come from softwood tress namely spruce, pine, fir and hemlock including hardwoods as well as eucalyptus, aspen, and birch [2].

1.1 Research Background

In recent years, researcher had been researching on suitable fibre which can be used to make paper. Changing the perspective by utilizing readily and abundant renewable source might help to combat and mitigate climate changes while still meeting future sources demand were a wise move. Thus, pineapple leaf fibre had been introduced as potential source for the pulp and paper industries as a non-wood fibre for papermaking [3].

Pineapple or its scientific name *Ananas comosus* is an important tropical fruit found in the Bromeliaceae family. Under the Malaysia National Agrofood Policy 2011-2020 plantation area were planned to be increased where Malaysian Pineapple Industry Board (MPIB) had expected to achieve 20000 ha of plantation area where 50% of it comes from MD2 variety to accommodate domestic and export market demand [4]. Thus, the usage of MD2 pineapple leaf for paper making will also create a way to utilize the discarded agricultural residue. MD2 pineapple leaf grows around 41-43 pieces of leaves with length and width ranging to 66-69 cm and 5.1-5.3 cm respectively. Pineapple leaves contain the lowest lignin percentage (10.5%) compared to coconut (32.8%), softwood (21-37%) and hardwood (14.34%). It suggests it is less resistant to bleaching and has a high fibre strength [5]. Moreover, the high percentage of Holocellulose α-cellulose will give positively affect the pulp yield of a fibre [6]. Pineapple leaf which has a favourably high cellulose content means that a high quality of paper can be produced. Pulp derived from pineapple leaf also gave excellent results (79.26%) of pulp yields [7]. Thus, it can be concluded that pineapple leaf is a suitable raw material to be used for soda-Anthraquinone (AQ) pulping process for this study.

The aim of this study is to produce paper pulp by using MD2 pineapple leaf via soda-Anthraquinone (AQ) pulping method. The effects of effects of the active alkali dosage used during pulping process on the physical and mechanical characteristics of MD2 pineapple leaf pulp and paper will also be evaluated. This will in turn create a way to utilize the widely discarded pineapple leaf in agricultural sector and manage agricultural residue from the plantation.

2. Materials and Methods

2.1 Material Preparation

MD2 pineapple leaf were provided by Nature Renascent Sdn Bhd. a company located from Skudai, Johor. The raw MD2 pineapple leaf will firstly be cleaned from any foreign substance or matter by using running water. The layer of wax located behind the leaves were removed by using sponge. The MD2 pineapple leaf will then be measured and cut with size of 3 cm × 3 cm (Figure 1a) and dried naturally under sunlight for seven (7) days (Figure 1b) until the leaf dried uniformly. The air dry (a.d.) MD2 pineapple leaf will then be stored in a container at room temperature. Five (5) sample weighing 5 grams each (Figure 1c) was inserted into the electronic drying oven and moisture content and oven dry (o.d.) weight of the pineapple leaf chips were calculated.

Figure 1: (a) 3 cm long pineapple leaf (b) drying process (c) moisture content analysis sample

2.2 Soda-Anthraquinone (AQ) Pulping

In this process, a series of process were initiated to cook the pineapple leaf. The digestion process had been conducted once, at Forest Research Institute Malaysia (FRIM) located in Selangor. The dried pineapple leaves were inserted into the rotary digestor after the oven-dry (o.d.) weight of the pineapple leaf were weighted and be mixed evenly with the chemicals and distilled water. Soda-Anthraquinone (AQ) pulping process was conducted using 900 g o.d. dried MD2 pineapple leaves. Table 1 display the fixed pulping parameters.

Table 1: Soda-Anthraquinone (AQ) pulping conditions of the MD2 pineapple leaf

Soda-Anthraquinone (AQ) Pulping Condition	Value
NaOH concentration	20 %
Liquor to fibre ratio	8:1
Cooking Temperature	170 °C
Time to Reach Cooking Temperature	90 minutes
Time at Cooking Temperature	90 minutes
Anthraquinone (AQ)	0.1 %

A similar study by Sibaly & Jeetah [8] by using 15% NaOH with 90 minutes cooking time produced pulp with tensile index and burst index of 6.5 N.m/g and 0.84 kPa.m^2/g respectively. Thus, this studies carried out the same parameter with increased chemical dosage at 20% NaOH to improve the screen yield and reduce screen rejects [9]. Addition of 0.1% anthraquinone of fibre dry weight was also used as a catalyst for digestion to increase the pulp physical and mechanical characteristics. The liquor to fibre ratio was also increased to 8:1 to prevent redeposition of lignin which can reduced the quality of the pulp [10]. Then, the softened pineapple leaf pulps were inserted into Hydra Pulper to wash the collected pulp and filter every single pulp possible to ensure a thorough cleaning of the pulp as well as disintegrating the pulp and prevent the pulp from clumping. Screening process was conducted by utilizing PTI Sommerville Screen Fractionators in accordance with TAPPI T-275 standard. The main test chamber of this machine consists of a slotted screen platted with slots with size of 0.15 mm which will allow the pulps to pass through the slots and be collected on the fine sieve. The screened reject and the screened yield collected from this machine were weighed based on o.d material basis.

2.3 Preparation of laboratory handsheet

Series of procedures were conducted to turn the virgin pineapple leaf pulp soda-AQ into 120 gsm paper sheets in accordance with the Technical Association of Pulp and Paper Industry (TAPPI) T-205 “Forming Handsheet for Physical Test of Pulp”. The pulp required for 12 sheets of paper amounting to 42 g o.d. had carefully weighted and were disintegrated using pulp disintegrator (Figure 2a) with 20,000 revolutions. Next, the disintegrated pulp was poured into the Stock Divider (Figure 2b). Additional water was added until it reaches 14 litres level. 2 sets of freeness (1 litre each) were collected. The freeness test was conducted according the TAPPI T 227 om-99. Water was added back into the stock divider until it reaches 14 litres again. Then, 2 sets of correction sample (1 litre each) were taken out to check the paper sheet weight is over or lower than as standard weight (2.44g). Since no water will be added back after correction test, the remaining 12 litres of diluted pulp slurry were used to produce testing samples using the hand sheet former (Figure 2c). A bloating paper along with a couching plate were placed on top of the formed sheet and rolled with a roller 5 to 6 times. Next, the formed hand sheet is removed carefully along with three pieces of bloating paper and the couch plate. To form uniformly flat handsheet, pneumatic sheet press (Figure 2d) with applied pressure of 345 kPa for 10 minutes for first press and another 5 minutes for second press. The paper sheets were placed on the surface of the polished steel plate and arranged up and down between the drying rings to produced paper in flat surface condition as (Figure 2e). A heavy weight was placed on top of the drying ring which will apply pressure to handsheet edge and ensure the drying process were done uniformly inside a temperature-controlled environment at 23 ºC ± 1 ºC and 50% ± 2.0% RH in accordance with TAPPI T 402 sp-03, for a least 24 hours before testing were conducted. Next, the dried handsheets (Figure 2f) were peeled from the plates and the drying ring and stored inside a control room for another 24 hours for conditioning.

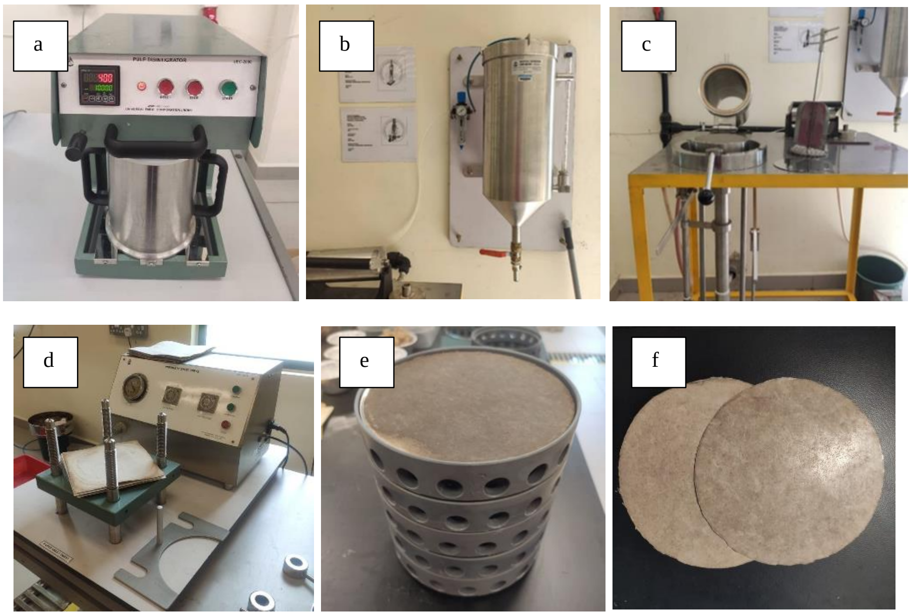

Figure 2: (a) Pulp Disintegrator (b) Stock Divider (c) Handsheet Former (d) Pneumatic Sheet Press (e) Drying Rings (f) Handsheets sample

2.3 Pulp and Paper Properties Testing Procedure

Eight (8) pieces of MD2 pineapple leaf paper samples had been selected for characteristic test. The test standard used for the characteristic test and its equipment involved are listed in Table 2 below. Findings were recorded and calculated to determine the means and standard deviation of the data.

Table 2: Standard used for each characteristics test used

No.	Test	Standard	Machine/Device
1	Freeness	TAPPI T 227 om-99	Freeness Tester
2	Drainage Time	TAPPI 221	Handsheet Former
3	Thickness	TAPPI T 411 om-97	Precision Micrometre
4	Density	MS ISO 534	-
5	Grammage	MS ISO 536	Drying Oven/ scale
6	Tensile	MS ISO 2470-1	Horizontal Tensile Testing Machine
7	Tearing Tester	MS ISO 1974	Tearing Tester
8	Bursting	MS ISO 2758	PTA-Line Burst Tester
9	Folding No	MS ISO 5626	Folding Endurance Tester

3. Results and Discussion

3.1 Pulp Characteristics

Properties such as pulp moisture content, pulp screened yield, freeness and pulp drainage time were the crucial factors which need to be determined in papermaking. Table 3 shows the pulp properties derived from pineapple leaf obtained in this experiment.

Table 3: MD2 pineapple leaf virgin (untreated) Soda-Anthraquinone (AQ) pulp characteristics

Pulp Properties	Screen Yield Percentage (%)	Moisture Content (%)	Freeness (ml)	Drainage Time (s)
Average	17.33	76.73	160.00	90.40
STDV	-	2.98	28.28	5.40

The study produced a total of 17.33% of screen yield percentage in which produces 720g a.d. weight of virgin pulp out of 1200g a.d. pineapple leaf used in digestion process. This may be due to high concentration of NaOH used compared to study by Wutisatwongkul et al. [11], where 15% NaOH concentration was used which produced 21.26% screen yield percentage. It is safe to assume that the percentage yield decreases as the concentration increases. The screen yield obtained in this study unable to achieve the range 40-55% yield for chemical pulping. This research yielded an average of 160 ml freeness which also reflected with long drainage time of 90.4 seconds in which considered a lengthy duration which indirectly effecting the production time of papermaking process. It demonstrates that the water content that was absorbed into the pineapple leaf is retained [12]. Even so, further optimization can be done to pulping parameter such as active alkali percentage, maximum cooking temperature, liquor to material ratio, time at maximum temperature and time to maximum temperature to improve the screen yield percentage of the pulp. The freeness and drainage time value can be improved by applying beating treatment to increase fines fraction which will lessen the likelihood of fibres sliding off the paper plane [13]. This will in turn, improve the CSF and decrease the drainage time.

3.2 Physical Characteristics

To evaluate the quality of a paper produced, the physical properties of the paper such as the grammage, thickness and the paper bulk density are to be investigated. As shown on Table 4, the physical properties of the MD2 pineapple leaf paper produced in the experiment were calculated and tabulated.

Table 4: Physical properties of MD2 pineapple leaf paper

Physical Properties	Grammage (gsm)	Thickness (μm)	Paper Bulk Density (g/cm^3)
Average	115	220.545	0.522
STDV	-	12.227	0.0256

3.2.1 Grammage

The grammage of a paper were evaluated in square metre (g/m^2) or often called gsm unit. The paper sample were taken from the tearing test in which comes total of 16 sample altogether which were conditioned for 24 hours by using drying oven. The grammage of the produced paper sample was 115 gsm in which is not far from the targeted value of 120 gsm +/- 5 gsm.

3.2.1 Paper Thickness and Bulk Density

To determine the thickness of the MD2 pineapple leaf paper sample produced 8 samples were selected and tested using the Bench-top Precision Micrometre machine where 5 different spot were evaluated. The average thickness of a single paper sample yielded an average of 220.545 µm. Meanwhile, for the paper bulk density is 0.521 g/cm^3. A study by Bloch [14] stated that the increase in grammage positively affect the density of paper sheet as well the inter-fibre pore size distribution of the sheet. Hence, it can be concluded a successful derivation of 120 gsm paper sheet would increase the overall physical and mechanical characteristics of the MD2 pineapple leaf pulp and paper.

3.3 Mechanical Characteristics

The mechanical properties of paper are characteristics that describe how the paper behave when subjected to mechanical forces. This will then be used to assess the quality of paper produced. Shown in Table 5 are the mechanical properties of the paper derived from virgin MD2 pineapple leaf pulp.

Table 5: Mechanical Properties of MD2 pineapple leaf paper

Mechanical Properties	**Tensile Index (N.m/g)**	**Tearing Index (mN.m^2/g)**	**Burst Index (kPa.m^2/g)**	**Folding no (double fold)**
Average	40.919	4.588	2.734	12.375
STDV	1.294	0.226	0.272	3.926

3.3.1 Tensile Index

The paper samples produced an average 70.414 N of strength before failure. By dividing the force applied to the contact area, the tensile strength can be computed with resulted in an average of 4694.125 N.m^2/g. On the other hand, tensile index was computed by dividing paper sample grammage with the tensile strength which yielded an average of 40.919 N.m/g. Research by Ferdous et al. [6] and Wutisatwongkul et al. [11] yielded nearly identical tensile index of 40.3 N.m/g and 44.13 N.m/g respectively.

3.3.2 Tearing Index

MD2 pineapple leaf paper recorded a value of 4.59 mN.m^2/g of tearing index. This value is higher that the tearing index of pineapple leaf soda pulp and paper reported Wutisatwongkul et al. [11] with a value of 1.68 mN.m^2/g. This might have been occurred due to better interfibre bonding and strength quality of the paper sample produced by soda-AQ pulping method.

3.3.3 Burst Index

Burst index value (2.73 kPa.m^2/g) of MD2 pineapple leaf soda-AQ paper shows higher value compared to several previous study where Ferdous et al. [6], Wutisatwongkul et al. [11], Daud et al. [7] and Sibaly & Jeetah [8] at 2.4 kPa.m^2/g , 1.76 kPa.m^2/g , 1.28 kPa.m^2/g and 0.84 kPa.m^2/g respectively.

3.3.4 Folding No (Double Fold)

MD2 pineapple leaf paper produced with untreated pulp has an average of folding no (double fold) of 12.375. This value is compatible with other unbeaten non-wood Soda-AQ paper, which are 3-9 double fold of Semantan Bamboo reported by Mohd Hassan et al. [15]. This low value could be improved by mechanical beating treatment as reported by Mohd Hassan et al. [16] observed that beaten pulp produced better folding endurance.

Table 6: Comparison of physical and mechanical characteristics of MD2 pineapple leaf pulp from various research

	References			
References	**Current study** - Soda Pulping - 20% NaOH charge - AQ charge: 0.1% - L:W of 8:1	**Sibaly & Jeetah (2017)** - Soda Pulping - 15% NaOH charge - AQ charge: 0.1%	**Wutisatwongkul *et al.* (2016)** - Chemical Pulping - 4 wt% NaOH charge	**Daud *et al*. (2015)** - Chemical Pulping - 15% NaOH charge - L: W of 7:1
Screen yield percentage (%)	17.33	-	21.29	-
Freeness (ml)	160	-	-	-
Grammage (gsm)	114.72	60	-	60
Thickness (μm)	220.55	304.0	-	-
Density (g/cm^3)	0.52	-	-	-
Tensile Index (N.m/g)	40.92	6.50	44.13	2.03
Tearing Index (mN.m^2/g)	4.59	-	1.68	7.92
Burst Index (kPa.m^2/g)	2.73	0.84	1.76	1.28
Number of double folds	12.38	-	-	-

3.5 Morphological Analysis

A sheet of MD2 pineapple leaf sample had been taken and used to study the morphology of the paper sample produced using the Scanning Electron Microscopy (SEM). Based on Figure 4.9 (a), long-fibred substance with excellent inter-bonding between the fibre can be observed. It also shows a compact structure with less loose structure smooth surfaces or void. At 500 X magnification shown in (Figure 3b) some fibrillation of fibre which act as a binder to fibres can be spotted. This aligned with a study by Wutisatwongkul et al. [11] where fibrillation of fibre had been identified which resulted on a higher mechanical properties of the paper sample produced. Compactness of individual fibre in a concentrated arrangement and connective material can be seen in (Figure 3c) which contributes to better mechanical strength of the paper sample produced [7].

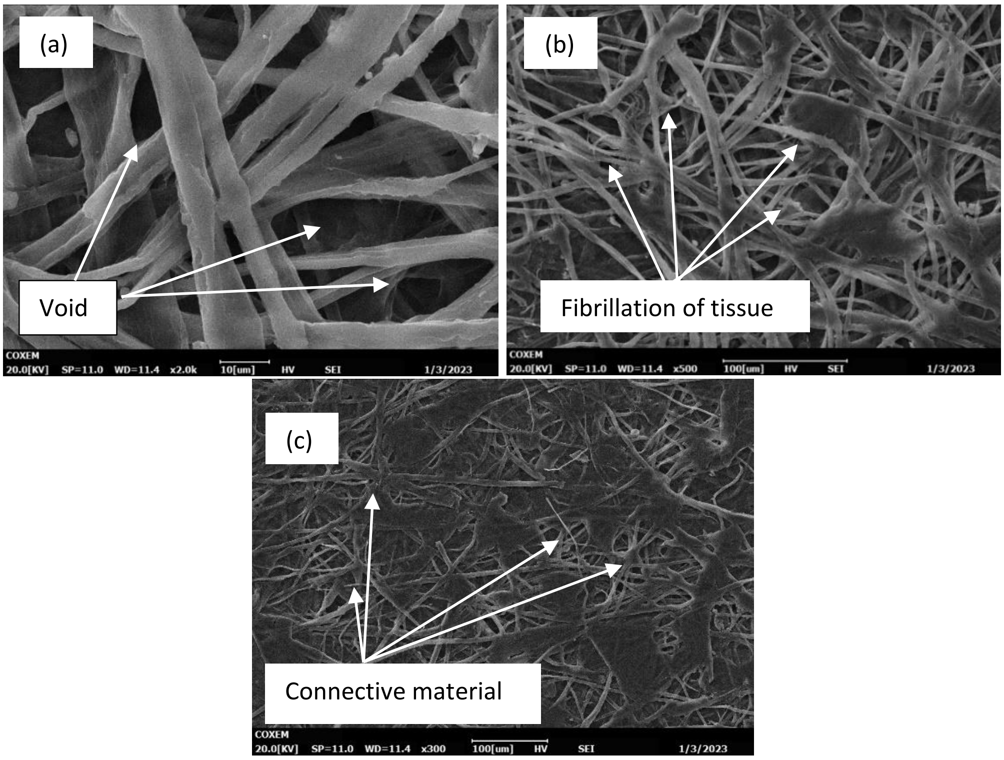

Figure 3: SEM image of MD2 pineapple leaf pulp at (a) 2000 X; (b) 500 X; (c) 300 X magnification

4. Conclusion

In conclusion, the objective of this research had been successfully achieved which is to produce paper pulp by using MD2 pineapple leaf via soda-Anthraquinone (soda-AQ) pulping method. The paper sample produced had achieved a 114.717 g/m^2, which is almost the same from the targeted grammage of 120 g/m^2 paper. Next, the effects of the soda-AQ pulping process on the physical and mechanical characteristics of MD2 pineapple leaf pulp and paper had been successfully determined and discussed in detail in prior chapter. In this research, it was discovered that the pulp moisture content and screen yield were at 76.73 % and 17.33 % respectively. The pulp drainage time and the Canadian Standard Freeness (CSF) were calculated on average of 90.4 seconds and 160 ml. The resulting paper sample physical properties which is the thickness, grammage and bulk density were 220.515 μm, 114.717 g/m^2, and 0.521 g/cm^3 respectively. The mechanical properties of the paper sample which are the tensile index, tear index, burst index and folding no (double fold) yielded 40.919 N.m/g, 4.588 Mn.m^2/g, 2.734 kPa.m^2/g and 12.375 respectively. Last but not least, the Scanning Electron Microscopy (SEM) image showed the compactness of individual fibre in a concentrated arrangement which is unquestionably appropriate for papermaking.

Thus, several ideas and recommendations were proposed to enhance the pulp and paper characteristics made from MD2 pineapple leaf. Further optimization of pulping parameters such as active alkali charge, liquor to material ratio, and the cooking temperature and cooking time during digestion process should be proposed to overcome the low screened yield. Moreover, pulp beating mechanical treatment should be conducted to increase the physical and mechanical characteristics of the virgin MD2 pineapple leaf pulp and paper. In addition, to enhance the quality of the paper, bleaching could also be done to create pulp with better brightness and Kappa number. As a conclusion, MD2 pineapple leaf has shown a promising potential as an alternative agricultural waste material for papermaking application.

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