การใช้เส้นใชนุ่นในการปรับปรุงความแข็งแรงของกระคาษ

นาขอภิพร อภิรักษ์ชัยสกุล

## **สูนย์วิทยทร**ัพยากร'

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาสาสตรมหาบัณฑิต สาขาวิชาเทคโนโลยีทางภาพ ภาควิชาวิทยาสาสตร์ทางภาพถ่ายและเทคโนโลยีทางการพิมพ์ คณะวิทยาสาสตร์ จุฬาลงกรณ์มหาวิทยาลัย ปีการศึกษา 2551 ลิขสิทธิ์ของจุฬาลงกรณ์มหาวิทยาลัย



#### THE USE OF KAPOK FIBER TO IMPROVE STRENGTH OF PAPER

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## สูนย์วิทยทรัพยากร

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งานวิจัยนี้มุ่งศึกษาหาภาวะการผลิตเยื่อจากเส้นใยนุ่นที่เหมาะสมเพื่อใช้ในการผลิตกระดาษ ตัวแปรที่ ้ศึกษาคือ ปริบาณโซเดียบไสดรอกไซด์ที่ใช้ในการต้มเยื่อ และระยะห่างระหว่างจานบดและจำนวนรอบบดในการ บดเยื่อ นอกจากนี้ ขังศึกษาอิทธิพลของเยื่อนุ่นต่อสมบัติกระดาษ เมื่อนำเยื่อนุ่นมาผสมกับเยื่อใยสั้นและเยื่อใย ยาวทางพาณิชย์ การทดลองเริ่มต้นจากนำเส้นใยนุ่นมาแช่น้ำเป็นเวลา 3 สัปดาห์ ผลิตเยื่อด้วยกระบวนการโซดา โดยใช้โซเดียมไฮดรอกไซด์ร้อยละ 10, 15, 20 และ 25 ของน้ำหนักเยื่อแห้ง ที่อุณหภูมิ 120 องศาเซลเซียส เป็น เวลา 2 ชั่วโมง บดเยื่อด้วยเครื่องบดแบบจานบดด้วยระยะระหว่างจานบด 10/1000 นิ้ว จำนวน 2 รอบ ทำ แผ่นกระดาษให้มีน้ำหนักมาตรฐาน 60 กรัมต่อตารางเมตร ทดสอบสมบัติกระดาษ เพื่อหาภาวะการผลิตเยื่อที่ให้ สมบัติด้านกวามแข็งแรงของกระดาษดีที่สุด นำเยื่อที่ผลิตด้วยภาวะนั้นมาบด โดยเปลี่ยนระยะระหว่างจานบด 3 ระดับ คือ 1) 7/1000 นิ้ว จำนวน 1 รอบ 2) 10/1000 นิ้ว จำนวน 1 รอบ ตามด้วย 7/1000 นิ้ว จำนวน 1 รอบ และ 3) 10/1000 นิ้ว จำนวน 2 รอบ หาภาวะการบดเยื่อที่ให้ความแข็งแรงของกระดาษสูงสุด แล้วจึงบดเยื่อตามภาวะ นั้น และนำเยื่อที่ได้มาฟอกโดยใช้ไฮโครเจนเพอร์ออกไซด์ร้อยละ 3 ของน้ำหนักเยื่อแห้ง ที่อุณหภูมิ 80 องศา เซลเซียส เป็นเวลา 2 ชั่วโมง นำเยื่อนุ่นที่ผ่านการฟอกแล้วมาผสมกับเยื่อใยสั้นและเยื่อใยยาวทางพาณิชย์ใน อัตราส่วนต่าง ๆ จากการทดลองพบว่า เมื่อใช้ปริมาณของโซเดียมไฮครอกไซด์เพิ่มขึ้น ทำให้ก่าสภาพระบายได้ ของเยื่อและความต้านทานแรงฉีกของกระดาษลดลง แต่ความหนาแน่น ความเรียบ ความแข็งแรงต่อแรงดึง และ ความแข็งแรงต่อแรงคันทะลุของกระคาษสูงขึ้น ปริมาณโซเดียมไฮครอกไซค์ที่เหมาะสมคือ ร้อยละ 20 ของ น้ำหนักเยื่อแห้ง เพราะให้ความแข็งแรงต่อแรงดึงสูงสุด และระยะระหว่างจานบดเยื่อที่เหมาะสมคือ 10/1000 ้นิ้ว จำนวน 2 รอบ เนื่องจากให้สมบัติกวามแข็งแรงสูงสุด เมื่อนำเยื่อนุ่นมาผสมกับเยื่อทางพาณิชย์พบว่า เยื่อนุ่น ช่วยเพิ่มความแข็งแรงต่อแรงคึง และความแข็งแรงต่อแรงคันทะอุให้แก่กระคาษ แต่จะทำให้ความต้านทานแรง ฉีกของกระคาษลุคลง จากการทุคลองยังพบอีกว่า กระคาษที่ผลิตจากเยื่อนุ่นจะให้ค่ามุมสัมผัสของน้ำสูงกว่า กระดาษที่ผลิตจากเยื่อทางพาณิชย์ ดังนั้น การนำเยื่อนุ่นมาผสมกับเยื่อทางพาณิชย์ช่วยเพิ่มความด้านทานน้ำของ กระดาษได้

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### KEY WORD: KAPOK / SODA PULPING / REFINING / PAPER PROPERTIES / CONTACT ANGLE APIPORN APIRAKCHAISKUL: THE USE OF KAPOK FIBER TO IMPROVE STRENGTH OF PAPER. THESIS PRINCIPAL ADVISOR: PROF. SUDA KIATKAMJORNWONG, Ph.D., THESIS CO-ADVISOR: KUNTINEE SUVARNAKICH, Ph.D., 103 pp.

This research aimed to find the optimal pulping condition of kapok fiber to be used in papermaking. The variables used were pulping condition in terms of sodium hydroxide dosages and refining condition in terms of disc gap distances and numbers of refining pass. The influence of kapok pulp on paper properties was also investigated when kapok pulp was mixed with commercial hardwood and softwood pulps. The experiment was started by immersing kapok fiber in water for 3 weeks. Then, kapok fibers were pulped using a soda process with different sodium hydroxide dosages (10, 15, 20, and 25% based on the oven-dried pulp weight) at 120 °C for 2 h. The kapok pulp was then refined twice by a disc refiner with a 10/1000 inch disc gap. The handsheets having a basis weight of 60 g/m<sup>2</sup> were made, and paper properties were examined to determine the optimum condition providing the best strength properties. The optimum pulping condition was then used in the next step in which the pulp was refined by 3 refining conditions which were 1) 7/1000 inch disc gap for one pass; 2) 10/1000 inch disc gap for one pass followed by 7/1000 inch disc gap for one pass; and 3) 10/1000 inch disc gap for two passes. The optimum refining condition was then used in the next step in which the pulp was bleached using hydrogen peroxide (3% based on the oven-dried pulp weight) at 80 °C for 2 h. The bleached kapok pulp was then mixed with commercial hardwood and softwood pulps at different ratios. The results indicated that higher sodium hydroxide dosage decreased the pulp freeness and tear resistance but increased apparent density, smoothness, tensile strength and burst strength. The optimum dosage of sodium hydroxide was 20% based on the ovendried pulp weight since the highest tensile strength was obtained. The optimum refining condition was the two-pass refining at the 10/1000 inch disc gap because the kapok pulp provided highest strength properties. The kapok pulp in mixed pulps improved tensile strength and burst strength of the sheet but decreased tear resistance. Furthermore, the result shows that the kapok sheet had the higher water contact angle than the sheets from the commercial pulp. Thus, the kapok pulp mixed with the commercial pulp could improve water repellency of the sheet.

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Student's signature. Apipern Apirakchaitkul Principal advisor's signature Ande Kitkamj - y Co-advisor's signature Kinth Sunth

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#### CHAPTER I

#### INTRODUCTION

#### 1.1 Scientific Rationale

Normally fibrous materials for papermaking are from wood. The use of nonwood fibers to replace wood fibers is gaining more popularity. Nonwood fibers are widely available at low cost, especially as waste from agricultural industries. The use of nonwood fibers can also reduce deforestation. Even in the case of farmed trees, cutting down the trees can still affect the eco-system. Some nonwood fibers such as rice straw have short fiber length which can improve paper properties by increasing apparent density of the sheet. Recently, new processes for nonwood pulping and new fibrous raw materials for pulping have been developed.

Kapok trees are cultivated in all areas of Thailand. The plant species is *Ceiba pentandra*. The kapok fiber is a silky fiber that works as a tissue to cover the tree seeds. The kapok fiber is generally used as stuffing for bed, pillow and cushion. Recently, the use of kapok fiber decreased because it was replaced by the synthetic fiber.

Many types of paper are produced from mixed pulp which is softwood and hardwood. Softwood pulp has long fibers which provide paper strength while hardwood pulp has short fibers which offer smoothness and good paper formation. In Thailand, hardwood pulp can be produced locally but softwood pulp must be imported. The kapok fibers are long, so they may be advantageous to substitute softwood pulp.

#### 1.2 Objective of Research Work

The objectives of this research were as follows:

- 1.2.1 To find an optimum condition for pulping the kapok fiber
- 1.2.2 To investigate the roles of kapok fibers in improving paper

properties made from commercial pulps, namely softwood and hardwood pulps mixed with the kapok pulp.

#### 1.3 Scope of the Research Work

Kapok fiber for papermaking was studied. Kapok fiber was pulped by a soda process with varying amounts of sodium hydroxide and mixed with various ratios of a commercial pulp (both softwood and hardwood). In summary, the factors investigated in this research work were as follows:

- 1.3.1 Effect of sodium hydroxide dosages on optical, structural and strength properties of paper
- 1.3.2 Effect of refining conditions on structural and strength properties of paper
- 1.3.3 Potential use of kapok pulp mixed with a commercial pulp

The results obtained from this research could give some technical information for introduction of new nonwood fibrous material for papermaking.

#### 1.4 Contents of Research Work

This thesis consists of 5 chapters including introduction, theoretical background and literature review, experimental, results and discussion, and conclusions and suggestions. Chapter 1 is an introduction of this thesis. Chapter 2 concerns a brief description of kapok fiber, the pulping process, the refining process, the bleaching process, and the short literature review of some previous reports. Chapter 3 contains the details of experimental materials and apparatus, and procedures of the experiment in this research. Chapter 4 is about the results and discussion on optimal refining and pulping conditions, and the influences of kapok pulp on paper properties made from commercial pulps mixed with the kapok pulp. The final chapter presents the conclusion of results obtained from the current experimental work and some suggestions for future study.

#### **CHAPTER II**

#### THEORETICAL BACKGROUND AND LITERATURE REVIEWS

#### 2.1 Theoretical Background

#### 2.1.1 Fiber

Paper has traditionally been defined as a felted sheet formed on a fine screen from a water suspension of fibers. The chemical composition of plant fibers consists of 4 classes of materials.

#### 2.1.1.1 Cellulose

Cellulose is the substance that determines the character of the fiber and permits its use in papermaking. Cellulose is a carbohydrate, meaning that it is composed of carbon, hydrogen and oxygen, with the latter two elements in the same proportion as in water. Cellulose is also a polysaccharide, indicating that it contains many sugar units.

The chemical formula for cellulose is  $(C_6H_{10}O_5)_n$ , where n is the number of repeating sugar units or the degree of polymerization (DP). Cellulose has an average DP of at least 9,000 – 10,000 and possibly as high as 15,000. An average DP of 10,000 would correspond to a linear chain length of approximately 5 micrometers in fibers [1]. Figure 2-1 shows a partial structure of cellulose.



Figure 2-1 Partial structure of cellulose [2].

The properties of cellulosic materials are related to the DP of the constituent cellulose molecules. Decreasing the average chain length or molecular weight (e.g., by chemical action) below a certain level will cause deterioration in fiber strength. One of the main objectives during pulping and bleaching operations is to minimize peeling reactions (in which individual glucose unit is split off at the end of the cellulose chain) and chain cleavage reactions (in which the chain is broken somewhere along its length).

#### 2.1.1.2 Hemicellulose

In general, the hemicellulose consists of polysaccharide polymer with a lower DP than cellulose (average DP of 100-200) and contains mainly the sugars D-glucose, D-mannose, D-galactose, D-xylose and L-arabinose (see Figure 2-2) [3]. Like cellulose, hemicellulose functions as supporting material in the cell walls.



Figure 2-2 The sugar moieties of wood hemicelluloses [2].

During chemical treatment to produce pulp, the amounts, locations, and structures of the various hemicelluloses usually change dramatically. Generally, hemicellulose is more easily degraded and dissolved than cellulose. The detailed structures of hemicelluloses have not been determined. Only the ratios of sugars that these polysaccharides contain have been studied.

#### 2.1.1.3 Lignin

Lignin is amorphous, highly complex, and mainly composed of aromatic polymers of phenyl propane units that are considered to be an encrusting substance. The chemistry of lignin is extremely complex [4], as shown in Figure 2-3. The structure consists primarily of phenyl propane units linked together in three dimensions. The three linkages between the propane side chains and the benzene rings are broken during chemical pulping operations to free the cellulosic fibers.



Figure 2-3 The structure of lignin [5].

#### 2.1.1.4 Extractives

Extractives are chemicals in the wood or fibers that can be extracted using solvents. In some cases, the extractives are classified by solvent used to extract them; such as, water-soluble or ethanol-soluble extractives. The extractives are a group of cell wall chemicals mainly consisting of fats, fatty acids, phenols, terpenes, resin, waxes, and many other minor organic compounds. The composition of extractives varies widely from species to species, and the total amount of extractives in a given species depends on growth conditions [2].

#### 2.1.2 Kapok Fibers



Figure 2-4 Kapok fruit containing kapok fiber [6].

The Kapok fiber can be obtained from the fruit of kapok tree (see Figure 2-4) which is cultivated in all area of Thailand. Kapok can be classified as follows:

Kingdom	Plantae
Division	Magnoliophyta
Class	Magnoliopsida
Order	Malvales
Family	Malvaceae
Genus	Ceiba
Species	C. pentandra

Table 2-1 Scientific classification of kapok tree [7]

Kapok tree can produce its fruit at the age of about 2 - 4 years. The kapok flower does not bloom simultaneously; therefore the fruit does not ripe at the same time. Too young kapok fruit gives low quality fiber. A kapok tree produces 300 - 500 fruits per annual. One kilogram of fibers can be obtained from 120 - 150 fruits. One fruit is composed of 44% peel, 32% seed, 7% spin and 17% fiber [8].

The kapok fiber is fluffy, light-weighted and inelastic to be spun. It's typically composed of 64% cellulose, 13% lignin and 23% pentosan [9]. Besides these constituents, they also contain waxy cutin on the fiber surface which makes them

water repellency. Except stuffing bed pillow and cushion, the kapok fiber can be used as a fuel and oil absorption.



Figure 2-5 The electron microscopic image of kapok fiber [10].

The kapok fiber has a hollow structure (see Figure 2-5) with thin fiber wall. The diameter including fiber wall is  $16.5 \pm 2.4$  micrometers. The lumen is  $14.5 \pm 2.4$  micrometers and the fiber length is 25 - 30 millimeters [10]. This structure is good for papermaking to improve paper strength and density.

#### 2.1.3 Pulping

Pulping represents the process by which wood or other lignocellulosic material is reduced to fibrous mass, denoted as pulp. The process of defibration can be accomplished mechanically, chemically, or by a combination of both treatments. The corresponding commercial processes are then classified as mechanical, chemical and semi-chemical pulping. Pulps produced in different ways have different properties that make them suited to particular products.

#### 2.1.3.1 Mechanical Pulping

The oldest and still a major method of mechanical pulping is the groundwood process, where a block of wood is pressed lengthwise against a wetted, roughened grinding stone. Fibers are removed from the wood, abraded and washed away from the stone surface with water [11].

A more highly developed technique in mechanical pulping then groundwood process involves shredding and defibering chips of wood between the rotating discs of a device called a refiner; the product is known as refiner mechanical pulp (RMP). RMP typically retains more long fibers than stone groundwood and yields stronger paper. However, if refining is done at high temperature, the pulp is then called thermomechanical pulp (TMP).

Mechanical pulping processes have the advantage of converting up to 95% of the dry weight of the wood into pulp, but require prodigious amounts of energy to accomplish this objective. Containing a large amount of short fibers, the mechanical pulp forms a highly opaque paper with good printing properties but the sheet is weak. In addition to that discolors easily when it is exposed to light due to its high lignin content.

#### 2.1.3.2 Chemical Pulping

In chemical pulping, the wood chips are cooked with appropriate chemicals in an aqueous solution at elevated temperature and pressure. The objective is to degrade and dissolve away the lignin and leave behind most of the cellulose and hemicellulose in the form of intact fibers. In practice, chemical pulping methods are successful in removing most of the lignin; however, they also degrade and dissolve a certain amount of the hemicellulose and cellulose so that the yield of pulp is low relative to mechanical pulping methods, usually between 40% and 50% of the original wood substance.

#### **Kraft Process**

The main active chemical agents in the kraft process are hydroxide and hydrosulfide anions which are present in the kraft cooking liquor, an aqueous solution of caustic sodium hydroxide and sodium sulfide, denoted as white liquor. The hydrosulfide ion plays an important role in kraft pulping by accelerating delignification and rendering nonselective soda cooking into a selective delignifying process. Delignification can be divided into three phase, namely the initial, bulk, and residual. In the initial phase, delignification is caused by the cleavage of alpha-aryl and beta-aryl ether bonds in the phenolic units of lignin which account for approximately 15-25% of native lignin. In this stage, the predominant part of the total carbohydrate loss can be observed. In the bulk delignification phase, the main part of the lignin is removed while at the same time only minor carbohydrate loss occurs. The cleavage of beta-aryl bonds in nonphenolic units of lignin is assumed to be the main delignification reaction. In the residual delignification phase, only approximately 10-15% of the native lignin is removed. However, with continuous delignification, the dissolution of carbohydrates extensively increases. In order to maintain high yields and to preserve a sufficiently high quality of the pulp, delignification is limited to a certain degree of delignification.

The following benefits over the sulfite process can be claimed that all wood species can be used as raw material. The kraft process is relatively insensitive to bark. The pulp is much stronger than sulfite pulp, and the main advantage of kraft pulping is chemical recovery. Nonetheless, kraft pulping caused emissions of malodorous gases. The pulp was also much darker than sulfite pulp but it can be improved by bleaching.

Soda process is the origin of kraft process. Only sodium hydroxide (NaOH) is used as a cooking liquor to dissolve the lignin. Although the soda process was replaced by kraft process, it is still used in several mills. [12].

#### **Chemical Reaction**

Alkaline darkening is occurs during kraft delignification. The color of unbleached pulps is caused by certain unsaturated structures (Chromophores). In addition, leucochromophores, which can be converted into chromophores by air oxidation, may be present in the pulp. Most of the chromophores are presumed to be derived from lignin [3].

Peeling reaction removes the terminal anhydro-sugar unit, generating a new reducing end group until a competitive stopping reaction sets in, forming a stable saccharinic acid end group. The elimination of the cellulose chain in  $\beta$ -position to the anionic intermediate leads to a dicarbonyl structure in the leaving unit, which is extremely unstable under alkaline condition and undergoes various degradation reactions. Depending on the reaction condition, about 50-60 glucose units are peeled off. If keto or aldehyde groups are present along the cellulose chain, they generally cause cleavage of the glycosidic bond in alkaline media by  $\beta$ -alkoxy elimination. Therefore, oxidized groups (C2, C3 and C6 position) are considered as "weak links" along the chain. Keto groups at C2 or an aldehyde at C6 and the anomeric carbon are referred to as "active carbonyls", leading to a new reducing end group, which might undergo further degradation reactions. A keto group at C3 is considered as an "inactive carbonyl", since  $\beta$ -elimination eventually forms a non-reducing end group and a stable acid [12].

#### 2.1.4 Refining

Refining or beating of chemical pulps is the mechanical treatment and modification of fibers so that they can be formed into paper or board of the desired properties. The main target of refining is to improve the bonding ability of the fibers so that they form strong and smooth paper sheet with good printing properties. Sometimes the purpose is to shorten too long fibers for a good sheet formation or to develop other pulp properties such as absorbency, porosity, or optical properties specifically for a given paper grade. Figure 2-6 shows two types of refiner.



Disc refiner

Figure 2-6 Two types of refiner [11].

The most commonly used refining or beating method is to treat fibers in the presence of water with metallic bars. The plates are grooved so that the bars that treat fibers and the grooves between bars allow transportation through the refining machine. The initial action is to partially remove the thin primary wall. Although the primary wall is permeable to water, it does not swell and prevents the fiber from swelling. Removal of the primary wall exposes the secondary wall and allows water to be absorbed into the molecular structure. The consequent loosening of the internal structure promotes fiber swelling and renders the fibers soft and flexible. This so-called "internal fibrillation" is generally regarded as the most importance primary effect of refining following removal of the primary wall. The further action of fibrillation involves loosening of the fibrils and rising of the finer microfibrils on the surfaces of the fibers, resulting in a very large increase in surface area for the beaten fibers. As the fibers become more flexible, the cell walls collapse into the lumens, thus creating ribbon-like elements of great conformability.

Fiber shortening (or cutting) always occurs to some extent during refining, mainly due to the decreased gap. Fiber cutting is often considered undesirable because it contributes to slower drainage and loss of strength. But, in some applications, a cutting action may be promoted to obtain good sheet formation from a long-fibered pulp furnish or to control sheet drainage on the paper machine. Refining also produces fines consisting of fragments of broken fibers and particles removed from the fiber walls. One obvious effect of refining is the dramatic change in the drainage or dewatering properties of the pulp. Pulp drainability is rapidly reduced as refining proceeds, mainly due to the increased concentration of fines [13].

#### 2.1.5 Bleaching

Cellulose and hemicellulose are inherently white and do not contribute to pulp color. It is generally agreed that "chromophoric groups" on the lignin are principally responsible for color; oxidative mechanisms are believed to convert part of the phenolic groups in lignin to quinone-like substances that are known to absorb light. Heavy metal ions (e.g., iron and copper) are also known to form colored complexes with the phenolic groups. Bleaching is defined as a chemical process aimed to remove color in pulps derived from residual lignin or other colored impurities while maintains cellulose chain length and brightens the fibers. Two approaches are used in the chemical bleaching of pulps. One approach is to utilize chemicals that selectively destroy some of the chromophoric groups, but do not materially attack lignin. The other approach is to almost totally remove residual lignin.

Peroxide is used in the bleaching of both mechanical and chemical pulps. When applied at moderate temperature up to 60 °C, peroxide is an effective ligninpreserving bleaching agent for mechanical pulp. At slightly higher temperature (i.e., 70-80 °C), peroxide is used as a part of a chemical pulp full bleach sequence to provide increments of brightness and to improve brightness stability. Peroxide bleaching is strongly affected by pH, which must be adjusted and buffered at about 10.5 for best results. At pH levels above 10.5, competition from undesirable side reactions detracts from the bleaching action. The pH is usually controlled by addition of sodium silicate. The silicate acts as both a stabilizer and a buffering agent in the peroxide bleaching system. Although higher temperature accelerates peroxide bleaching reactions, undesirable side reactions can gain greater momentum. The stability of the peroxide is also adversely affected at higher temperature, especially in the presence of heavy metal ions. Chelating agents (usually EDTA or DTPA) are commonly used in the peroxide stage to prevent metals such as manganese, copper and iron from decomposing peroxide [14]. From an environmental standpoint, bleaching without the use of any chlorine-containing chemicals is a desirable option. Bleaching systems using hydrogen peroxide are being used by a few mills with apparent success. The chemical cost is generally higher, but these "environmentally friendly" pulps often sell for a premium price.

#### 2.1.6 Pulp and Paper Properties

#### 2.1.6.1 Dewatering Properties of Pulps

Characterization of pulp slurries often involves measuring the drainage resistance. The most common methods are the Canadian Standard Freeness (CSF) and Schopper-Riegler (SR). Both methods are a relative measure of the drainability of a pulp suspension.

CSF measurement involves filtering 1 L of diluted pulp suspension with a consistency of 3 g/L freely through the screen plate of the testing device in Figure 2-7. Faster slowing of draining due to fiber mat accumulation on the screen plate gives a smaller CSF number. CSF is the amount of water passing through the side orifice of the tester. The measurement principle of SR number is the same as CSF. In SR testing, 1 L of pulp suspension with a consistency of 2 g/L filters through the wire of the apparatus. More rapid slowing of drainage gives a higher SR number. This means that SR number is directly proportional to the drainage resistance of the stock but inversely proportional to CSF number [15].



Figure 2-7 Schematic illustration of CSF-tester [15].

#### 2.1.6.2 Fiber Length

Various optical methods are presently available for measurement of fiber length. The primary principle is to allow a dilute pulp suspension flow through a cuvette. A light source illuminates the flow for detection of fibers with a camera. The image analysis technique provides the characteristic values for the fibers. Fiber quality analyzer (FQA) shown in Figure 2-8 detects fiber deformation. The patented flow cell orients the fibers in the middle of the flow to prevent the fibers from blocking the flow cell. The unit uses a charge-coupled device (CCD) camera and a polarized light source. The measurement gives fiber length distribution, fiber curl, mean kink angle, and kink index.



Figure 2-8 The Fiber Quality Analyzer [16].

#### 2.1.6.3 Basis Weight or Grammage

To determine the grammage of a sample, one can measure its weight in grams and its area in meters. If a gram balance and metric cutter are not available, one can convert from pounds and inches to grams and meters. Dividing weight by area gives grammage. Since there is basis weight variation within paper, using a small sample for this test should be avoided.

#### 2.1.6.4 Thickness

Thickness, or "caliper" as it is sometimes called, is another property that is always specified for a grade of paper. It is defined as the perpendicular distance between the two principal surface of paper and paperboard under specified conditions. The millimeter is the standard thickness unit in the rest of the world. In the SI unit thickness is presented in micrometers.



Figure 2-9 A typical micrometer used for measuring the thickness of paper [17].

Many types of micrometers have been used for measuring paper thickness, with the most common type in use today probably being the instrument shown in Figure 2-9. In this instrument, the jaws continuously cycle between their separated and closed positions. The operator inserts a paper sample between the jaws while they are separated and then reads the thickness from the dial when the jaws are closed.

#### 2.1.6.5 Apparent Density

Dividing the grammage by the thickness in micrometers gives the apparent density of paper in g/cm<sup>3</sup> [18]. Typical values of apparent density range from 0.5 in bulky papers to 0.75 for highly bonded sheets. Some papers such as glassine have densities of 1.0 or greater. Since cellulose has a density of 1.5, this means that most papers are more than 50% air. Apparent density is one of the most significant properties of paper. It influences almost all mechanical, physical, and electrical properties, as well as many other properties such as absorption and printability.

## 2.1.6.6 Smoothness

There are various methods to testing smoothness such as optical method, friction method, profile measurement, optical contact area, ink contact area and air flow measurement. The air flow method has been the most popular, especially for quality control. These instruments either measure the time required for a given volume of air to flow between the paper surface and an optically flat surface pressed against it, or they measure the flow rate of the same air.



Figure 2-10 Bekk Smoothness Tester [19].

The Bekk smoothness tester is shown in Figure 2-10. In this instrument, the time is measured for vacuum to decrease from 380 mm Hg to 360 mm Hg due to the passage of 10 cm<sup>3</sup> of air from the room past the interface between the lower paper surface and polished glass head. A rubber backing prevents the flow of air through the paper specimen [20].

#### 2.1.6.7 Brightness

In the paper industry, a special process is used to characterize the brightness because this is one of the most important optical properties of paper. The brightness is based on a measurement of the reflectance by paper at a single wavelength, 457 nm. The brightness test was designed primarily as a test to measure the effectiveness of bleaching in removing yellowness from pulps. It is expressed as a percentage of the brightness of a white standard [21].

#### 2.1.6.8 Opacity

Opacity is determined by the amount of light transmitted by a paper. If all of the light is transmitted and none is reflected or absorbed, the opacity will be zero. If no light is transmitted and all of it is reflected or absorbed, the opacity will be 100%. Although opacity can be measured by the amount of transmitted light, it is usually measured as a contrast ratio. This is the ratio of the diffused reflectance from a single sheet of paper backed by a black body to the reflectance of that same spot backed by an opaque [22]. It might seem incorrect to measure opacity by measuring reflectance, when opacity is determined by transmitted light. However, contrast ratio is an indirect measurement of light transmission. If light which has been transmitted through a sheet of paper strikes a white body on the other side of the sheet, it will be reflected back into the sheet and part of it will emerge as additional reflected light. However, if the transmitted light strikes a black body, it will be absorbed and there will be no addition to the reflected beam. The difference between these two reflectances is dependent upon the amount of light transmitted through the sheet.

#### 2.1.6.9 Tensile Strength

Tensile strength is a very useful property to describe the general strength of any material. It is a direct indication of the durability and potential end use performance of a number of papers that receive direct tensile stresses in use, such as wrapping, bag and printing papers. In general, a certain minimum tensile strength is required of any paper that undergoes a web converting operating where it is subjected to tensile stresses while being pulled through the process. Printing papers are a primary example of this.

Tensile strength is the maximum force per unit width that a paper strip can resist before breaking when applying the load in a direction parallel to the length of the strip. In the tensile strength tester, the test piece is stretched to the point where rupture occurs. The maximum tensile force that the test piece can withstand before it breaks and the corresponding elongation of the strip are measured and recorded. Tensile strength expression uses kN/m.

Tensile strength of a paper depends on fiber strength but primarily on the degree of bonding between fibers. The result obtained also depends on the testing conditions. An increase in the rate of loading will increase the tensile strength. An increase in moisture content of the paper will decrease the tensile strength while increasing elongation. In addition, it is highly dependent on directionality of the paper. The tensile strength measured in different directions of the sheet is often used as an indicator of fiber orientation [20].

#### 2.1.6.10 Stretch or Elongation

The elongation or stretch at break is the increase in length of the strip to its breaking point expressed in percentage of the original length. The tensile testers are designed to measure the stretch of a test specimen in the course of measuring tensile breaking strength [15].



Figure 2-11 Stress-strain curve [15].

The maximum load and elongation values are plotted and then calls stress-strain curve (see Figure 2-11). The area under the stress-strain curve which is named tensile energy absorption or TEA is proportional to the energy that the paper can absorb up to the breaking point. For a given tensile breaking strength, then, increasing the stretching ability of a paper will increase its TEA. In fact, in certain cases it may be advantageous to increase the stretch even if the tensile strength falls off.

#### 2.1.6.11 Internal Tearing Resistance

Tearing strength has long been widely used as a mill control test because, in some ways, it reflects the general mature of the fibers present in the paper. The internal tearing resistance or tearing strength is the mean force required to continue the tearing of paper from an initial cut in a single sheet. If this cut is in the machine direction, the result is machine direction tearing resistance. Correspondingly, the cross direction tearing resistance is the result of a test in the cross direction. The tearing strength is highly dependent on the fiber orientation of the sheet. Figure 2-12 shows the principle of tear testing.



Figure 2-12 Principle of tear testing [15].

The most common instrument used for the measurement of internal tearing resistance is the Elmendrof tear tester. This is a physical pendulum that applies the tearing force by moving the pendulum in a plane perpendicular to the initial plane of the test piece. The work done in tearing the test piece is measured by the loss in potential energy of the pendulum. The unit for tearing strength is Newton (N) or millinewton (mN) [20].

The tearing strength of a paper depends on fiber length, fiber strength, degree of bonding between fibers, degree of orientation of fibers in the paper. Longer and stronger fibers provide higher tearing strength. At low levels of bonding, the tearing strength grows with an increasing bonding. At higher levels of bonding degree, the fiber strength determines the level of tearing strength.

#### 2.1.6.12 Bursting Strength

Bursting strength is an old test for paper strength. It is a rapid and easy test to perform and does not require test pieces cut exactly. Furthermore, it cannot be measured as a function of the directionality of the paper. The burst test has been developed empirically. It is not clearly defined in physical terms. Bursting strength somehow relates mathematically and physically to the tensile strength of paper. Bursting strength is the maximum pressure that the paper can resist without breaking with pressure applied perpendicular to the plane of the test piece. The most common tester used for bursting strength measurements is the Mullen tester. A test piece placed over a circular elastic diaphragm is rigidly clamped at the periphery but free to bulge with the diaphragm. The hydraulic fluid pressure increases by pumping at a constant rate to bulge the diaphragm until the test piece ruptures. The bursting strength of the test piece is the maximum value of the applied hydraulic pressure. The unit for bursting strength is kilopascal, kPa [20].

#### 2.1.6.13 Zero-Span Tensile Strength

A special case of tensile strength testing with a span length can be as close to zero as possible. This test indicates the strength of the individual fibers [23] rather than the strength of the paper itself. Normal tensile testing indicates the combined effect of the strength of the fibers, their bonding, and variation in the structure of paper. The zero-span test provides a possibility to measure the fiber strength separated from other effects.

The zero-span tensile test can use a tensile tester equipped with special clamps to bring the span length as close to zero as possible. Special testers built specifically for zero-span testing are also available. One such instrument is the Pulmac tester. The tensile strength values obtained with zero-span testing are very sensitive to variations in the 0 - 1 mm span length. The assumption of the influence of the fiber bonding being eliminated from the test results is valid only if the span is really zero.

The zero-span tensile strength value is a good indicator of the average strength of individual fibers. It is therefore a useful test to indicate the effect of pulping processes, beating, etc., on the fiber strength. Differences between fibers from different wood species are also detectable. The test therefore has primary use for pulp testing. In this context, the test often uses both dry and rewetted laboratory sheets that rewetting eliminates the effect of fiber bonding on the results.
#### 2.1.6.14 Contact Angle Analysis

The hydrophobicity/hydrophilicity of the sheet is defined by contact angle of the sheet. There are two approaches for contact angle analysis: static and dynamic contact angle measurement. In static method, the liquid-solid angle of a sessile drop of liquid on the surface of a specimen is observed and measured by means of contact angle meter. The contact angle ( $\theta$ ) of a drop of liquid on the surface of a specimen is shown in Figure 2-13. The cosine of the contact angle (Cos  $\theta$ ) is related to the surface energy of the specimen by the Young equation [1].



Figure 2-13 Schematic diagram of the contact angle at the solid-liquid interface.

For paper testing, static measurement is not an accurate way to measure contact angle due to the structure of paper which is porous and rough. Dynamic method is generally used. In dynamic method, the contact angle is measured from the time of contact until specific time. The results are plot between contact angles versus times.

Many contact angle measurement used fluid droplet method, the image of the fluid droplets can be obtained as they impinge upon solid surface. Various recoding techniques can be employed, including high framing rate photography and video recoding, direct digitization of a video signal, or even real-time digital analysis. High-speed movies or photos can be analyzed by hand or automatically analyzed by computer. Different types of analysis have been employed, from measuring the contact angle and volume of droplets deposited with near zero momenta, to measurements of the maximum spreading of droplets impinging on surfaces with well-controlled momenta. The particular advantage of this type of measurement is that the dynamic interactions (wetting, imbibition, elastic deformation) between fluids and surfaces can be characterized on time scales.

#### 2.2 Literature Reviews

Wongsaisuwan *et al.* [24] studied the pulpability of kapok using a soda pulping process. Kapok fibers were pulped using a liquor-to-wood ratio (L: W) equals to 29:1 for 2 h with the pulping temperature of 100 °C. Then, the pulp was shredded by a blender before being bleached with hydrogen peroxide. The variables in this study were sodium hydroxide dosage (10% and 15% based on oven-dried pulp weight), pulp shredding time (10 and 15 min.) and hydrogen peroxide dosage (1% and 3% based on oven-dried pulp weight). The results showed that higher dosage of sodium hydroxide provides stronger pulp with higher brightness but lower freeness as compared to lower dosage. Pulp becomes brighter with higher dosage of hydrogen peroxide while longer pulp shredding time gives the weaker pulp with lower freeness.

Malab et al. [25] blended 75% kapok fibers with 25% bamboo pulp, then treated the mixture with detergent and sodium hydroxide to produce paper that was light, pleasingly warm and naturally off-white in color. The combination of the two non-wood species would even reduce the volume of bamboo pulp, chemical and energy uses without adversely affecting the strength properties.

Hori *et al.* [26] examined the oil absorption ability of kapok fiber. It was found that kapok fiber is significantly hydrophobic and does not get wet with water. Thus, the absorptivity of oil was tested. The fiber selectively absorbed significant amounts of oil (40 g/g of fiber) from oil suspension in freshwater.

Lim and Huang [27] studied the use of kapok fiber to absorb oil. It was found that kapok fiber has a hollow structure with large lumen. They compared the performance with polypropylene (PP), a widely used commercial oil sorbent for oil spill cleanup. The oils investigated were diesel, hydraulic oil (AWS46), and engine oil (HD40). The result showed that the kapok fiber had significantly higher sorption capacities for the three oils than PP. The hydrophobic–oleophilic characteristics of the kapok fiber could be attributed to its waxy surface, while its large lumen contributed to its excellent oil absorbency and retention capacity.

# CHAPTER III

# EXPERIMENTAL

#### **3.1 Materials**

- 3.1.1 Sodium Hydroxide: NaOH, pure pellets from Merck KGaA Corporation, Germany
- 3.1.2 Sodium Silicate Neutral Solution: Na<sub>2</sub>SiO<sub>3</sub>, laboratory grade from Panreac Quimica Corporation, Spain
- 3.1.3 Hydrogen peroxide: H<sub>2</sub>O<sub>2</sub>, liquid, analytical grade from POCH Corporation, Poland
- 3.1.4 Potassium Permanganate: KMnO<sub>4</sub> , liquid, analytical grade from Merck KGaA Corporation, Germany
- 3.1.5 Potassium iodide: KI, analytical grade from Merck KGaA Corporation, Germany
- 3.1.6 Sodium thiosulfate solution: Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, liquid, analytical grade from Merck KGaA Corporation, Germany
- 3.1.7 Kapok Fibers (generally available in Thailand)
- 3.1.8 Softwood pulp (Crofton Pulp & Paper Mill, Canada)
- 3.1.9 Hardwood pulp (Phoenix Pulp & Paper Public Co., Ltd, Thailand)

## **3.2 Apparatus**

- 3.2.1 Autoclave digester: UEC-2017A (Universal Engineering Corporation, India)
- 3.2.2 Moisture balance: FD-600 (Kett Electric Laboratory Corporation, Japan)
- 3.2.3 Disc refiner : Sprout-Waldron Bulletin 1480 (Koppers Company, USA)
- 3.2.4 Valley beater: UEC-2018A (Universal Engineering Corporation, India)

- 3.2.5 Disintegrator: Formax T-100 (Adirondack Machine Corporation, USA)
- 3.2.6 Freeness tester: Regmed CF/A (Regmed Corporation, Brazil)
- 3.2.7 Fiber Length Measurement: Fiber Quality Analyzer (Optest Equipment Inc., Canada)
- 3.2.8 Sheet former: RK-2A KWT (Paper Testing Instruments, Austria)
- 3.2.9 Smoothness tester: Digi-Bekk (Toyo Seiki Co., Ltd, Japan)
- 3.2.10 Optical tester: Color-Touch PC (Technidyne Corporation, USA)
- 3.2.11 Tensile strength tester: Strograph E-S (Toyo Seiki Co., Ltd, Japan)
- 3.2.12 Tear resistance tester: Protear (Thwing-Albert Instrument Company, USA)
- 3.2.13 Burst strength tester : SE002P (Lorentzen & Wettre Corporation, Sweden)
- 3.2.14 Contact angle measurement : Pocket goniometer PG-3 (Fibro System Corporation, Sweden)
- 3.2.15 Zero span tensile strength tester : Pendulum Tensile Strength Tester with special clamps adjustable to zero (Toyo Seiki Co., Ltd, Japan)
- 3.2.16 Scanning Electron Microscope (SEM) : JSM-5410LN (JOEL Corporation Ltd., Japan)

# **3.3 Experimental Plan**

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The experiment was divided into 3 phases. In phase I, the effect of degree of delignification i.e. NaOH dosages on the strength properties of paper was studied. The optimum dosage was determined to be used in phase II which concerned with the influence of refining in terms of disc gap and number of passes on the strength properties of paper; and phase III involved the potential use of kapok pulp mixed with the commercial softwood and hardwood pulp as a new type of paper. The flow charts in Figures 3-1, 3-2, and 3-3 demonstrate the experimental plan for phase I, phase II, and phase III, respectively.



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Figure 3-1 Phase I - Effects of sodium hydroxide dosages employed in pulping.



Figure 3-2 Phase II - Effects of disc gaps and numbers of passes during refining.



Figure 3-3 Phase III - Effect of kapok pulp mixed with the commercial pulps on paper properties.

\*The kapok pulp was added to the commercial pulps in the ratios of 0:100, 25:75, 50:50, 75:25, and 100:0 (kapok pulp: commercial pulp)

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#### 3.4 Procedure

#### 3.4.1 Sample Preparation

The kapok fibers were immersed in water for 3 weeks. It was then squeezed out the excess water and cut to 3-5 cm long pieces. The moisture content of the pulp was determined using the moisture balance (FD-600).

#### 3.4.2 Pulping Process

The kapok fibers were pulped using a soda process in which sodium hydroxide (NaOH) was used as a pulping chemical to react with the fibers. The amounts of sodium hydroxide used in this experiment were 10%, 15%, 20% and 25% based on the oven-dried pulp weight. The liquor-to-wood ratio of the experiment was 17:1. The kapok fibers were packed into stainless steel containers along with the cooking liquor. The pulping was done using an autoclave digester (UEC-2017A) that an initial temperature was set at 40 °C. Then the digester was heated to 120 °C within 30 min and the pulping was proceeded at this temperature for another 120 min. When the pulping was completed, the pulp samples were washed with water. The moisture content of the pulp was determined to calculate the pulp yield.

#### 3.4.3 Refining Process

The pulp from the pulping process was refined by the disc refiner (Sprout-Waldron Bulletin 1480) using 12 inch disc size at 1500 rpm. The refining conditions used in this experiment were 7/1000 inch disc gap for one pass refining, 10/1000 inch disc gap for one pass refining followed by 7/1000 inch disc gap for one pass refining and 10/1000 inch disc gap for two passes refining. The refining was run at about 5% pulp consistency. After the pulping and refining, the excess water was removed for easy storage.

#### 3.4.4 Bleaching Process

The pulp was bleached using hydrogen peroxide at the dosage of 3% based on the oven-dried pulp weight at 1.5% pulp consistency (only the pulp for phase III was bleached). The bleaching temperature was 80 °C at 2 h. Sodium silicate was used as the buffer and stabilizer.

# 3.4.5 Stock Preparation

The kapok pulp was disintegrated using a disintegrator (Formax T-100) for 100,000 revolutions in hot water (80 °C) to do latency removal. In phases I and II, the unbleached kapok pulp was used as it was, while in phase III the bleached kapok pulp was mixed with a commercial hardwood pulp, softwood pulp and mixed pulp (consisted of 25% softwood pulp and 75% hardwood pulp). The kapok pulp was added to the commercial pulps in the ratios of 0:100, 25:75, 50:50, 75:25, and 100:0. Each commercial pulp was beaten in a valley beater (UEC-2018A) in which 360 g of the oven-dried pulp in 23 L of water was accommodated according to TAPPI Standard method T-200. The softwood pulp and hardwood pulp were beaten to meet the Canadian Standard Freeness of 350 mL and 300 mL respectively.

# 3.4.6 Papermaking Process

The pulps from the stock preparation were weighed to obtain adequate material for making handsheets with a basis weight of 60 g/m<sup>2</sup>. The handsheets were made using Rapid-Köthen sheet former (Paper Testing Instruments) according to ISO5269-2. The drying time for the handsheets in the machine was 8 min. The total of 6 handsheets per test condition was prepared for investigating the optical and physical properties.

# 3.4.7 Pulp and Paper Testing

# 3.4.7.1 Kappa Number of Pulp

The Kappa number of pulp is the volume (in milliliters) of 0.1N potassium permanganate solution consumed by one gram of moisture-free pulp (TAPPI T-236). The pulp was weighed and disintegrated in distilled water at 25 °C. During the disintegration, 100 mL of 0.1N potassium permanganate solution and 100 mL of 4.0N sulfuric acid solution were added to the disintegrated specimens and a stopwatch was started. At the end of the exact 10.0 min, the reaction was stopped by adding 20 mL of 16.6% potassium iodide solution. After the mixing, the free iodine

was titrated with the 0.1N sodium thiosulfate solution and starch solution was used as an indicator. To determine the Kappa number, the blank test using exactly the same method but omitting the pulp was done.

### 3.4.7.2 Residual Alkali

The residual alkali in the black liquor is an excess alkali that the pulp could not be used to react with lignin and extractives. The method determined a residual alkali by taking 50 mL of the black liquor from the pulping process and 100 mL of 20% barium chloride solution was then added. Then, distilled water was added until the total solution was 500 mL. The solution was set aside until precipitation took place and then the precipitate was filtered; and 100 mL of the transparent liquid was then extracted with 100 mL of distilled water. The extracted solution was titrated with 0.1N hydrochloric acid until the pH reached 4.5 and the residual alkali was then calculated.

## 3.4.7.3 Canadian Standard Freeness (CSF)

Canadian standard freeness determines the rate of drainage of a dilute pulp suspension and expresses it in terms of freeness. According to TAPPI T-227, 3 g oven-dried pulp sample was weighed and diluted to 1 L. To calculate the freeness, the stock temperature and consistency of the pulp are accurately measured to obtain the correct result.

## 3.4.7.4 Conditioning of Sample

Before testing paper properties, the handsheet was conditioned in a room having 65% relative humidity and temperature is strictly controlled at 27 °C for 24 h (for more detail, consult ISO 187).

#### 3.4.7.5 Apparent Density

The apparent density was calculated from caliper and basis weight of the sheet. The caliper was determined using a micrometer. The results were from the average of ten measurements. The caliper in micrometer was divided by grammage to give the apparent density.

## 3.4.7.6 Smoothness (Bekk method)

The Bekk smoothness was measured by following TAPPI T-479. The specimen was clamped under a clamping pressure of approximately 100 kPa. The vacuum pump raised the column to 380 mm in a mercury scale. As soon as the mercury dropped to 380 mm, the stopwatch was started to run until the mercury passed through 360 mm and the stopwatch was stopped. The smoothness was measured at 5 areas per one side of the handsheet.

#### 3.4.7.7 Brightness and Opacity

The ISO brightness and ISO opacity were measured according to ISO 2470 and ISO 2471, respectively. The measurement was done under the light source of CIE Illuminance C. The result was an average of ten areas per handsheet.

#### 3.4.7.8 Internal Tear Resistance

The internal tear resistance was measured following TAPPI T-414. The handsheet weighing 200 g was used in the pendulum instrument. The unit was reported in millinewton. For the tear index, tear resistance was divided by the basis weight.

## 3.4.7.9 Tensile Strength and Elongation

The tensile strength and elongation were measured according to TAPPI T-494. The clamp of the instrument was set at 10 cm. The specimens having 1.5 cm wide were prepared from the handsheets. 12 pieces of the handsheet specimens were measured for each sample by loading each of them to the clamp to elongate. To calculate the tensile strength, the resulting value was multiplied by 1.5 cm, a width of the specimen strip. For the tensile index, tensile strength was divided by its basis weight.

## 3.4.7.10 Burst Strength

The burst strength was measured by following TAPPI T-403. The result was an average of the burst strength in kPa from two sides of the handsheet. For calculation of the burst index, the burst strength was divided by its basis weight.

# 3.4.7.11 Contact Angle

The contact angle of the materials was measured in a dynamic mode. The initial contact angle of the first drop of water was measured after placing it on the substrate for 10 s. Both the top and bottom sides of the sheet were measured.

## 3.4.7.12 Fiber Length Distribution

The fiber length distribution was measured by using Fiber Quality Analyzer (FQA) according to ISO 16065. The total number of fibers counted was 5000 fibers.

## 3.4.7.13 Zero-Span Tensile Strength

The zero-span tensile strength was measured according to TAPPI T-231. The pendulum tensile strength tester with the special clamps adjusted to the zero-span. The specimen strip made of handsheet having a 1.5 cm width was measured. To get zero-span tensile strength, the load was multiplied by 1.5 cm (the width of the specimen strip). For the zero-span tensile index, the zero-span tensile strength was divided by its basis weight.

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# CHAPTER IV

# RESULTS AND DISCUSSION

### Phase I: Effects of Sodium Hydroxide Dosages Employed in Pulping

#### 4.1 Effects of Sodium Hydroxide Dosages on Pulp Properties

The effects of different dosages of sodium hydroxide used in pulping on properties of kapok pulp such as kappa number, residual alkali and pulp yield were studied. The results were further analyzed using the analysis of variance (ANOVA) to give P-value, F calculated and F critical. If the P-value is less than 0.05 and if F calculated is higher that F critical ( $F_{crit}$ ), the effect observed is statistically significant at 95% level of confidence.

## 4.1.1 Kappa Number

The Kappa number is a measure of the lignin content of pulp. The kappa test is an indirect method of determining lignin by the consumption of permanganate ion by lignin [28]. The kappa number of kapok pulp measured in this experiment is shown in Table 4-1. Generally, when a higher dosage of sodium hydroxide is added to the pulp, kappa number decreases due to more lignin and extractives removal. The results from this experiment; however, did not abide by this rule. It is possible that kappa number in this case might not be directly related to the degree of delignification of the pulping process due to other substances present in the pulp which might consume permanganate as well. Also, pulp aging time before performing the kappa number determination is critical. If pulp is kept too long before being tested, the kappa result might be error. As shown in Table 4-2 (the ANOVA of Kappa number), the P-value was higher than 0.05 and the F calculated was lower than F<sub>crit</sub>. This means that in this experiment, the effect of sodium hydroxide dosages on pulp kappa number is not statistically significant.

### 4.1.2 Residual Alkali

Residual alkali indicates the amount of excess sodium hydroxide present in the system after pulping. A high amount of residual alkali ensures complete chemical reactions. Excessive alkali usage; however, means a higher production cost. In this experiment, when the concentration of sodium hydroxide in cooking liquor was increased, the amount of excess sodium hydroxide reacting with lignin was also increased. This resulted in the higher amount of residual alkali in the black liquor as well (shown in Table 4-1). The ANOVA of the residual alkali (Table 4-3) shows that the P-value was lower than 0.05 and the F was higher than F<sub>crit</sub>, so the effect of different sodium hydroxide dosages on residual alkali was statistically significant. The suitable amount of residual alkali is generally higher than 5 g/L to prevent lignin precipitation on the fibers [29]. The optimum sodium hydroxide concentration in this experiment was more than 20% sodium hydroxide dosage.

## 4.1.3 Pulp Yield

In general, the pulp yield obtained from the chemical pulping process is approximately 50% by weight of the raw material. The results from Table 4-1 show that a high sodium hydroxide concentration could also result in carbohydrate degradation due to peeling reaction. This led to a lower overall pulp yield. The ANOVA of pulp yield as illustrated in Table 4-4 reveals that the P-value was lower than 0.05 and the F was higher than  $F_{crit}$ . Thus, the effect of different sodium dosages on pulp yield was statistically significant.

Properties	Sodium hydroxide (NaOH) concentration (%)								
Tioperties	10	15	20	25					
Kappa number	72.74	86.25	99.83	81.08					
Residual alkali (g/L)	2.294	3.177	5.054	6.152					
Pulp yield (%)	62.40	51.25	44.69	40.46					

## Table 4-1 Pulp properties

Table 4-2 ANOVA of kappa number

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	774.4037	3	258.1346	2.204321	0.230073	6.591392
Within Groups	468.4157	4	117.1039			
Total	1242.819	7		G		

Table 4-3 ANOVA of residual alkali

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	18.43197	3	6.143989	8.048478	0.036026	6.591392
Within Groups	3.053491	4	0.763373			
Total	21.48546	7				

Table 4-4 ANOVA of pulp yield

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	548.1866	3	182.7289	37.11493	0.002229	6.591392
Within Groups	19.6933	4	4.923325			
Total	567.8799	7				

## 4.1.4 Canadian Standard Freeness (CSF)

The Canadian standard freeness of kapok pulp with varying amounts of sodium hydroxide is shown in Figure 4-1. The results indicate that increasing sodium hydroxide concentration provided lower pulp freeness because sodium hydroxide probably removed hydrophobic lignin, thus increased the hydrophilic cellulose contact area to absorb water and slowed the drainage. More fines generated due to more severe pulping condition could also result in lower freeness due to their much higher surface area (5-10 times that of fibers). It was found from the ANOVA of Canadian standard freeness in Table 4-5 that the P-value was lower than 0.05 and the F was higher than F<sub>crit</sub>, so the effect of sodium hydroxide dosages on pulp freeness was statistically significant.



Figure 4-1 Canadian standard freeness of the pulp from different NaOH dosages.

Table 4-5 ANO	VA of Can	adian standard	freeness
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Source of Variation	SS	df	MS	F	P-value	F <sub>crit</sub>
Between Groups	20037.25	3	6679.083	86.25128	0.000432	6.591392
Within Groups	309.75	4	77.4375			
Total	20347	7				

# 4.1.5 Fiber Length Distribution

The fiber length distribution of kapok pulp from different pulping conditions is shown in Figure 4-2. The results show that various sodium hydroxide dosages had little effect on fiber length distribution. However, it was found that 25% NaOH dosage produced more short fibers than other conditions. When NaOH dosage was increased, the numerical average fiber length was also decreased (see Table 4-6). This might be because the fibers were damaged by a peeling reaction under the strong alkali condition and this led to carbohydrate degradation. This severe condition also resulted in more fines produced which well corresponded to the freeness results obtained (Figure 4-1).



Figure 4-2 Fiber length distribution of kapok pulp from different NaOH dosages.

Dronorty	Sodium hydroxide (NaOH) concentration (%)							
Property	10	15	20	25				
Numerical average fiber length (mm)	0.907	0.908	0.827	0.807				
Fines (%)	21.538	21.638	22.582	23.688				

Table 4-6 Average fiber length of kapok pulp from different NaOH dosages

# 4.2 Effects of Sodium Hydroxide Dosages on Paper Properties

# 4.2.1 Structural Properties

# 4.2.1.1 Caliper and Apparent Density

The caliper and the apparent density of handsheets from different sodium hydroxide dosages used in pulping are shown in Figures 4-3 and 4-4, respectively. The results show that increasing concentration of sodium hydroxide offered lower caliper and higher apparent density maybe because higher lignin removal due to higher sodium hydroxide dosage liberated more fibers and rendered these free fibers to collapse and become more conformable. So, the denser sheet was obtained. In addition to that, shorter fibers and more fines generated by higher dosage of sodium hydroxide also filled in voids and pores in the sheet structure, resulting in thinner and denser sheets. However, the results illustrated in Figure 4-3 and 4-4 do not agree well with the statistic results. Tables 4-7 and 4-8 show the ANOVA of caliper and apparent density, respectively. It could be surprisingly seen that the P-values in both cases were higher than 0.05 and the F values were lower than  $F_{crit}$ . Thus, it can be concluded that the sheet caliper and density were not statistically affected by various sodium hydroxide dosages. This might probably due to its surface irregularity.



Figure 4-3 Caliper of the sheets from different NaOH dosages.



Figure 4-4 Apparent density of the sheets from different NaOH dosages.

Table 4-7 ANOVA of caliper

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	0.000707	3	0.000236	4.2774	0.097084	6.591392
Within Groups	0.00022	4	5.51E-05			
Total	0.000928	7				

Table 4-8 ANOVA of apparent density

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	0.04285	3	0.014283	3.548654	0.12639	6.591392
Within Groups	0.0161	4	0.004025			
Total	0.05895	7				

## 4.2.1.2 Bekk Smoothness

The result in Figure 4-5 shows that higher sodium hydroxide dosages increased Bekk smoothness possibly due to more fiber collapse and conformability which caused the surface of the sheets to be more compact. Shorter fibers and fines also contributed to the smoother surface by filling in pores and voids in the sheet. The top side of the sheets had higher Bekk smoothness than the bottom side because the bottom side of the sheet had more long fibers that gave an irregular surface. Tables 4-9 and 4-10 show the ANOVA of Bekk smoothness for the bottom side and top side, respectively. The results indicate that the P-values were lower than 0.05 and the F values were higher than  $F_{crit}$ ; therefore, the effect of sodium hydroxide dosages on Bekk smoothness was statistically significant.



Figure 4-5 Bekk smoothness of the sheets from different NaOH dosages.

Table 4-9 ANOVA of Bekk smoothness of the sheet bottom side

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	39.33534	3	13.11178	12.93984	0.015843	6.591392
Within Groups	4.05315	4	1.013287			
Total	43.38849	7				

Table 4-10 ANOVA of Bekk smoothness of the sheet top side

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	72.8413	3	24.28043	8.588991	0.0323	6.591392
Within Groups	11.3077	4	2.826925	ทยา		
Total	84.149	7				

## 4.2.2 Optical Properties

#### **Brightness and Opacity**

The results in Figure 4-6 show that the sodium hydroxide dosage seemed to have a small impact on brightness and opacity. Tables 4-11 and 4-12 show the ANOVA of brightness and opacity, respectively. For the brightness, the P-value was higher than 0.05 and the F was lower than Fcrit. Consequently, the effect of sodium hydroxide dosages on pulp brightness was statistically insignificant. This finding is quite surprising since generally pulp brightness is higher when a higher amount of sodium hydroxide is used because more lignin is removed. However, it was criticized that higher concentrations of sodium hydroxide may cause the alkali darkening reaction which results in structural changes of the chromophoric groups in the residual lignin. These chromophoric groups can absorb light and thus cause brightness of the pulp to decrease. For opacity, the P-value was lower than 0.05 and the F was higher than F<sub>crit</sub>. So, the effect of sodium hydroxide dosages on opacity was statistically significant. This could be probably explained by the effect of sodium hydroxide on lignin removal leading to sheet compactness which reduced light scattering of the sheet. Also, increasing sodium hydroxide dosage caused more fines content which might lead to sheet compactness as well.



Figure 4-6 Brightness and opacity of the sheets from different NaOH dosages.

Table 4-11 ANOVA of brightness

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	11.85174	3	3.950579	2.572473	0.191782	6.591392
Within Groups	6.14285	4	1.535713			
Total	17.99459	7				

Table 4-12 ANOVA of opacity

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	0.8848	3	0.294933	10.33041	0.023547	6.591392
Within Groups	0.1142	4	0.02855			
Total	0.999	7				

## 4.2.3 Strength Properties

### 4.2.3.1 Tear Resistance

Tear resistance is affected by many factors such as intrinsic fiber strength, fiber length, and fiber bonding; however, the most important one is the fiber strength. The tear index which is equal to tear resistance divided by grammage defines tear resistance of the paper. The results in Figure 4-7 indicate that when the sodium hydroxide dosage was increased, the tear index tended to drop. This may be caused by the peeling reaction which destroys endwise chains of cellulose and hemicellulose; and the alkaline hydrolysis leads to a drop in the degree of polymerization of the fibers [30]. The decrease in tear resistance may also be caused by shorter fiber length and higher amount of fines present. Nonetheless, the effect of various sodium hydroxide dosages was statistically insignificant as proven by ANOVA of tear index (Table 4-13). The P-value was higher than 0.05 and the F was lower than F<sub>crit</sub>. This statistically insignificant effect of sodium hydroxide dosages on tear index might possibly be caused by the poor formation and irregular surface of the sheet which affects tear resistance of the sheet.



Figure 4-7 Tear index of the sheets from different NaOH dosages.

Table 4-13 ANOVA of tear index

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	0.0439	3	0.014633	1.258781	0.400481	6.591392
Within Groups	0.0465	4	0.011625			
Total	0.0904	7				

# 4.2.3.2 Tensile Strength

Many factors affect tensile strength, the most important one being fiber bonding which is a function of fiber contact area. The tensile index which is equal to tensile strength divided by grammage describes tensile strength of paper. The results in Figure 4-8 demonstrate that tensile index was increased when increasing the sodium hydroxide concentration. This might probably be due to more lignin removal and better separation of individual fiber at higher sodium hydroxide concentration. This then contributed to higher numbers of cellulose contact areas for better bonding. Nevertheless, the tensile index reached a maximum at 20% sodium hydroxide. When using 25% sodium hydroxide, the tensile index tended to drop, possibly because of the peeling reaction which cleaves the end of cellulose chains resulting in loss of fiber strength. The ANOVA of tensile index in Table 4-14 shows that P-value was lower than 0.05 while the F was higher than  $F_{crit}$ . Thus, the effect of sodium hydroxide dosages on the tensile strength was statistically significant.



Figure 4-8 Tensile index of the sheets from different NaOH dosages.

Table 4-14 ANOVA of tensile index

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	1022.092	3	340.6975	138.6993	0.000169	6.591392
Within Groups	9.8255	4	2.456375			
Total	1031.918	7		ากร		

# 4.2.3.3 Burst Strength

The factors affecting burst strength are similar to the case of tensile strength. Fiber bonding is also the most important factor that affects burst strength. The burst index is another indicator for burst strength of paper and it is defined by the burst strength divided by the grammage of the paper. From Figure 4-9, it was found that the burst index increased with higher amount of sodium hydroxide dosage because fiber bonding had been improved. Like the tensile index, the burst index reached its maximum at 20% sodium hydroxide dosage. Table 4-15 showing the ANOVA results of the burst index reveals that the P-value was lower than 0.05 and the F was higher than  $F_{crit}$ . Consequently, the effect of sodium hydroxide dosages on burst strength was statistically significant.



Figure 4-9 Burst index of the sheets from different NaOH dosages.

Table 4-15 ANOVA of burst index

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	3.2133	3	1.0711	8.209235	0.034853	6.591392
Within Groups	0.5219	4	0.130475			
Total	3.7352	7	รัพย	ากร		

#### 4.2.3.4 Elongation

The elongation of the sheets is a sheet properties related to tensile strength of the sheet. Thus, the results obtained from elongation should have the same trend as in the case of tensile strength. It is clear from Figure 4-10 that sheet was more elongated when increasing the concentration of sodium hydroxide because individual fiber became free and more conformable at higher sodium hydroxide concentrations. As shown in Table 4-16 for the ANOVA of elongation, the P-value was lower than 0.05 and the F was higher than F<sub>crit</sub>. So, the effect of sodium hydroxide concentration on the sheet elongation was statistically significant.



Figure 4-10 Elongation of the sheets from different NaOH dosages.

Table 4-16 ANOVA of elongation

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	0.15255	3	0.05085	8.404959	0.033501	6.591392
Within Groups	0.0242	4	0.00605			
Total	0.17675	7				

## 4.2.3.5 Zero-Span Tensile Strength

Unlike the tensile strength, zero-span tensile strength is mostly influenced by intrinsic fiber strength rather than fiber bonding and has closer relationship to tear resistance. The zero-span tensile index which explains zero-span tensile strength of paper is equal to zero-span tensile strength divided by grammage. The results as indicated by Figure 4-11 show similar trend to those of tear resistance (Figure 4-7). It was found that higher sodium hydroxide dosages slightly decreased zero-span tensile strength. The statistical results of zero-span tensile index were also similar to those of tear index. Table 4-17 illustrating the ANOVA of the zero-span tensile index provides that the P-value was higher than 0.05 and the F was lower than  $F_{crit}$ . Hence, the effect of sodium hydroxide concentration on zero-span tensile index was statistically insignificant.



Figure 4-11 Zero-span tensile index of the sheets from different NaOH dosages.

Table 4-17 ANOVA of zero-span tensile ind	ex
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Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	15.39689	3	5.132296	0.056385	0.980066	6.591392
Within Groups	364.0873	4	91.02182			
Total	379.4842	7	รัพย	ากร		

# **4.2.4 Surface Properties**

## Water Contact Angle

Figures 4-12 and 4-13 show the change of water contact angle over time of the sheets prepared from the kapok pulp which was cooked at various sodium hydroxide concentrations. It was found that higher concentration of sodium hydroxide gave lower water contact angle possibly due to higher waxes removal to give a more hydrophilic surface. Normally, the kapok fiber has cutin wax coated on the surface. When kapok fibers were pulped, the heat and the chemical reaction from sodium hydroxide might cause waxes to soften and to be easily released from the fiber surface.



Figure 4-12 Water contact angle of the sheets from different NaOH dosages (Top side).



Figure 4-13 Water contact angle of the sheets from different NaOH dosages (Bottom side).

As discussed earlier, it can be concluded from phase I that the optimum sodium hydroxide dosage for kapok cooking was 20% based on oven-dried pulp weight, because it provided optimum paper strength properties. So, this condition was further used for the pulping in phase II. To make the picture of the overall statistical analysis results from Phase I clearer, all ANOVA results were summarized in Table 4-18.

Properties	P-value	Statistical analysis result
Kappa number	0.230073	insignificant
Residual alkali	0.036026	significant
Pulp Yield	0.002229	significant
Freeness	0.000432	significant
Caliper	0.097084	insignificant
Apparent density	0.12639	insignificant
Smoothness (bottom side)	0.015843	significant
Smoothness (top side)	0.0323	significant
Brightness	0.191782	insignificant
Opacity	0.023547	significant
Tear resistance	0.400481	insignificant
Zero-span tensile strength	0.980066	insignificant
Tensile strength	0.000169	significant
Elongation	0.033501	significant
Burst strength	0.034853	significant

Table 4-18 Summarized statistical analysis results from Phase I

Note: if P-value was lower than 0.05, then the result was not statistically insignificant.

#### Phase II: Effects of Disc Gaps and Numbers of Passes during Refining

### 4.3 Effects of Disc Refining Gaps on Pulp Properties

Pulp is usually refined before handsheet is made. Refining causes pulp to become fibrillated. These fibrillated fibers increase the surface areas for fiber bonding and then lead to better strength of sheet. However, if the pulp has been severely or intensively refined, pulp is then damaged since more fibers are cut instead of fibrillated and more fines content is produced. This can deteriorate overall paper strength. The factors affecting refining include disc rotation speed, disc design, disc diameter, disc gap and so on [31].

### 4.3.1 Canadian Standard Freeness (CSF)

The Canadian standard freeness of the pulp with varying disc gaps during the refining processes was shown in Table 4-19. The pulp that was refined once by the 7/1000 inch disc gap gave the lower freeness than that was refined twice by the 10/1000 inch disc gap because 7/1000 inch disc gap provided a narrower gap than 10/1000 inch gap. This narrower gap encouraged more fiber cutting mechanism instead of fibrillation and this led to higher fines content and short fibers. Higher amount of short fibers and fines content decreases pulp freeness due to higher surface areas for water holding. It should be noted that the pulp which was refined by the 10/1000 inch disc gap for one pass followed by the 7/1000 inch disc gap for one pass had too low freeness to be measured and made into handsheets due to more fiber cutting and damage instead of fibrillation.

Table 4-19 Canadian standard freeness of the pulp from different disc refining gaps

Property	Disc gap (inch)*number of passes				
rioperty	10/1000*2	7/1000*1	(10/1000*1) + (7/1000*1)		
Freeness (CSF, mL)	284±6.36	244±7.78	ND		

#### 4.3.2 Fiber Length Distribution

Figure 4-14 shows fiber length distribution of the pulp from different disc refining gaps. The results depict that the pulp refined once by 7/1000 inch disc gap had a slightly higher amount of short fibers (0.4-1.2 mm long) and a slightly lower amount of medium fibers (1.3-2.5 mm long) than those were refined twice by the 10/1000 inch disc gap due to more fiber shortening caused by the narrower gap of 7/1000 inch disc gap. This finding agrees well with the results from Table 4-20 which shows the average fiber length and fines content. It can be seen that pulp refined twice by the 10/1000 inch disc gap had the lower fines content and the higher average fiber length as compared to the pulp refined once by 7/1000 inch disc gap. This discovery also supports the reason why the 7/1000 inch disc gap.

Properties	Disc gap (inch)*number of passe			
Properties	10/1000*2	7/1000*1		
Numerical average fiber length (mm)	0.883	0.803		
Fines (%)	22.94	23.58		

Table 4-20 Average fiber length of the pulp from different disc refining gaps



Figure 4-14 Fiber length distribution of the pulp from different disc refining gaps.

# 4.4 Effects of Disc Refining Gaps on Paper Properties

## 4.4.1 Structural Properties

The structural properties from different disc refining gaps are shown in Table 4-21. The disc refining gaps evaluated here did not strongly affect the caliper and apparent density of the sheet. However, the smoothness of the sheet made from the pulp refined once by 7/1000 inch disc gap was much higher than refined twice by 10/1000 inch disc gap because the 7/1000 inch disc gap was narrower, thus it could shorten more fibers. When the content of short fibers increased, it caused compaction of the paper structure. In addition to that, short fibers could fill in pores and voids on the surface of the sheet and then give the higher sheet smoothness.

Properties	Disc gap (inch) *number of passes				
rioperues	10/1000*2	7/1000*1			
Caliper (mm)	0.073±0.0011	0.074±0.0009			
Apparent Density (g/cm3)	0.79±0.03	0.80±0.02			
Bottom Smoothness (s)	5.22±0.46	9.00±0.55			
Top Smoothness (s)	9.33±0.78	13.08±1.13			

Table 4-21 Structural properties from different disc refining gaps

#### 4.4.2 Optical Properties

The optical properties of the sheet from different disc refining gaps are shown in Table 4-22. The results indicate that the disc gap did not have a strong impact on brightness and opacity of the sheets. Pulp refined once by 7/1000 inch disc gap seemed to offer slightly higher brightness than pulp refined twice by 10/1000 inch disc gap. This might be because 7/1000 inch disc gap provided higher content of shorter fibers and fines. This could lead to higher light scattering coefficient of the pulp. However, in the case of opacity, the effects of different disc gaps used seemed to be faded away. Generally, opacity should be higher with higher amount of short fibers due to more surface areas for light scattering. Nevertheless, if the amount of short fibers and fines content is too high, these short fibers and fines might fill in the air volumes in the sheet. So, the light refraction due to the different between air refractive index and fiber refractive index is decreased and this could lead to lower opacity of the sheet instead.

Properties	Disc gap (inch) *number of passes			
Flopentes	10/1000*2	7/1000*1		
Brightness (%)	18.51±0.07	19.41±0.30		
Opacity (%)	96.66±0.22	96.61±0.31		

Table 4-22 Optical properties from different disc refining gap

# 4.4.3 Strength Properties

As previously explained, the aim of pulp refining is to fibrillate pulp fibers so that the bonding areas are increased and strength properties will become higher. If; however, pulp is refined too long or pulp is intensively refined, pulp fibers are then cut and become shorten instead of fibrillated. This will instead damage the strength properties of the pulp. It can be seen from the Table 4-23 that tear index and zero-span tensile index of the sheet made from the pulp refined once by the 7/1000 inch disc gap was lower than that of the sheet produced from the pulp refined twice by the 10/1000 inch disc gap. This might be because narrower gap of the 7/1000 inch disc gap rendered higher amount of short fibers and fines. Also, 7/1000 inch disc gap. This caused lower tensile index, burst index and elongation of handsheets.

Properties	Disc gap (inch) *number of passes		
	10/1000*2	7/1000*1	
Tear index (mN m <sup>2</sup> /g)	1.62±0.12	1.50±0.10	
Tensile index (Nm/g)	108.76±7.12	95.02±4.85	
Burst index (kPa m <sup>2</sup> /g)	5.55±0.47	5.50±0.33	
Elongation (%)	2.14±0.35	1.97±0.20	
Zero-span tensile index (Nm/g)	52.05±8.58	47.69±10.86	

Table 4-23 Strength properties from different disc refining gaps

#### 4.4.4 Surface Properties

#### Water Contact Angle

When the pulp was refined twice through the 10/1000 inch disc gap, the water contact angle measured was slightly higher than those refined once through the 7/1000 inch disc gap (Figure 4-15). This is possibly caused by the irregular surface of the sheet.



Figure 4-15 Water contact angle of the sheets from different disc refining gaps.

Therefore, it could be concluded from phase II that the optimum refining condition was to refine twice through the 10/1000 inch disc gap because it provided optimum strength properties. So, this condition was further used for the refining in phase III.

## Phase III: Effects of the Kapok Pulp Mixed with the Commercial Pulps

# **Fiber Length Distribution**

The fiber length distributions of the kapok pulp and the commercial pulps are shown in Figure 4-16. The results demonstrate that the kapok pulp had lower amount of short fibers than the hardwood pulp while the softwood pulp had the lowest content of short fibers. The kapok pulp; however, had a higher amount of both medium and long fibers than the hardwood pulp while the softwood pulp contained the highest amount of long fibers. Table 4-24 reveals that numerical average fiber length of the kapok pulp was between those of the softwood and hardwood pulps. The kapok pulp had the lowest fines followed by the hardwood pulp and softwood pulp, respectively.



Figure 4-16 Fiber length distributions of the kapok, hardwood and softwood pulps.

Properties .	Pulp types		
	Kapok	Softwood	Hardwood
Fiber length (mm)	0.834	1.253	0.496
Fines (%)	22.732	43.188	33.976

Table 4-24 Average fiber length of the kapok and the commercial pulps

# 4.5 Effects of the Kapok Pulp Mixed with the Commercial Pulps on Paper Properties

# 4.5.1 Structural Properties

# 4.5.1.1 Caliper and Apparent Density

The caliper and apparent density of the sheets prepared from different ratios of the kapok pulp to the commercial pulps which were the softwood pulp, hardwood pulp and mixed pulp between the hardwood and softwood pulps are shown in Figures 4-17 and 4-18, respectively. The sheets made from the 100% hardwood pulp had a lower caliper and higher apparent density than the sheets containing the 100% softwood pulp and 100% kapok pulp because the shorter fibers of the hardwood pulp could fill in voids and pores in the sheet structure and the sheets became more compact. The results also show that the kapok pulp seemed to decrease the caliper and increase apparent density of the sheets in all pulp mixtures. Since the kapok fibers have the thinner cell wall, thus they are easily collapsed and make more compact sheets. However, the sheets containing the 100% kapok pulp did not produce sheets with the lowest caliper or the highest apparent density. This might be probably due to the fact that kapok fibers tended to become easily entangled when the fibers stayed alone. Thus, the pure kapok sheets seemed to provide a poor sheet formation and irregular sheet surface. However, the ANOVA results as indicated in Tables 4-25 to 4-30 show that the effects of the kapok pulp on caliper and apparent density were statistically significant only in the case of the pulp mixture prepared by the kapok pulp mixed with the softwood pulp. This is based on that the P-values were lower than 0.05 and F values were higher than  $F_{crit}$  (Tables 4-26 and 4-27).



Figure 4-17 Calipers of the sheets made from kapok pulp mixed with the commercial pulps at different ratios.


Figure 4-18 Apparent density of the sheets made from the kapok pulp mixed with the commercial pulps at different ratios.

Table 4-25 ANOVA of caliper (the kapok pulp mixed with the hardwood pulp)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	5.74E-05	4	1.43E-05	2.314516	0.191039	5.192163
Within Groups	3.1E-05	5	6.2E-06			
Total	8.84E-05	9				

Table 4-26 ANOVA of apparent density (the kapok pulp mixed with the hardwood pulp)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	0.005713	4	0.001428	4.930005	0.055094	5.192163
Within Groups	0.001449	5	0.00029			
Total	0.007162	9				

Table 4-27 ANOVA of caliper (the kapok pulp mixed with the softwood pulp)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	9.14E-05	4	2.29E-05	10.38636	0.012222	5.192163
Within Groups	1.1E-05	5	2.2E-06			
Total	0.000102	9				

Table 4-28 ANOVA of apparent density (the kapok pulp mixed with the softwood pulp)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	0.010461	4	0.002615	9.230345	0.01573	5.192163
Within Groups	0.001417	5	0.000283			
Total	0.011878	9				

Table 4-29 ANOVA of caliper (the kapok pulp blended with the mixed pulp)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	5.46E-05	4	1.36E-05	2.967391	0.131953	5.192163
Within Groups	2.3E-05	5	4.6E-06			
Total	7.76E-05	9				

Table 4-30 ANOVA of apparent density (the kapok pulp blended with the mixed pulp)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	0.006496	4	0.001624	3.881974	0.084649	5.192163
Within Groups	0.002092	5	0.000418			
Total	0.008587	9				

#### 4.5.1.2 Bekk Smoothness

Figures 4-19 and 4-20 show Bekk smoothness of the sheets made from the kapok pulp mixed with the commercial pulps at different ratios. Figure 4-19 illustrates Bekk smoothness of the sheet bottom side while Figure 4-20 reveals Bekk smoothness of the sheet top side, respectively. The sheets with the 100% hardwood pulp had the higher smoothness than the sheets with the 100% softwood pulp and 100% kapok pulp because the hardwood pulp had the shorter fibers than the softwood pulp that could fill in voids and pores in the paper structure leading to smoother surface. The sheets with the 100% kapok pulp had smoothness close to the sheets made from the softwood pulp probably because the kapok fibers were quite long and became entangled easily. This might cause a poor sheet formation and irregular surface of the sheet. The results also illustrate that kapok pulp decreased smoothness of the sheets in every pulp mixture but had smaller effects on the sheets prepared by the blend of kapok and softwood pulps. The ANOVA results as appeared in Tables 4-31 to 4-36 show that the effects of the kapok pulp in all pulp mixtures except the case of the kapok pulp mixed with the softwood pulp are statistically significant on sheet smoothness. This might be because the sheets from both the 100% kapok pulp and the 100% softwood pulp already had low smoothness. Even when both pulps were mixed, the smoothness was not different either. The smoothness on the top side of the sheet was higher than the bottom side because the top side had more short fibers and fines which caused the smoother sheet surface.



Figure 4-19 Bekk smoothness of the sheets made from the kapok pulp mixed with the commercial pulps at different ratios (the sheet bottom side).

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Figure 4-20 Bekk smoothness of the sheets made from the kapok pulp mixed with the commercial pulps at different ratios (the sheet top side).

Table 4-31 ANOVA of Bekk smoothness on the sheet bottom side (the kapok pulp mixed with the hardwood pulp)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	241.5611	4	60.39028	34.71205	0.000769	5.192163
Within Groups	8.69875	5	1.73975			
Total	250.2599	9				

Table 4-32 ANOVA of Bekk smoothness on the sheet top side (the kapok pulp mixed with the hardwood pulp)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	466.781	4	116.6953	44.83571	0.000416	5.192163
Within Groups	13.01365	5	2.60273			
Total	479.7947	9				

Table 4-33 ANOVA of Bekk smoothness on the sheet bottom side (the kapok pulp mixed with the softwood pulp)

Source of Variation	SS	df	MS	F	P-value	F <sub>crit</sub>
Between Groups	4.27806	4	1.069515	4.596901	0.06267	5.192163
Within Groups	1.1633	5	0.23266			
Total	5.44136	9				

Table 4-34 ANOVA of Bekk smoothness on the sheet top side (the kapok pulp mixed with the softwood pulp)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	4.60086	4	1.150215	2.527556	0.168347	5.192163
Within Groups	2.27535	5	0.45507			
Total	6.87621	9				

Table 4-35 ANOVA of Bekk smoothness on the sheet bottom side (the kapok pulp blended with the mixed pulps)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	102.9861	4	25.74652	20.32935	0.00271	5.192163
Within Groups	6.33235	5	1.26647			
Total	109.3184	9				

Table 4-36 ANOVA of Bekk smoothness on the top side (kapok pulp blended with the mixed pulps)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	171.2751	4	42.81878	23.45438	0.001943	5.192163
Within Groups	9.1281	5	1.82562			
Total	180.4032	9				

#### 4.5.2 Optical Properties

#### 4.5.2.1 Brightness

As illustrated in Figure 4-21, the sheets made from the 100% commercial pulps (hardwood and softwood) had higher brightness than those made from the 100% kapok pulp. The yellowness of the kapok pulp decreased brightness of the sheets obtained from all pulp mixtures. This was also confirmed by the statistical results as shown in the ANOVA tables from Tables 4-37 to 4-39. All P-values were much lower than 0.05 and all F values were much higher than  $F_{crit}$ . So, in all pulp mixtures, the effect of the kapok pulp on brightness was statistically significant.



Figure 4-21 Brightness of the sheets made from the kapok pulp mixed with the commercial pulps at different ratios.

Table 4-37 ANOVA of brightness (the kapok pulp mixed with the hardwood pulp)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	1715.546	4	428.8864	239.6589	6.76E-06	5.192163
Within Groups	8.94785	5	1.78957			
Total	1724.493	9				

Source of Variation	SS	df	MS	F	P-value	F <sub>crit</sub>
Between Groups	1110.179	4	277.5447	87.92243	8.06E-05	5.192163
Within Groups	15.7835	5	3.1567			
Total	1125.962	9				_

Table 4-38 ANOVA of brightness (the kapok pulp mixed with the softwood pulp)

Table 4-39 ANOVA of brightness (the kapok pulp blended with the mixed pulp)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	1667.722	4	416.9305	189.1528	1.22E-05	5.192163
Within Groups	11.021	5	2.2042			
Total	1678.743	9				

#### 4.5.2.2 Opacity

The opacity values of the sheet made from kapok pulp mixed with the commercial pulps at different ratios are shown in Figure. 4-22. It is discovered that the opacity was slightly decreased with increasing the ratios of kapok pulp because the kapok fiber tended to collapse and result in a denser paper structure that decreased opacity of the sheet. However, Figure 4-22 does not show the explicit result. Differences in the opacity values are shown in Tables 4-40 to 4-42 by using ANOVA. Those ANOVA tables indicate that the P-values were lower than 0.05 and the F values were higher than  $F_{crit}$ . The results were thus significantly different and the effect of kapok pulp on opacity in all pulp mixtures was statistically significant.



Figure 4-22 Opacity of the sheets made from the kapok pulp mixed with the commercial pulps at different ratios.

Table 4-40 ANOVA of opacity (the kapok pulp mixed with the hardwood pulp)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	12.65686	4	3.164215	36.66105	0.000675	5.192163
Within Groups	0.43155	5	0.08631			
Total	13.08841	9				

Table 4-41 ANOVA of opacity (the kapok pulp mixed with the softwood pulp)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	6.20746	4	1.551865	14.06949	0.006268	5.192163
Within Groups	0.5515	5	0.1103			
Total	6.75896	9				

Source of Variation	SS	df	MS	F	P-value	F <sub>crit</sub>
Between Groups	10.64174	4	2.660435	25.06298	0.001663	5.192163
Within Groups	0.53075	5	0.10615			
Total	11.17249	9				

Table 4-42 ANOVA of opacity (the kapok pulp blended with the mixed pulp)

#### 4.5.3 Strength Properties

#### 4.5.3.1 Tear Resistance

The tear resistance of the sheet can also be defined by tear index which is equal to tear resistance divided by grammage. Since one of the key characteristics of kapok fiber is its brittleness, the sheets made from the 100% kapok pulp had the lowest tear index as compared to the sheets made by the 100% commercial pulps. Figure 4-23 demonstrates the tear index of the sheets made from the kapok pulp mixed with the commercial pulps at different ratios. It shows that as the ratios of kapok pulp increased, the tear index decreased. This was also confirmed by the ANOVA results in Tables 4-43 to 4-45. It can be seen that the results were significantly different and the effect of kapok pulp on tear index was statistically significant in all pulp mixtures since all P-values were much lower than 0.05 while all F-values were much higher than F<sub>crit</sub>.



Figure 4-23 Tear index of the sheets made from the kapok pulp mixed with the commercial pulps at different ratios.

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	21.49936	4	5.37484	320.5033	3.28E-06	5.192163
Within Groups	0.08385	5	0.01677			
Total	21.58321	9				

Table 4-43 ANOVA of tear index (the kapok pulp mixed with the hardwood pulp)

Table 4-44 ANOVA of tear index (the kapok pulp mixed with the softwood pulp)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	137.688	4	34.42201	543.534	8.81E-07	5.192163
Within Groups	0.31665	5	0.06333			
Total	138.0047	9				

Table 4-45 ANOVA of tear index (the kapok pulp blended with the mixed pulp)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	77.19266	4	19.29817	74.66596	0.00012	5.192163
Within Groups	1.2923	5	0.25846			
Total	78.48496	9				

#### 4.5.3.2 Tensile Strength

The tensile index which defines the tensile strength of paper is equal to tensile strength divided by grammage. As indicated in Figure 4-24, the sheets with the 100% kapok pulp had the highest tensile index. This might be because the kapok fibers are long and the lumens are easily collapsed when forming paper. This caused higher bonded areas and denser sheet which led to superior tensile index of the sheet. The sheets with the 100% hardwood pulp produced the lowest tensile index. The sheets made from the pulp blended between 75% hardwood and 25% softwood pulps had the medium tensile index while the sheets with the pure softwood pulp gave the

highest tensile index. When the ratios of kapok pulp increased, the tensile index also increased as shown in Figure 4-24. However, the results are more prominent when the kapok pulp was mixed with the hardwood pulp. The ANOVA results in Tables 4-46 to 4-48 show that all the kapok pulp ratios in all pulp mixtures were significantly different. Therefore, the effect of kapok pulp on tensile index of all pulp mixtures was statistically significant since all P-values were lower than 0.05 and all F-values were higher than  $F_{crit}$ .



Figure 4-24 Tensile index of the sheets made from the kapok pulp mixed with the commercial pulps at different ratios.

Table 4-46 ANOVA of tensile index (the kapok pulp mixed with the hardwood pulp)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	2049.447	4	512.3618	293.2373	4.1E-06	5.192163
Within Groups	8.7363	5	1.74726			
Total	2058.183	9				

Source of Variation	SS	df	MS	F	P-value	F <sub>crit</sub>
Between Groups	424.1867	4	106.0467	18.98511	0.003173	5.192163
Within Groups	27.9289	5	5.58578			
Total	452.1156	9				

Table 4-47 ANOVA of tensile index (the kapok pulp mixed with the softwood pulp)

Table 4-48 ANOVA of tensile index (the kapok pulp blended with the mixed pulp)

Source of Variation	SS	df	MS	F	P-value	F <sub>crit</sub>
Between Groups	1234.281	4	308.5701	97.30298	6.28E-05	5.192163
Within Groups	15.85615	5	3.17123			
Total	1250.137	9				

#### 4.5.3.3 Burst Strength

The burst index defines burst strength of the paper. It is equal to burst strength divided by grammage. The sheets with the 100% kapok pulp had the highest burst index similar to the sheets containing the 100% softwood pulp (Figure 4-25) since both the kapok fiber and softwood fiber are long and have high contact areas available for fiber bonding. This was also confirmed by the ANOVA of tensile index (the kapok pulp mixed with the softwood pulp) appeared in Table 4-49 that the results were insignificantly different. The effect of the kapok pulp on burst strength when it was mixed with the softwood pulp was not statistically significant. The sheets with the 100% hardwood pulp had the lowest burst index because the hardwood pulp contained a higher amount of short fibers that caused poor bonding. When the ratios of the kapok pulp were increased, the burst indexes of the pulp mixtures between the kapok and the hardwood pulps and those between the kapok and the mixed pulps (75% hardwood: 25% softwood) also increased because fiber bonding was drastically improved. Tables 4-49 and 4-51 show the ANOVA results of the burst indexes of the kapok pulp mixed with the hardwood pulp and the kapok pulp blended with the mixed pulp, respectively. The results were significantly different. The effect of the kapok

pulp on burst index in both cases was statistically significant since the P-values were lower than 0.05 and F-values were higher than F<sub>crit</sub> (see Tables 4-48 and 4-50).



Figure 4-25 Burst index of the sheets made from the kapok pulp mixed with the commercial pulps at different ratios.

Table 4-49 ANOVA of burst index (the kapok pulp mixed with the hardwood pulp)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	6.15266	4	1.538165	87.6947	8.11E-05	5.192163
Within Groups	0.0877	5	0.01754			
Total	6.24036	9				

Table 4-50 ANOVA of burst index (the kapok pulp mixed with the softwood pulp)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	0.01936	4	0.00484	0.161333	0.949223	5.192163
Within Groups	0.15	5	0.03			
Total	0.16936	9				

Source of Variation	SS	df	MS	F	P-value	F <sub>crit</sub>
Between Groups	2.30504	4	0.57626	8.51448	0.018646	5.192163
Within Groups	0.3384	5	0.06768			
Total	2.64344	9				

Table 4-51 ANOVA of burst index (the kapok pulp blended with the mixed pulp)

#### 4.5.3.4 Elongation

The elongation of the sheets from different ratios of the kapok pulp is shown in Figure 4-26. The sheets with 100% kapok pulp had the lowest elongation because the key characteristics of the kapok fiber were its brittleness and inelasticity which reduced the elongation of the sheet when the load was applied. Thus, when the ratios of kapok pulp increased, the elongation also decreased in all pulp mixtures. The results were also confirmed by the ANOVA results in Tables 4-52 to 4-54 showing that the results were significantly different. Consequently, the effects of kapok pulp on elongation for all pulp mixtures were statistically significant as indicated by the P-values higher than 0.05 and F-values higher than  $F_{crit}$ .



Figure 4-26 Elongation of the sheets made from kapok pulp mixed with the commercial pulps at different ratios.

Table 4-52 ANOVA of elongation (the kapok pulp mixed with the hardwood pulp)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	3.91726	4	0.979315	37.05316	0.000658	5.192163
Within Groups	0.13215	5	0.02643			
Total	4.04941	9				

Table 4-53 ANOVA of elongation (the kapok pulp mixed with the softwood pulp)

Source of Variation	SS	df	MS	F	P-value	F <sub>crit</sub>
Between Groups	0.67286	4	0.168215	29.87833	0.001099	5.192163
Within Groups	0.02815	5	0.00563			
Total	0.70101	9				

Table 4-54 ANOVA of elongation (the kapok pulp blended with the mixed pulp)

Source of Variation	SS	df	MS	F	P-value	Fcrit
Between Groups	2.75126	4	0.687815	29.57072	0.001126	5.192163
Within Groups	0.1163	5	0.02326			
Total	2.86756	9				

#### 4.5.3.5 Zero-Span Tensile Strength

The zero-span tensile index defines the zero-span tensile strength of paper and it is equal to the zero-span tensile strength divided by grammage. It was found that the sheets made from the 100% softwood pulp had the highest zero-span tensile index while the sheets made from the 100% hardwood pulp had the lowest zero-span tensile index, as shown in Table 4-55. The zero-span tensile index of the sheet made from the 100% kapok pulp fell in between them. The reason for this was probably that the measurement was done in a dry state where the effect of fiber bonding was still continued. The zero-span tensile strength observed was, therefore; a function of both intrinsic fiber strength and fiber bonding. If the measurement had done in a wet state, the effect of fiber bonding would have been eliminated which would have resulted in the brittle kapok pulp having a lower zero-span tensile index.

Table 4-55 Zero-span tensile index of kapok pulp and commercial pulp

Property .	Pulp types				
	Kapok	Softwood	Hardwood		
Zero-span tensile index	56 69+9 70	92 57+6 69	44 21+0 27		
(Nm/g)	30.0818.70	82.37±0.08	44.2119.27		

#### 4.5.4 Morphological/Surface Properties

#### 4.5.4.1 Water Contact Angle

The photos of a droplet of water on the sheets prepared from different pulps at an initial contact time (at 0 s contact time) are shown in Figure 4-27. The result illustrates that the sheet made from the kapok pulp had a higher water contact angle than the sheets made from either the softwood pulp or hardwood pulp due to the unique characteristics of the kapok fiber having waxes coated on the surface. Although the kapok fibers were pulped, some of the waxes were still present.



Figure 4-27 First water droplet on the sheets made from (a) the kapok pulp, (b) the softwood pulp, and (c) the hardwood pulp. Figures 4-28 to 4-33 present the water contact angles of the sheets made from the kapok pulp mixed with the commercial pulps. The handsheets from the 100% kapok pulp had the highest water contact angles. When the ratios of the kapok pulps increased, the water contact angles also increased. The results from both the sheet top side and the sheet bottom side were similar. When time of contact has passed, the water contact angles decreased because of absorption and penetration of the water droplets into the sheet.



Figure 4-28 Water contact angles of the sheets made from the kapok pulp mixed with the commercial hardwood pulp at different ratios (the sheet bottom side).



Figure 4-29 Water contact angles of the sheets made from the kapok pulp mixed with the commercial hardwood pulp at different ratios (the sheet top side).



Figure 4-30 Water contact angles of the sheets made from the kapok pulp mixed with the commercial softwood pulp at different ratios (the sheet bottom side).



Figure 4-31 Water contact angles of the sheets made from the kapok pulp mixed with the commercial softwood pulp at different ratios (the sheet top side).



Figure 4-32 Water contact angles of the sheets made from the kapok pulp mixed with the mixed softwood and hardwood pulps (SW: HW = 25:75) at different ratios (the sheet bottom side).





Table 4-56 to 4-58 conclude the overall statistical analysis results from Phase III. Table 4-56 is for the condition of the kapok pulp mixed with the hardwood pulp. Table 4-57 is the case that the kapok pulp was mixed with the softwood pulp while table 4-58 reveals the case when the kapok pulp was blended with the mixed softwood and hardwood pulps.

 
 Table 4-56 Summarized statistical analysis results from the kapok pulp mixed with with the hardwood pulp

Properties	P-value	Statistical analysis result
Caliper	0.191039	insignificant
Apparent density	0.055094	insignificant
Smoothness (bottom side)	0.000769	significant
Smoothness (top side)	0.000416	significant
Brightness	6.76E-06	significant
Opacity	0.000675	significant
Tear resistance	3.28E-06	significant
Tensile strength	4.1E-06	significant
Elongation	0.000658	significant
Burst strength	8.11E-05	significant

Note: if P-value was lower than 0.05, then the result was not statistically insignificant.

Properties	P-value	Statistical analysis result
Caliper	0.012222	significant
Apparent density	0.01573	significant
Smoothness (bottom side)	0.06267	insignificant
Smoothness (top side)	0.168347	insignificant
Brightness	8.06E-05	significant
Opacity	0.006268	significant
Tear resistance	8.81E-07	significant
Tensile strength	0.003173	significant
Elongation	0.001099	significant
Burst strength	0.949223	insignificant

Table 4-57 Summarized statistical analysis results from the kapok pulp mixed with the softwood pulp

Note: if P-value was lower than 0.05, then the result was not statistically insignificant.

 Table 4-58 Summarized statistical analysis results from the kapok pulp blended

 with the mixed pulp

Properties	P-value	Statistical analysis result
Caliper	0.131953	insignificant
Apparent density	0.084649	insignificant
Smoothness (bottom side)	0.00271	significant
Smoothness (top side)	0.001943	significant
Brightness	1.22E-05	significant
Opacity	0.001663	significant
Tear resistance	0.00012	significant
Tensile strength	6.28E-05	significant
Elongation	0.001126	significant
Burst strength	0.018646	significant

Note: if P-value was lower than 0.05, then the result was not statistically insignificant.

#### 4.5.4.2 SEM Micrograph

The SEM micrographs of the sheets made from the kapok pulp, hardwood pulp and softwood pulp are shown in Figures 4-34. The kapok fibers appeared to be long and quite uniform and contained high contact areas for bonding. The fibers were also collapsed and more conformable. The hardwood pulp had short fibers providing poorer bonding while the softwood pulp had long and irregular fibers offering high contact areas for bonding like the kapok pulp. It can be observed that increases in the kapok pulp ratios in the hardwood pulp resulted in improved fiber bonding as illustrated in Figures 4-35. For the softwood pulp, the effect was unnoticeable as shown in Figures 4-36.



Figure 4-34 SEM micrograph of the sheet made from (a) the kapok pulp, (b) the hardwood pulp, and (c) the softwood pulp.



Figure 4-35 SEM micrographs of the sheets made from the kapok pulp mixed with the commercial hardwood pulp at different ratios.

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Figure 4-36 SEM micrographs of the sheets made from the kapok pulp mixed with the commercial softwood pulp at different ratios.

#### **CHAPTER V**

#### CONCLUSIONS AND SUGGESTIONS

#### **5.1 Conclusions**

In this work, the kapok fibers were pulped using soda process. After evaluating the effects of sodium hydroxide on pulping, it can be concluded that when the sodium hydroxide dosage was increased, the freeness (Canadian standard method) was decreased. The numerical average fiber length decreased and the fines content increased by using higher sodium hydroxide dosage. Higher sodium hydroxide dosage also led to lower caliper but higher apparent density and Bekk smoothness. The sodium hydroxide dosage had little effect on brightness and opacity. The increasing of sodium hydroxide concentration caused lower tear index but higher tensile and burst strengths. The tensile index reached its maximum when using 20% sodium hydroxide dosage which was an optimum condition in this experiment. However, the tensile strength tended to drop when using excess sodium hydroxide. The sodium hydroxide dosage had little effect on zero-span tensile strength. Generally, kapok fibers are coated with cutin wax leading to high contact angle of the sheets. However, the contact angle of the kapok sheet decreased when increasing the sodium hydroxide dosage.

For the refining process, the pulp refined at the 10/1000 inch disc gap followed by the 7/1000 inch disc gap had too low freeness to be measured and make into handsheets. When pulp was once refined at 7/1000 inch disc gap, the numerical average fiber length was decreased and the fines content was increased and this caused higher smoothness of handsheets. The caliper, apparent density, brightness, and opacity were not different when using different disc gaps and numbers of refining pass. For strength properties, the sheets made from the pulp refined twice through the 10/1000 inch disc gap gave the higher water contact angle and strength properties, namely tear resistance, tensile strength, burst strength, and zero-span tensile strength.

The fiber length distribution and the numerical average fiber length of the kapok pulps were between the softwood and hardwood pulps but the kapok pulps had the lowest fines content. The zero-span tensile strength of the sheets made from kapok pulp was between the hardwood and softwood pulp. When kapok pulp was mixed with commercial pulps and the ratio of kapok pulp increased, Bekk smoothness and caliper of the sheets made from the mixed pulps were decreased but the apparent density was increased. The kapok pulp in this experiment was bleached through only single-stage so it had lower brightness than the commercial pulps that were bleached through multi-stage. When the amount of kapok pulp was increased in the mixed pulps, the brightness and opacity were decreased. The characteristics of the kapok fibers were brittle but it still provided high tensile and burst strengths. So, when kapok pulp was mixed with commercial pulps, it tended to increase tensile and burst strengths but lower tear resistance and elongation. The sheets made from the kapok pulp had the higher contact angles than the sheets from either softwood pulp or hardwood pulp. So, when the kapok pulp was added to the mixed pulps, the contact angles were increased.

Consequently, it was found from this research work that kapok pulp may be used or substituted commercial pulps in pulp and paper industry especially in packaging paper which requires tensile strength and water resistance. However, the lower tear resistance should be concerned. Paper containing kapok pulp may also provide good printability and good quality for oil-based printing inks.

## 5.2 Suggestions

Due to their hydrophobicity, kapok fibers had to be immersed in water for a minimum of 2 weeks in order to get the swollen fibers. Thus, this can be improved by treating the fibers with some type of surfactants or detergent to increase their hydrophilicity.

In this research, kappa number of the kapok pulp was not found to be an indication for the degree of delignification of the pulping process. Other methods such as chlorine consumption according to ISO 3260 may be used.

Measurement of the formation of the sheets may be beneficial in order to confirm the poor formation of sheets containing kapok fibers.

The contact angle measurement using a non-polar solvent should be performed if the kapok pulp is applied to be used in packaging.



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### APPENDICES

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### APPENDIX A



1. SEM micrographs of kapok fiber with different magnification



.

2. SEM micrographs of softwood fiber with different magnification

200X

500X

3. SEM micrographs of hardwood fiber with different magnification



#### APPENDIX B

n	NaOH Dosage (%)						
Properties	10	15	20	25			
Kappa Number	72.12	79.57	91.81	69.90			
Residual Alkali (g/L)	2.48	3.28	5.14	6.18			
Pulp Yield (%)	64.62	52.72	46.23	41.05			
Freeness (CSF, mL)	407	368	302	285			
Caliper (mm)	0.11	0.08	0.08	0.08			
Apparent Density (g/cm <sup>3</sup> )	0.60	0.78	0.80	0.86			
Bottom Smoothness (s)	2.83	6.33	7.51	8.04			
Top Smoothness (s)	5.27	10.05	10.59	12.28			
Brightness (%)	22.63	19.76	19.40	18.42			
Opacity (%)	98.13	97.47	97.48	96.91			
Tear Resistance (mN)	105.91	108.53	100.03	99.37			
Tear index (mN m <sup>2</sup> /g)	1.63	1.66	1.58	1.55			
Tensile Strength (N/m)	5163.36	6504.45	7153.34	6577.76			
Tensile index (Nm/g)	79.43	99.52	112.89	102.48			
Burst Strength (kPa)	256.48	376.15	374.64	382.58			
Burst index (kPa m <sup>2</sup> /g)	3.94	5.79	5.91	5.96			
Elongation (%)	1.67	2.00	2.09	2.12			
Zero Span (N/m)	3327.23	2885.78	3122.85	2959.35			
Zero Span Index (Nm/g)	55.45	48 10	52.05	49 32			

•

## Phase I : Effects of sodium hydroxide dosages on kapok pulp and paper properties (1<sup>st</sup> replicate)

	NaOH Dosage (%)						
Properties	10	15	20	25			
Kappa Number	73.36	92.93	107.85	92.25			
Residual Alkali (g/L)	2.11	3.07	4.97	6.12			
Pulp Yield (%)	60.17	49.78	43.14	39.87			
Freeness (CSF, mL)	396	357	290	269			
Caliper (mm)	0.09	0.08	0.08	0.07			
Apparent Density (g/cm <sup>3</sup> )	0.76	0.83	0.85	0.90			
Bottom Smoothness (s)	3.90	5.95	9.02	10.17			
Top Smoothness (s)	6.62	9.77	13.11	16.07			
Brightness (%)	19.89	18.48	17.79	17.68			
Opacity (%)	97.67	97.45	97.40	97.01			
Tear Resistance (mN)	127.49	109.83	102.64	105.91			
Tear index (mN m <sup>2</sup> /g)	1.92	1.69	1.62	1.63			
Tensile Strength (N/m)	5388.36	6504.45	6992.22	6491.11			
Tensile index (Nm/g)	81.24	101.30	110.17	100.07			
Burst Strength (kPa)	328.69	368.30	381.37	389.05			
Burst index (kPa m <sup>2</sup> /g)	4.95	5.68	6.01	6.00			
Elongation (%)	1.83	2.04	2.13	1.98			
Zero Span (N/m)	3809.55	4095.68	3678.75	3744.15			
Zero Span Index (Nm/g)	63.49	68.26	61.31	62.40			

.

Phase I : Effects of sodium hydroxide dosages on kapok pulp and paper properties (2<sup>nd</sup> replicate)

Properties	NaOH Dosage (%)			
	_10	15	20	25
Kappa Number	72.74	86.25	99.83	81.08
Residual Alkali (g/L)	2.29	3.18	5.05	6.15
Pulp Yield (%)	62.40	51.25	44.69	40.46
Freeness (CSF, mL)	401	362	296	277
Caliper (mm)	0.10	0.08	0.08	0.07
Apparent Density (g/cm <sup>3</sup> )	0.68	0.80	0.82	0.88
Bottom Smoothness (s)	3.37	6.14	8.27	9.10
Top Smoothness (s)	5.95	9.91	11.85	14.17
Brightness (%)	21.26	19.12	18.60	18.05
Opacity (%)	97.90	97.46	97.44	96.96
Tear Resistance (mN)	116.70	109.18	101.34	102.64
Tear index (mN m <sup>2</sup> /g)	1.78	1.68	1.60	1.59
Tensile Strength (N/m)	5275.83	6504.44	7072.78	6534.44
Tensile index (Nm/g)	80.33	100.41	111.52	101.27
Burst Strength (kPa)	292.58	373.20	378.00	385.81
Burst index (kPa m <sup>2</sup> /g)	4.44	5.73	5.96	5.98
Elongation (%)	1.75	2.01	2.10	2.05
Zero Span (N/m)	3568.39	3490.73	3400.80	3351.75
Zero Span Index (Nm/g)	59.47	58.18	56.68	55.86

.

## Phase I : Effects of sodium hydroxide dosages on kapok pulp and paper properties (Average result)
Descrition	Disc gap	o (inch)
Properties	10/1000*2	7/1000*1
Freeness (CSF, mL)	284	244
Caliper (mm)	0.073	0.074
Apparent Density (g/cm <sup>3</sup> )	0.79	0.80
Bottom Smoothness (s)	5.22	9.00
Top Smoothness (s)	9.33	13.08
Brightness (%)	18.51	19.41
Opacity (%)	96.66	96.61
Tear Resistance (mN)	92.84	88.91
Tear index (mN m <sup>2</sup> /g)	1.62	1.50
Tensile Strength (N/m)	6220.56	5627.23
Tensile index (Nm/g)	108.76	95.02
Burst Strength (kPa)	317.53	325.51
Burst index (kPa m <sup>2</sup> /g)	5.55	5.50
Elongation (%)	2.14	1.97
Zero Span (N/m)	3122.85	2861.25
Zero Span Index (Nm/g)	52.05	47.69

### Phase II : Effects of refining disc gaps and numbers of refining passes on kapok pulp and paper properties

Properties	Ratio of Kapok pulp (%)					
roperties	100	75	50	25	0	
Caliper (mm)	0.085	0.073	0.079	0.080	0.081	
Apparent Density (g/cm <sup>3</sup> )	0.78	0.88	0.83	0.82	0.8	
Bottom Smoothness (s)	5.98	10.54	15.60	18.22	18.74	
Top Smoothness (s)	9.22	15.81	21.20	26.28	26.55	
Brightness (%)	26.70	36.92	46.28	56.98	65.38	
Opacity (%)	96.14	98.06	99.47	99.38	99.52	
Tear Resistance (mN)	92.83	152.33	223.59	306.89	360.88	
Tear index (mN m <sup>2</sup> /g)	1.40	2.37	3.41	4.68	5.61	
Tensile Strength (N/m)	7011.11	6705.56	6269.45	5589.45	4311.11	
Tensile index (Nm/g)	106.02	104.21	95.62	85.37	67.11	
Burst Strength (kPa)	388.58	373.36	350.03	294.24	248.29	
Burst index (kPa m <sup>2</sup> /g)	5.87	5.81	5.34	4.49	3.87	
Elongation (%)	2.10	2.52	2.74	3.43	3.84	

Phase III : Effects of the kapok pulp when mixed with the commercial hardwood pulp on paper properties (1<sup>st</sup> replicate)

Properties	Ratio of Kapok pulp (%)					
rroperties	100	75	50	25	0	
Caliper (mm)	0.083	0.080	0.082	0.080	0.081	
Apparent Density (g/cm3)	0.80	0.84	0.81	0.83	0.80	
Bottom Smoothness (s)	7.33	11.99	17.70	21.22	18.49	
Top Smoothness (s)	10.76	16.66	24.40	29.62	27.79	
Brightness (%)	27.85	39.12	48.68	55.52	63.42	
Opacity (%)	96.89	98.06	98.93	99.29	99.49	
Tear Resistance (mN)	110.49	162.14	237.97	292.89	362.85	
Tear index (mN m <sup>2</sup> /g)	1.66	2.41	3.61	4.44	5.64	
Tensile Strength (N/m)	7051.67	6854.45	6183.33	5416.67	4297.78	
Tensile index (Nm/g)	105.84	101.90	96.94	82.16	66.87	
Burst Strength (kPa)	403.56	374.42	335.20	300.11	241.13	
Burst index (kPa m <sup>2</sup> /g)	6.05	5.56	5.09	4.55	3.75	
Elongation (%)	2.28	2.66	3.11	3.66	3.99	

Phase III : Effects of the kapok pulp when mixed with the commercial hardwood pulp on paper properties (2<sup>nd</sup> replicate)

Properties	Ratio of Kapok pulp (%)					
	100	75	-50	25	.0	
Caliper (mm)	0.084	0.077	0.080	0.080	0.081	
Apparent Density (g/cm3)	0.79	0.86	0.82	0.82	0.80	
Bottom Smoothness (s)	6.66	11.26	16.65	19.72	18.62	
Top Smoothness (s)	9.99	16.24	22.80	27.95	27.17	
Brightness (%)	27.27	38.02	47.48	56.25	64.40	
Opacity (%)	96.52	98.06	99.20	99.34	99.51	
Tear Resistance (mN)	101.66	157.23	230.78	299.89	361.87	
Tear index (mN m <sup>2</sup> /g)	1.53	2.39	3.51	4.56	5.63	
Tensile Strength (N/m)	7031.39	6780.00	6226.39	5503.06	4304.44	
Tensile index (Nm/g)	105.93	103.06	94.77	83.76	66.98	
Burst Strength (kPa)	396.07	373.89	342.61	297.18	244.71	
Burst index (kPa m <sup>2</sup> /g)	5.96	5.68	5.22	4.52	3.81	
Elongation (%)	2.19	2.59	2.92	3.54	3.91	

Phase III : Effects of the kapok pulp when mixed with the commercial hardwood pulp on paper properties (Average result)

Properties	Ratio of Kapok pulp (%)					
rroperties	100	75	50	25	0	
Caliper (mm)	0.085	0.083	0.083	0.088	0.090	
Apparent Density (g/cm <sup>3</sup> )	0.78	0.79	0.79	0.73	0.73	
Bottom Smoothness (s)	5.98	5.79	6.51	7.10	7.94	
Top Smoothness (s)	9.22	8.97	9.91	10.38	11.87	
Brightness (%)	26.70	31.99	39.78	47.61	56.96	
Opacity (%)	96.14	97.17	97.88	98.30	98.71	
Tear Resistance (mN)	92.83	302.05	508.64	630.89	800.22	
Tear index (mN m <sup>2</sup> /g)	1.40	4.59	7.77	9.81	12.21	
Tensile Strength (N/m)	7011.11	6941.11	6590.00	6133.89	5562.22	
Tensile index (Nm/g)	106.02	105.16	100.70	95.41	84.84	
Burst Strength (kPa)	388.58	399.75	385.16	382.38	400.41	
Burst index (kPa m <sup>2</sup> /g)	5.87	6.06	5.89	5.94	6.11	
Elongation (%)	2.10	2.47	2.50	2.76	2.92	

Phase III : Effects of the kapok pulp when mixed with the commercial softwood pulp on paper properties (1<sup>st</sup> replicate)

Properties	Ratio of Kapok pulp (%)					
rroperties	100	75	50	25	0	
Caliper (mm)	0.083	0.080	0.086	0.088	0.090	
Apparent Density (g/cm3)	0.80	0.83	0.77	0.74	0.73	
Bottom Smoothness (s)	7.33	6.11	6.83	7.62	7.77	
Top Smoothness (s)	10.76	9.40	9.50	10.44	10.52	
Brightness (%)	27.85	35.73	43.10	49.89	57.15	
Opacity (%)	96.89	97.83	98.10	98.36	98.94	
Tear Resistance (mN)	110.49	353.69	534.79	633.51	795.65	
Tear index (mN m <sup>2</sup> /g)	1.66	5.28	8.01	9.65	12.13	
Tensile Strength (N/m)	7051.67	7020.00	6855.56	6444.45	5993.34	
Tensile index (Nm/g)	105.84	104.62	102.75	98.38	91.36	
Burst Strength (kPa)	403.56	385.27	386.09	379.23	377.02	
Burst index (kPa m <sup>2</sup> /g)	6.05	5.74	5.79	5.78	5.75	
Elongation (%)	2.28	2.54	2.59	2.86	2.95	

Phase III : Effects of the kapok pulp when mixed with the commercial softwood pulp on paper properties (2<sup>nd</sup> replicate)

Properties	Ratio of Kapok pulp (%)					
rroperties	100	75	50	25	0	
Caliper (mm)	0.084	0.082	0.085	0.088	0.090	
Apparent Density (g/cm3)	0.79	0.81	0.78	0.74	0.73	
Bottom Smoothness (s)	6.66	5.95	6.67	7.36	7.85	
Top Smoothness (s)	9.99	9.19	9.71	10.41	11.19	
Brightness (%)	27.27	33.86	41.44	48.75	57.05	
Opacity (%)	96.52	97.50	97.99	98.33	98.83	
Tear Resistance (mN)	101.66	327.87	521.71	632.20	797.93	
Tear index (mN m <sup>2</sup> /g)	1.53	4.93	7.89	9.73	12.17	
Tensile Strength (N/m)	7031.39	6980.56	6722.78	6289.17	5777.78	
Tensile index (Nm/g)	105.93	104.89	101.72	96.90	88.10	
Burst Strength (kPa)	396.07	392.51	385.63	380.80	388.71	
Burst index (kPa m <sup>2</sup> /g)	5.96	5.90	5.84	5.86	5.93	
Elongation (%)	2.19	2.51	2.55	2.81	2.94	

Phase III : Effects of the kapok pulp when mixed with the commercial softwood pulp on paper properties (Average result)

Properties	Ratio of Kapok pulp (%)					
Properties	100	75	Ratio of Kapok provide   75 50   0.081 0.079   0.83 0.84   7.78 11.73   12.88 16.50   35.57 46.49   98.59 98.71   198.09 332.12   2.96 5.00   5933.34 6665.00   103.72 100.41   413.98 364.29   6.19 5.49	25	0	
Caliper (mm)	0.085	0.081	0.079	0.079	0.087	
Apparent Density (g/cm <sup>3</sup> )	0.78	0.83	0.84	0.82	0.74	
Bottom Smoothness (s)	5.98	7.78	11.73	14.64	15.42	
Top Smoothness (s)	9.22	12.88	16.50	20.74	22.18	
Brightness (%)	26.70	35.57	46.49	55.35	63.02	
Opacity (%)	96.14	98.59	98.71	99.23	99.28	
Tear Resistance (mN)	92.83	198.09	332.12	462.87	632.86	
Tear index (mN m <sup>2</sup> /g)	1.40	2.96	5.00	7.21	9.88	
Tensile Strength (N/m)	7011.11	6933.34	6665.00	5750.00	5001.11	
Tensile index (Nm/g)	106.02	103.72	100.41	89.37	78.07	
Burst Strength (kPa)	388.58	413.98	364.29	317.81	319.18	
Burst index (kPa m <sup>2</sup> /g)	5.87	6.19	5.49	4.94	4.98	
Elongation (%)	2.10	2.52	2.88	3.33	3.71	

## Phase III : Effects of the kapok pulp when blended with the commercial mixed pulp (1<sup>st</sup> replicate)

Note: the commercial mixed pulp was obtained from mixing 25% of softwood pulp with 75% of hardwood pulp

Properties	Ratio of Kapok pulp (%)					
	100	75	50	25	0	
Caliper (mm)	0.083	0.079	0.078	0.080	0.081	
Apparent Density (g/cm3)	0.80	0.81	0.83	0.80	0.79	
Bottom Smoothness (s)	7.33	10.83	12.85	14.31	15.00	
Top Smoothness (s)	10.76	15.14	18.90	19.57	20.27	
Brightness (%)	27.85	39.24	48.64	56.55	64.11	
Opacity (%)	96.89	97.96	98.98	99.17	99.44	
Tear Resistance (mN)	110.49	220.32	360.88	493.60	550.48	
Tear index (mN m <sup>2</sup> /g)	1.66	3.45	5.62	7.66	8.58	
Tensile Strength (N/m)	7051.67	6693.34	6304.45	5486.67	4831.11	
Tensile index (Nm/g)	105.84	104.74	98.16	85.10	75.36	
Burst Strength (kPa)	403.56	355.00	339.20	320.53	290.56	
Burst index (kPa m <sup>2</sup> /g)	6.05	5.56	5.28	4.97	4.53	
Elongation (%)	2.28	2.83	3.19	3.41	3.67	

## Phase III : Effects of the kapok pulp when blended with the commercial mixed pulp (2<sup>nd</sup> replicate)

Note: the commercial mixed pulp was obtained from mixing 25% of softwood pulp with 75% of hardwood pulp

D	Ratio of Kapok pulp (%)					
Properties	100	75	50	25	0	
Caliper (mm)	0.084	0.080	0.078	0.080	0.084	
Apparent Density (g/cm <sup>3</sup> )	0.79	0.82	0.84	0.81	0.76	
Bottom Smoothness (s)	6.66	9.31	12.29	14.48	15.21	
Top Smoothness (s)	9.99	14.01	17.30	20.16	21.23	
Brightness (%)	27.27	37.40	47.56	56.00	63.57	
Opacity (%)	96.52	98.27	98.84	99.20	99.36	
Tear Resistance (mN)	101.66	209.21	346.50	478.24	591.67	
Tear index (mN m <sup>2</sup> /g)	1.53	3.21	5.31	7.43	9.23	
Tensile Strength (N/m)	7031.39	6813.33	6484.72	5618.33	4916.11	
Tensile index (Nm/g)	105.93	104.23	99.28	87.23	76.71	
Burst Strength (kPa)	396.07	384.49	351.75	319.17	304.87	
Burst index (kPa m <sup>2</sup> /g)	5.96	5.87	5.38	4.96	4.76	
Elongation (%)	2.19	2.67	3.03	3.37	3.69	

### Phase III : Effects of the kapok pulp when blended with the commercial mixed pulp (Average result)

Note: the commercial mixed pulp was obtained from mixing 25% of softwood pulp with 75% of hardwood pulp

VITA

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