

THE EFFECT OF CHEMICAL COMPOSITIONS
AND BEATING TIME ON THE PRODUCTION
OF CELLULOSIC PACKAGING MATERIALS
(PULP) USING OIL PALM EMPTY FRUIT
BUNCH (EFB)

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DEGREE OF BACHELOR OF CHEMICAL
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ABSTRACT

In palm oil mill processing, the waste product from palm oil mill was identified consists of oil palm trunks (OPT), oil palm fronds (OPD), empty fruit bunches (EFB), palm pressed fibers (PPF) and palm kernel shells. Follow Prasertsan, during palm oil processing, more than 70% by weight of fresh fruit bunches were left over as a waste. Analysts estimate that processing of palm oil mill yield 17 million tones of empty fruit bunches per year. Researcher believed that empty fruit bunches has potential as packaging product since its content high cellulose. Due to high user of non-degradable packaging, government put the full effort to find the alternative ways to reduce the use of non-degradable packaging. Empty fruit bunches was see as an alternative to make degradable packaging product. In order to make packaging product from empty fruit bunch, soda antraquinone (soda AQ) and sodium hydroxide (NaOH) is used as chemical pulping because this chemical pulping will produce the better properties of pulp to make the packaging product. Sample of pulp was made at different soda composition and at different time of beating process in order to get optimum condition of properties of packaging product. After the best sample was selected, the standard physical testing was carried out. The study shows that beating effect and different composition of soda produces pulp at different fiber morphology, strength and packaging properties.

ABSTRAK

Dalam pemrosesan kilang minyak sawit, produk sisa terdiri daripada batang kelapa sawit, pelepah kelapa sawit, tandan buah kosong, gentian sawit dan tempurung isirung sawit. Mengikut Prasertan dan Prasertan, semasa pemrosesan minyak sawit, lebih daripada 70% berat tandan buah segar ditinggalkan sebagai sisa. Penganalisis menganggarkan bahawa pemrosesan kilang minyak sawit menghasilkan 17 juta tan tandan buah kosong. Penyelidik percaya bahawa tandan buah kosong mempunyai potensi sebagai produk pembungkusan kerana mengandungi kandungan selulosa yang tinggi. Disebabkan pengguna yang tinggi dalam pembungkusan bukan-degradasi, kerajaan meletakkan usaha untuk mencari cara-cara alternatif untuk mengurangkan penggunaan pembungkusan bukan-degradasi. Tandan buah kosong telah dilihat sebagai alternatif untuk membuat produk pembungkusan terurai. Untuk membuat pembungkusan produk daripada tandan buah kosong, soda natrium hydroxide dan antraquinone soda digunakan sebagai pulpa kimia kerana pulpa kimia ini akan menghasilkan sifat-sifat pulpa yang lebih baik untuk membuat produk pembungkusan. Contoh pulpa telah dibuat pada komposisi soda yang berbeza dan pada masa yang berlainan semasa proses pemukul untuk mendapatkan keadaan optimum sifat-sifat produk pembungkusan. Selepas sampel yang terbaik telah dipilih, ujian fizikal telah dijalankan. Kajian menunjukkan bahawa kesan pemukul dan komposisi soda yang berbeza menghasilkan pulpa yang berbeza dari segi morfologi gentian, kekuatan dan ciri-ciri pembungkusan.

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LIST OF ABBREVIATION

%	Percent
°C	Degree C
AQ	Antraquinone
Ca	Calcium
CPO	Crude palm oil
CSF	Canadian Standard Freeness
DCE	Direct contact evaporator
EFB	Empty fruit bunch
FFB	Fresh fruit bunch
g	Gram
H ₂ SO ₃	Sulforous acid
HCL	Hydrochloric acid
HSO ₃ ⁻	Bisulfite ion
K	Potassium
kg	kilogram
kPa	Kilopascal
m	Metre
Mg	Magnesium
min	Minute
ml	Mililitre
mN	Milnewton
MnT	Million tones
N	Newton
N	Nitrogen
Na ₂ S	Sodium sulfide
Na ₂ S ₂ O ₃	Sodium thiosulfate
NaOH	Sodium hydroxide
NDCE	Indirect contact evaporator
OPF	Oil palm frond
OPT	Oil palm trunk
P	Phosphorous
SO ₂	Sulfur dioxide
SR	Schopper-Riegel
w	Weight
α	alpha

CHAPTER 1

INTRODUCTION

1.1 RESEARCH BACKGROUND

Malaysia is one the largest producers of palm oil and was estimated that the amount of EFB available in a year is about 4.43 million tones (dry wt) (Joseph and Chandran, 2002). The EFB alone contributes around 19.5 million tons in 2008 and these were returned to the plantation for mulching at 60 tons per hectare (Omar E *et al.*, 2011). The primary solid wastes of palm oil mill processing are 23% empty fruit bunch, 14% fibers and 7% shells of oil palm per ton of fresh fruit bunch (FFB) (Omar E *et al.*, 2011). The wastes was produced by sterilization the oil palm fresh fruit with steam and then trashed in rotating drum to remove the fruit from its bunch. Currently, palm oil mills reuse mainly as biomass to produce fuel while waste from palm oil recycle back to the plantation as fertilizer.

Despite the huge production, the oil consist only a minor fraction of the total biomass produced in the plantation. The remaining consists of a huge amount of lignocellulosic materials such as fronds, trunk and empty fruit bunches. Therefore, huge amount of waste generated and largely unutilized. World pulp consumption was about

300 million tons in 1996/1997 by the year 2010(MPOB 2006). Besides that, the wood sources from the forest decrease every year, so that; the source for pulp and pulp production has been decreased. In view of the shortage of conventional raw material for pulping and the increasing of demand of pulp products worldwide, non-wood plants and agricultural residues attracted renewed interest. Then, the oil palm industry must be prepared to take advantage of the situation and utilize the available biomass in the possible manner.

As the alternative way to reuse this waste, the packaging product from EFB was introduced. The main component to make the packaging from empty fruit bunch is cellulose. The EFB contains important constituents such as lignin 18%-23% (w/w), α -cellulose 35% (w/w) and hemicelluloses 25% (w/w) (Abdul Azis *et al.*, 1989; Yojiro *et al.*, 1990; Mohamad *et al.*, 1999; Rosnah *et al.*, 2000). Holocellulose contains a composite of hemicelluloses and cellulose. The EFB is extracted using acidified sodium chloride method to produce the holocellulose and then, the cellulose is separated from holocellulose using 17.5% sodium hydroxide solution.

Packaging made from non-degradable plastic which can not be disposed mainly used in food and pharmaceutical industries over the years until now. Over the years, this non-degradable packaging plastic got a lot of attention from government and become one of the most important issues to environment. This product cannot be disposed in the soil for a long time and cannot break down naturally and produced 'white pollution' to our environment. Due to this situation, the search for alternative packaging product has gained importance. Package-related environmental pollution and disposal problems researches are also done to limit these problems (Rhim *et al.*, 2007). Packaging product empty fruit bunches are potential element to degradable product and compatible with the environment due to increasing awareness among the public of the disadvantage on using non-degradable material as packaging product. Biodegradable has its own benefits in different ways that is environmental friendly has always been the top priority whenever it is applied. These environmentally-friendly packaging materials help reduce our dependence on foreign oil, reduce global warming, and help reduce greenhouse gases. In addition, they divert tons of plastic and other non-degradable materials from

our landfills. Until now, there are variety of active packaging technology have been developed to produce better quality product.

Over 67 million tones of packaging waste are generated annually comprising about one-third of all municipal solid waste (Klingbeil, 2000). When discarded, food packaging can become the most obvious source of litter generated by the public. This has caused increasing environmental concern and creates the new regulation for packaging in order to reduce amounts of packaging waste.

In recent years, development of biodegradable packaging material from renewable natural resources has received widespread government support and many national or international organizations have been established to facilitate the development in this area. The objectives in the development of biodegradable packaging are to utilize renewable and potentially more sustainable sources of raw material and to facilitate integrated waste management approaches as to reduce landfill. To date, significant technological development has been achieved to produce biodegradable materials for packaging applications with comparable functionalities to those of traditional oil-based plastic packaging.

1.2 PROBLEM STATEMENT

Plastic disposal is a huge problem the world over, and one that is only set to grow in the coming years. We use well over 4 million tones of plastic in the UK alone, of which packaging accounts for about 35%. Most of this packaging goes into our household waste and so into landfill. How long that plastic takes to degrade depends on what type it is, but some estimates say that a plastic carrier bag will take over 1000 years to break down. In recent year, in order to manage this problem, the traditional petroleum-based packaging was replaced with bioplastic packaging. Bioplastics are derived from things like corn starch, pea starch and vegetable oil. Bioplastics are meant to be compostable, meaning that they will break down quickly either in a landfill or a home compost heap. It sound like perfect solution but bioplastics come with their own problem. Problem with bioplastics is their production is still reliant upon fossil fuels and biofuel. Just like with biofuels, crops must be grown to provide raw material to make the packaging product. As alternative solution for this entire problem, the empty fruit bunches have been used to make packaging product. By using EFB in packaging product, waste from palm oil mill can be manage with converted it to valuable product and at the same time can provide additional income to the plantation. The excessive amount of biomass waste produced by Malaysian country is surprisingly high. As we know, about 43.4 million tones of EFB were produced in a year. With this amount, there must be problem to plant to put this waste material. So, they provide new product from empty fruit bunch to manage this waste. Packaging product from empty fruit bunch are more easily to degradable and break down in a landfill other than plastic.

1.3 OBJECTIVE OF RESEARCH

1. To investigate the potential of cellulose as the packaging material
2. To study effect different chemical compositions on pulping process
3. To study effect beating time on tensile strength, burst, tear index and breaking length

1.4 SCOPE OF STUDY

1. Selected the best pulp when different chemical composition used during pulping process
2. Checking the composition contain in EFB
3. The effect of beating on strength properties
4. The optimum condition of beating time on strength properties
5. EFB as a main raw material

1.5 BENEFIT AND SIGNIFICANT OF RESEARCH

1. Environmental friendly
2. Alternative way to produce valuable product from EFB
3. Reduce the huge production of biomass residue
4. Contribute to the society
5. Reduce deforestation and environmental problem
6. Reduces the growth in the consumption of plastic product

CHAPTER 2

LITERATURE REVIEW

2.1 BACKGROUND

The oil palms (*Elaeis guineensis*) comprise two species of the Arecaceae or palm family. Mature trees are single-stemmed and grow up to 20m tall. The leaves are pinnate and reach between 3 or 5m long. The flowers are produced in dense clusters; each individual flower is small with three sepals and three petals. The fruit takes 5-6 months to mature from pollination to maturity. Maturity means it comprises an oily, fleshy outer layer (the pericarp), with a single seed (kernel), also rich in oil. Oil palms are commonly used in commercial agriculture in the production of palm oil. The oil palm is a tropical palm tree therefore it can be cultivated easily in Malaysia. The oil palm tree in Malaysia originated from West Africa where it was growing wild and later developed into an agricultural crop. The first commercial oil palm estate in Malaysia was set up in 1917 at Tennamaran Estate, Selangor (MPOB, 2006). The growth of the industry has been phenomenal and Malaysia is now the largest producer and exported of palm oil in the world. About 52% or 26.3 million tones (MnT) of the total world oils and fats exported in years 2006. Nowadays, Malaysia is one of the largest palm-oil produce and currently produces around 6.5 million metric tons of world palm oil production (Law, Jiang., 2001). At present, the total area under oil palm cultivation is

about 3.5 million hectares, while the statistic of oil palm production for the year 2001 was 11.8 million tonnes (Hussin, Mokhtar, Wan, Ropandi.,2002).

2.2 PALM OIL BIOMASS

It is reported that Malaysia produces 30 million tons annually of oil palm biomass, including trunks, fronds, and empty fruit bunch (MPOB, 2001). Malaysia produced about 13.2 million tons of oil palm biomass including trunk, fronds, and empty fruit bunches (Kamaruddin et.al., 1997). About 5.8 ton of fresh fruit bunch (FFB) produces 1 ton of crude palm oil (CPO) (Pleanjai et al., 2004). Several by products such as fibre (30%), shell (6%), decanter cake (3%) and empty fruit bunch (EFB) (28.5%) are also produced during processing of oil from FFB (Pleanjai et al., 2004). Despite the huge production of oil palm production, oil consists of only a minor fraction of the total biomass produced in the plantation. The remainder consist huge amount of lignocellulosic materials in the form of fronds, trunks and empty fruit bunches. Therefore, a lot of waste from oil-palm biomass produce make it cannot be handle properly and give a lot of side effect to the environment and the country. The burn method has been replaced by the no-burn technique, which is both environmentally friendly but cause a lot of problem to the plantation (Noor, 2003). About 9.9 million tons of palm oil wastes are generated every year in Malaysian alone, and this keep increasing at 5% annually (Yang et al., 2006). Biomass may vary in its physical and chemical properties due to its diverse origin and species. However, biomass is structurally composed of cellulose, hemicellulose, lignin, extractives and inorganics. (Husin, Ridzuan,2002).

2.3 CHARACTERISTIC OF OIL PALM BIOMASS

2.3.1 Nutrient Composition of Palm Oil Biomass

The major structural chemical components with high molar masses are carbohydrate polymers and oligomers and lignin. Minor low-molar-mass extraneous materials mostly organic extractives and inorganic minerals are also present in biomass. The major constituents consist of cellulose (a polymer glucosan), hemicelluloses (also called polyose), lignin, organic extractives, and inorganic minerals. Oil palm biomass

contains quite significant amount of organic nutrient, which contributes to its fertilizer values such as the data below.

Table 2.1: Nutrient composition of oil palm biomass

Oil palm biomass	Dry matters (tonne/hectare)	Nutrient (kg/hectare)			
		nitrogen	phosphorous	Potassium	Magnesium
Trunk	75.5	368.2	35.5	527.4	88.3
Fronds (replanting)	14.4	150.1	13.9	193.9	24.0
Fronds (pruning)	10.4	5.4	10.0	139.4	17.2
Empty fruit bunch (EFB)	1.6	107.9	0.4	35.3	2.7

Sources: (<http://www.bfdic.com>)

The oil palm biomass contains about 18 – 21% of lignin, and 65-80% of holocellulose (α -cellulose and hemicellulose), which are more or less comparable with that of other wood or lignocellulosic materials.

Table 2.2: Proximate analysis of biomass of oil palm biomass (% , dry weight)

composition	Oil palm trunk (OPT)	Oil palm fronds (OPF)	Empty fruit bunch (EFB)
Lignin	18.1	18.3	21.2
Hemicellulose	25.3	33.9	24.0
A-cellulose	45.9	46.6	41.0
Holocellulose	76.3	80.5	65.5
Ash	1.1	2.5	3.5
Alcohol-benzene solubility	1.8	5.0	4.1

Source: (<http://www.bfdic.com>)

2.3.2 Properties of Empty Fruit Bunches

Oil palm empty fruit bunches were used as an alternative raw material to obtain cellulosic pulp. Pulping was done by using high-boiling point organic solvents of decreased polluting power relative to classical (Kraft, sulphite) solvents but affording operation at similar pressure levels. Empty Fruit Bunch is composed of 45-50% cellulose and about equal amounts (25-35%) of hemicellulose and lignin (Deraman 1993). Due to oil palm empty fruit bunch is available in large quantities and has fairly high cellulose contents. So empty fruit bunch fiber is appears to be a potential substrate for enzyme and other chemical production. (Deraman, 1993). The empty fruit bunches (EFB) are the solid residue that is produced in the highest amount from the fresh fruit bunches (FFB) of oil palm. The lignocellulosic biomass contained in both EFB and PPF can be used for ethanol production. In the case of ethanol from EFB, the lignocellulosic content must be previously in form of fibers to be used. Typically the empty fruit bunch (EFB) on a dry basis is 120 to 260 kg for one fresh fruit bunch (EFB) on an as received basis (Arrieta FRP et al, 2007). Given the current yield of FFB and the area of harvested production, Colombia produces 1 Mt of EFB, at a rate of 438 km² (Mesa J, 2009).

Table 2.3: nutrient contain of EFB

Nutrient content of EFB	Composition as a percentage of dry matter
Nitrogen (N)	0.44
Phosphorous (P)	0.144
Potassium (K)	2.24
Magnesium (Mg)	0.36
Calcium (Ca)	0.36

Source: (<http://www.etawau.com>)

2.4 CHEMICAL PULPING

Chemical pulping is the process to dissolve the lignin in the wood to create a pulp and the most common method in pulping process. Chemical pulping creates higher sheet strength than mechanical pulping. However yields 40 to 50 percent pulp, where mechanical pulping yields 95 percent pulp. There are two types of chemical pulping which is sulfate pulping (most commonly known as Kraft pulping) and sulfite pulping. In Kraft pulping, pulp that product has higher sheet strength compare to sulfite pulping. However, sulfite pulp is easier to bleach and can produce more bleach pulp. It also easier refines process for pulpmaking. The major different in chemical pulping is the types of chemical used to dissolved the lignin contain in fibres (MacDonald, Ronald G, 1969).

2.4.1 Kraft Pulping

Kraft pulping creates dark brown pulp which is used for boxes, pulp bags, and wrapping pulp. Kraft pulp can also be used for writing pulp and pulpboard when bleached, and for diapers when fluffed. There are three main steps involved in Kraft pulping which are digestion, washing and chemical recovery (MacDonald, Ronald G, 1969).

2.4.1.1 Digestion

The first step in pulping wood is to “cook” the wood chips. A digester, heated by steam, “cooks” the wood chips in white liquor (a mix of sodium hydroxide (NaOH) and sodium sulfide (Na₂S) until done. The cooking process dissolves most of the lignin and only some of the hemicellulose, leaving mostly cellulose to hold the fibers together. The digester system may be a batch or a continuous process (MacDonald, Ronald G, 1969).

2.4.1.2 Washing

Washing removes weak black liquor from the pulp which is sent to the chemical recovery process. This also prevents contamination during subsequent processing steps.

Types of washers used include rotary vacuum washer (most common type of washer), diffusion washers, rotary pressure washers, horizontal belt washers, wash press, and dilution/extraction. All the washer types use water (fresh or recycled) and are usually placed in series to achieve higher removal efficiency. The rinsed pulp is screened for oversize particles and then excess water is removed. This is done in a gravity thickener (more commonly known as a decker) (MacDonald, Ronald G, 1969).

2.4.1.3 Chemical recovery

The reason Kraft pulping is economically successful is that the used cooking liquor can be recovered and reused in the chemical recovery process. The first step in recovering the chemicals from the black liquor is evaporation. This removes excess water from the black liquor and maximizes the fuel value for the recovery furnace. There are two types of evaporators generally used in the chemical recovery process: direct (DCE) and indirect (NDCE) contact evaporators. Some types of DCE include the multiple-effect evaporator (most common), flash evaporation and thermocompressor evaporation. DCE use heat from direct contact with the recovery furnace flue gases, while NDCE uses indirect contact. Black liquor oxidation is needed after DCE, but not after NDCE. After DCE, the black liquor is normally oxidized with air to control the sulfide level and prevent the release of odorous compounds. This is done by countercurrently passing the black liquor through an air stream using a porous diffuser, sieve tray tower, packed tower or agitated air sparge. The oxidation reaction converts sodium sulfide (Na_2S) to sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) (MacDonald, Ronald G, 1969).

After NDCE or black liquor oxidation, the black liquor is then forced through spray nozzles into the recovery furnace, where it is burned providing heat to generate steam. This also conserves the inorganic chemicals, which create a molten smelt on the floor of the furnace. The molten smelt, composed of sodium sulfide and sodium carbonate, is drained from the recovery furnace hearth through smelt spouts. In a smelt dissolving tank, the smelt is quenched with water, producing green liquor. Sodium carbonate from the smelt is then converted to sodium hydroxide in the causticizer by adding calcium hydroxide. The calcium carbonate resulting from the reaction precipitates from the solution and is collected and sent to the lime kiln where it is

converted to lime (calcium oxide). The calcium oxide is then slaked to produce calcium hydroxide for reuse in the causticizer (MacDonald, Ronald G, 1969).

2.4.2 Sulfite pulping

Sulfite pulping produces a lighter pulp than Kraft pulping. It can be used for newsprint and when bleached can be used for writing pulps and for the manufacture of viscose rayon, acetate filaments and films, and cellophane. Sulfite pulping follows many of the same steps as Kraft pulping. The major difference in sulfite pulping is that the digester “cooks” with a mixture of H_2SO_3 (sulfurous acid) and HSO_3^- (bisulfite ion in the form of calcium, magnesium, sodium, or ammonium bisulfate). The pulp continues on through the same processes as in the Kraft pulping process. However, the chemicals separated from the pulp in the washers may or may not go into a recovery process. Chemical recovery in sulfite pulping is practiced only if it is economical. If chemical recovery does occur the liquor goes through an evaporator and then to a recover furnace. Here, smelt is not formed, but ash and SO_2 are formed. (MacDonald, Ronald G, 1969).

2.4.2.1 Bleaching

The purpose of the bleaching process is to enhance the physical and optical qualities (whiteness and brightness) of the pulp by removing or decolorizing the lignin. Two approaches are used in the chemical bleaching of pulps. One approach called brightening, uses selective chemicals, such as hydrogen peroxide, that destroy chromatographic groups but do not attack the lignin. Brightening produces a product with a temporary brightness (such as newspulp) that discolors from exposure to sunlight or oxygen. The other approach (true bleaching) seeks to almost totally remove residual lignin by adding oxidizing chemicals to the pulp in varying combinations of sequences, depending on the end use of the product. This creates a longer lasting (sometimes permanent) whiteness, but it weakens the fibers and reduces sheet strength. The most common bleaching and brightening agents are chlorine, chlorine dioxide, hydrogen peroxide and sodium hydroxide (MacDonald, Ronald G, 1969).

Typically, the pulp is treated with each chemical in a separate stage. Each stage includes a tower, where the bleaching occurs; a washer, which removes bleaching chemicals and dissolved lignins from the pulp prior to entering the next stage; and a seal tank, which collects the washer effluent to be used as wash water in other stages or to be sewerred. Bleaching processes use various combinations of chemical stages called bleaching sequences (MacDonald, Ronald G, 1969).

The first stage in the bleaching process is the chlorination stage, whose primary function is to further delignify the pulp. Chlorine reacts with lignin to form compounds that are water-soluble or soluble in an alkaline medium, which aids in delignifying the pulp before it proceeds to the next bleaching stage (MacDonald, Ronald G, 1969).

The next stage after chlorination is typically the extraction stage. This stage and the remaining stages serve to bleach and whiten the delignified pulp. The extraction stage removes the chlorinated and oxidized lignin by solubilization in a caustic solution. Chlorine dioxide is often used in bleaching, either in the chlorination stage (as a substitute for some of the chlorine usage - chlorine dioxide substitution) or as an additional chlorine dioxide stage. Chlorine dioxide has 2.63 times greater oxidizing power (on a pound per pound basis) than chlorine and is used for nearly all high brightness pulps (MacDonald, Ronald G, 1969).

The next stage is the actual bleaching stage. Hypochlorite is a true bleaching agent that destroys certain chromophobic groups of lignin. It also attacks the pulp so high cellulose degradation occurs in Kraft pulp. Application of hypochlorite to Kraft pulp is usually used only as an intermediate stage of the sequence or to produce semi-bleached pulps. In the bleach process, residual chlorine must be removed through washing in vacuum washers (MacDonald, Ronald G, 1969).

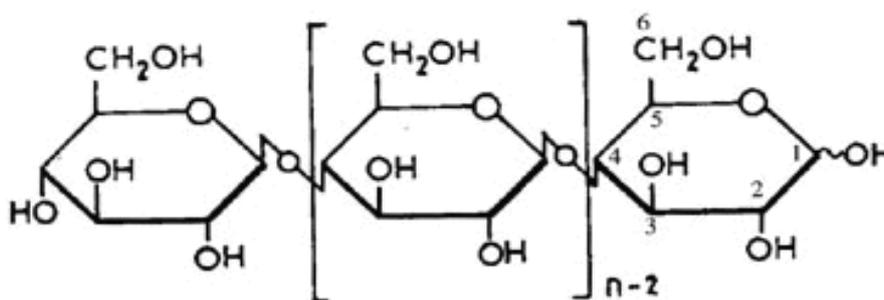
2.5 CELLULOSE

2.5.1 Chemistry of cellulose

Chemistry of cellulose is primarily the chemistry of alcohols and it form many derivatives of alcohol such as esters, ethers, etc. These derivatives form the basis for industrial technology of based cellulose. Cellulose derivatives are used commercially in transient intermediates or as permanent product. Figure 1 below show the molecular structure of cellulose.

Cellulose does not melt or dissolve in common solvent due to the strong hydrogen bonds that occur between cellulose chains. Thus, it is difficult to convert the short fibers from wood pulp into the continuous filaments that need in artificial silk. According Pyen (1838), cellulose has been shown to be a long chain polymer with repeating units of D-glucose which is a simple sugar. In the cellulose chain, the glucose units are in 6-membered rings called pyranoses. There are joined by single oxygen atoms called acetal linkages between the C-1 of one pyranoses ring and the C-4 of the next ring.

2.5.2 Structure and properties



Cellulose

Figure 1: molecular structure of cellulose