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ENZYMATIC SCOURING OF VARIOUS FABRICS

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for the Degree of Master of Science in Applied Polymer Science and Textile Technology

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กระบวนการกำจัดสิ่งสกปรกด้วยสารเคมีก่อนการย้อมผ้า เพื่อให้ผ้าสามารถดูดซึมน้ำได้ดีขึ้น จำเป็นต้องทำที่อุณหภูมิสูงประมาณ 80-100°C และยังก่อให้เกิดของเสียอันตรายอีกด้วย งานวิจัยนี้จึงได้ทดลองนำเอนไซม์มาใช้กำจัดสิ่งสกปรกบนผ้าฝ้าย ผ้าพอลิเอสเทอร์ และผ้าฝ้ายผสมพอลิเอสเทอร์ เอนไซม์ที่ใช้ คือ เอนไซม์ไลเปสจาก Procine Pancreas เอนไซม์โปรทีเอสจาก Aspergillus oryzae และเอนไซม์เซลลูเลสจาก Aspergillus niger ซึ่งมีแอคติวิตี 15 หน่วย/กรัม 14,000 หน่วย/กรัม และ 25,000 หน่วย/กรัม ตามลำดับ ผ้าที่ผ่านการกำจัดสิ่งสกปรกด้วยเอนไซม์แล้วได้ถูกนำไปทดสอบหาสมบัติต่างๆ ของผ้าตามมาตรฐานการทดสอบ เช่น การดูดซึมน้ำ น้ำหนักที่สูญเสียไป ความแข็งแรง ความขาว ความสามารถในการย้อมติดสี และสมบัติอื่นๆ และเปรียบเทียบผลกับผ้าที่ผ่านการกำจัดสิ่งสกปรกด้วยวิธีที่ใช้ในปัจจุบัน ผลการทดสอบพบว่าการกำจัดสิ่งสกปรกบนผ้าด้วยเอนไซม์ให้ผลเป็นที่น่าพอใจ และให้ผลใกล้เคียงกับการกำจัดสิ่งสกปรกบนผ้าด้วยวิธีที่ใช้ในปัจจุบัน โดยผ้าที่ผ่านการกำจัดสิ่งสกปรกด้วยเอนไซม์มีความสะอาดและสามารถดูดซึมน้ำได้ทันที ซึ่งภายหลังการกำจัดสิ่งสกปรก ผ้ามีการสูญเสียน้ำหนักไปประมาณร้อยละ 0.2-1.0 หากแต่ผ้าที่ได้กลับมีความแข็งแรง ความขาว และนุ่มเพิ่มขึ้น นอกจากนี้ ยังพบว่าการใช้เอนไซม์ไลเปส โปรทีเอส และเซลลูเลส เดี่ยวๆ เพื่อกำจัดสิ่งสกปรกบนผ้าฝ้าย หรือ ผ้าฝ้ายผสมพอลิเอสเทอร์ไม่สามารถทำให้ผ้าดูดซึมน้ำได้ทันที จึงได้แบ่งการกำจัดสิ่งสกปรกออกเป็น 2 ขั้นตอน โดยขั้นตอนแรกทำการกำจัดสิ่งสกปรกบนผ้าด้วยเอนไซม์ไลเปส โปรทีเอส หรือไลเปสผสมโปรทีเอส แล้วตามด้วยการกำจัดสิ่งสกปรกด้วยเอนไซม์เซลลูเลสในขั้นที่สอง ส่วนการกำจัดสิ่งสกปรกบนผ้าพอลิเอสเทอร์สามารถกระทำได้อย่างมีประสิทธิภาพดีโดยใช้เอนไซม์ไลเปสชนิดเดียว

ภาควิชาวัสดุศาสตร์ ลายมือชื่อนิสิต..
สาขาวิชาวิทยาศาสตร์พอลิเมอร์ประยุกต์ฯ ลายมือชื่ออาจารย์ที่ปรึกษา..
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Prior to dyeing, a fabric is required to scour in a chemical solution in order to remove the impurities and to improve the fabric absorbency. A conventional scouring is usually conducted at high temperatures (80-100°C) and produces a chemical waste pollutant. In this work, various enzymatic scouring processes were introduced for scouring various fabrics, cotton; polyester; and cotton/polyester blends. Three enzymes including lipase from Procine Pancreas, protease from Aspergillus oryzae and cellulase from Aspergillus niger, were used and their activities were 15 units/g, 14,000 units/g, and 25,000 units/g, respectively. For a comparison, various conventional scouring processes were also carried in this experiment. After scouring, the fabrics were tested for the water absorbency, the weight loss, the strength, the whiteness, the dyeability, and other properties, according to the standard test methods. It was found that the enzymatic scouring results were very impressive and were comparable to the conventional scouring results. The enzymatic scoured fabrics were clean and absorbed water instantaneously. Although they lost 0.2-1.0% of the fabric weight after scouring, they gained strength and whiteness. In addition, the process made the fabric soften as well. Lipase, protease, and cellulase were less effective for scouring cotton or cotton/polyester blends when each enzyme was used alone. To successfully scour these fabrics, two scouring steps were needed by scouring with lipase, protease or lipase and protease in the first step and scouring with cellulase in the second step. Lipase could successfully scour the polyester fabric and acquire desirable fabric properties.

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Academic year 2002

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CHAPTER 1

Introduction

Greige fabrics usually do not absorb water, instead, they repel it. This is because there are some hydrophobic substrates covering the fiber surface. These substrates include natural waxes, fats, oils, pectins, and others and they must be removed by scouring processes before the fabric can be dyed, printed or finished. After passing a complete scouring, the fabric contains an adequate absorbency and is ready for coloring or finishing. There are various scouring procedures for various fiber types. For examples, cotton and cotton blends are required to scour at high temperatures such as 80 -100 ^{o}C and need caustic soda as a scouring agent with an addition of a wetting agent and a sequestering agent. Polyester is scoured at around 60 ^{o}C using soda ash as a scouring agent. These conventional scouring processes produce large quantities of waste water and require high consumptions of water and energy.

Enzymatic scouring is another alternative for cleaning the fabric before coloring and finishing. This process consumes less energy and produces unharmful waste water.

In this study, three enzymes containing lipase, protease, and cellulase were used for scouring cotton, cotton/polyester blends, polyester, and nylon fabrics. The fabrics were then tested for properties in order to determine the effectiveness of these enzymatic scouring processes, and compared with the conventional scouring processes.

CHAPTER 2

Literature Survey

2.1 Cotton Fiber

Cotton is one of the most important and a widely used fiber in the textile industry. It is a single cell fiber and develops from the epidermis of the seed [1]. Raw cotton has a creamy tint off-white color. It is smooth and soft, very absorbent and cool touching when the impurities have been removed.

The mature cotton fiber forms a flat ribbon varying in width between 12 and 20 μm. It is highly convoluted and the number of convolutions varies between 4 to 6 per mm., reversing in the direction for every millimeter along the fiber length. These characteristics make cotton easy to recognize under both optical and electron microscopes (Figure 2.1) [2]. The cross section bean-shape of the fiber is described as a bilateral structure shown in Figure 2.2.

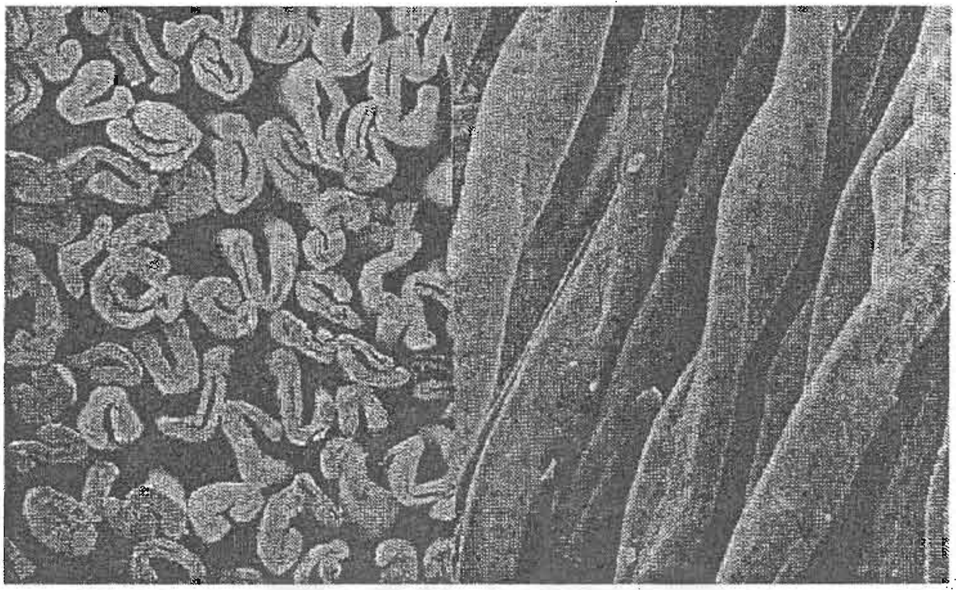


Figure 2.1 Scanning electron micrograph of raw cotton fibers [2].

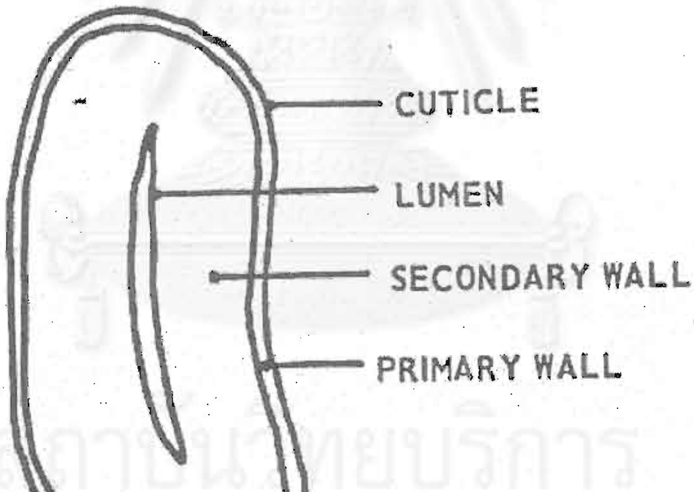


Figure 2.2 Bilateral structure of mature cotton [3].

2.1.1 Cotton Morphology

Cotton fiber has a fibrillar structure as illustrated in Figure 2.3. The outermost layer of the fiber is the thin waxy cuticle, which protects the fiber from its environment. Beneath this layer is the primary wall of the fiber cell, which is composed of fine threads of cellulose laid down during growth and spiraled round the longitudinal fiber axis at an angle of about 70° [4]. Winding layer is the very first layer of the secondary thickening and differs somewhat in structure from either the primary wall or the remainder of the secondary wall. Secondary wall consists of concentric layers of cellulose which constitute the main portion of cotton fiber. Lumen is the central cavity or canal of the fiber and lumen wall appears to be more resistant to certain reagent than secondary wall layer. It is highly irregular in both size and shape and often contains solid dried matter, largely nitrogeneous in compositions [1].

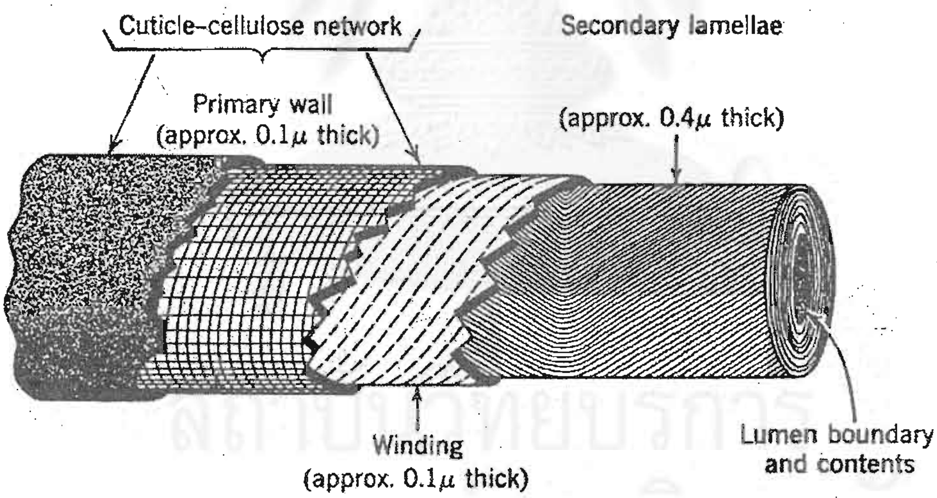


Figure 2.3 Schematic diagram of cotton fiber structure [1].

2.1.2 Molecular Structure of Cotton Fiber

Cotton consists of practically pure cellulose and may be chemically described as poly(1,4-β-D-anhydroglucopyranose) (Figure 2.4). The helical reversal structure of natural cellulose shows the constantly recurring cellobiose unit, consisting of two glucose units each with six carbon atoms. The length of the unit cell along the fiber axis is 10.4 Å calculated from the cellobiose unit. In natural cellulosic fibers, there are 3,000-5,000 glucose units joined together. This corresponds to a molecular weight of the order of 300,000- 500,000 [5].

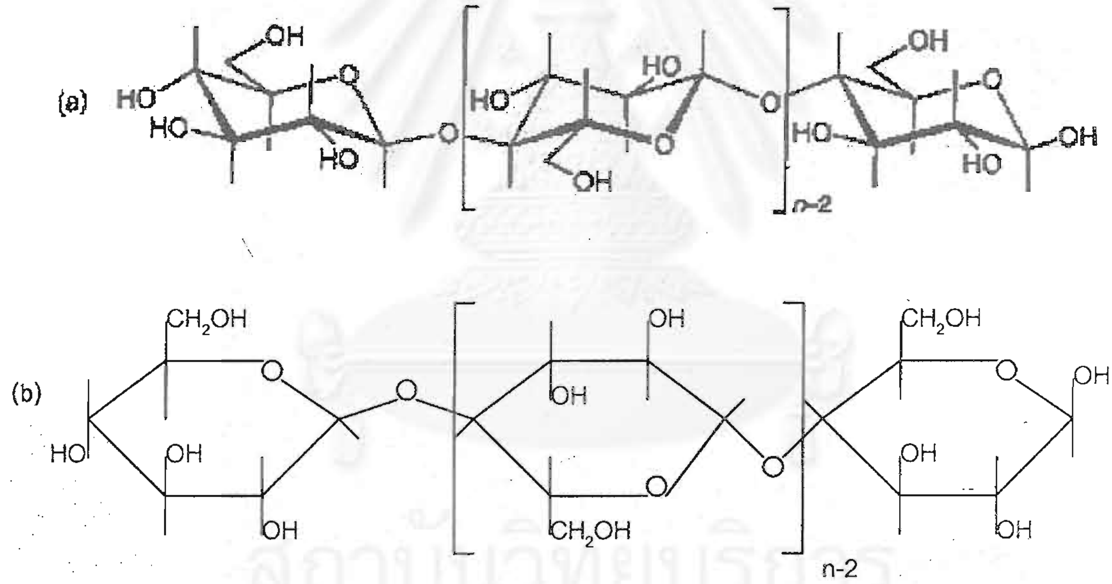


Figure 2.4 Cellulose: (a) fully extended conformational formula; (b) the Haworth projection formula. n= degree of polymerization (DP) [2].

2.1.3 Properties of Cotton Fiber

The properties of cotton fiber are listed in Table 2.1

Table 2.1 Properties of cotton fiber [6].

Microscopic Features	
Length (cm):	0.3 to 5.5 (depending on the source)
Cross-section:	Kidney shaped
Color:	Usually a creamy off white color
Light reflection:	Low luster, dull appearance
Physical Properties	
Tenacity (g/den):	3.0 to 5.0 (dry) 3.6 to 6.0 (wet)
Elongation (%):	3 to 7% elongation at break At 2% elongation, recovery is 70%
Moisture content (%) at 65%RH, 21 ^{o}C:	8.5%
Resilience:	Low
Abrasion resistance:	Fair to good
Specific gravity:	1.54
Chemical Properties	
Bleaches:	Highly resistant to all bleaches
Dyeability:	Good affinity for dyes. Dyeable with direct, reactive, vat, and sulfur dyes.

Table 2.1 (Continued)

Acids and alkalies:	Highly resistant to alkalies. Strong acids and hot dilute acids cause fiber damage.
Organic solvents:	Resistant to most organic solvents.
Stain:	Poor resistant to water-borne stains.
Sunlight and heat:	Good resistant to high temperatures. Prolonged exposure to light causes yellowing due to oxidation.
Biological Properties	
Fungi and molds:	Highly susceptible to attack by mildew.
Insects:	Starch cotton is attacked by silverfish.
Flammability Behavior	Burn rapidly. Smoldering red after glow.
Electrical and Thermal Conductivity	Good heat conductor

2.1.4 Constituents of Raw Cotton

The idealized constructions of raw cotton are illustrated in Figure 2.5 . Cotton consists of cellulosic and noncellulosic materials. The noncellulosic components found in mature cotton fibers are located in the cuticle and the primary cell wall. The surface layers, which contain lipids, waxes, pectins, organic acids, protein/nitrogeneous substances, noncellulosic polysaccharides and other unidentified substances, constitute approximately 10% of the total fiber weight [7-10]. The chemical composition of a mature cotton fiber is presented in Table 2.2.

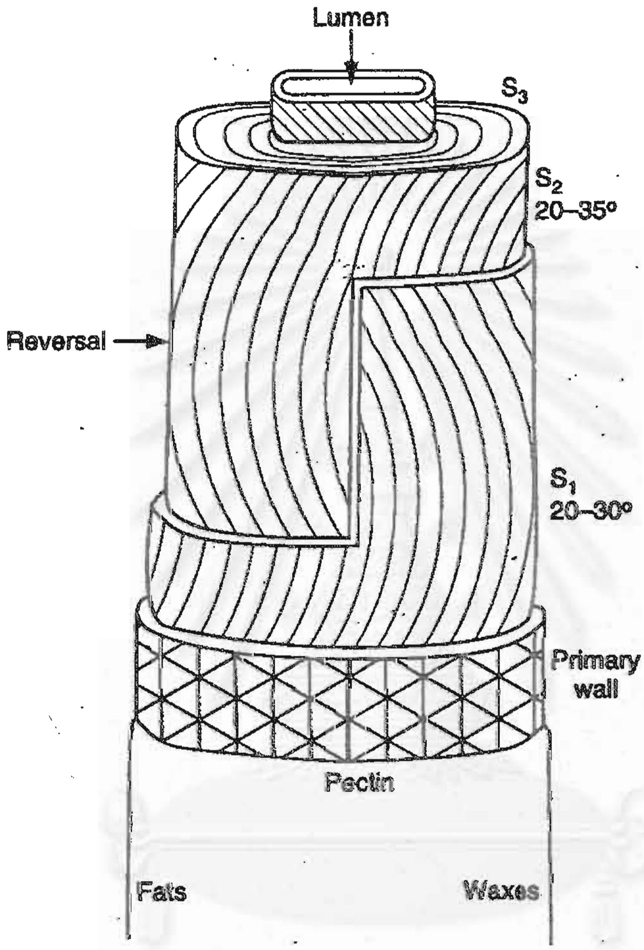


Figure 2.5 Idealized diagram of cotton morphology [2].

Table 2.2 The composition of a mature dry cotton fiber [11].

Constituent	Composition of Cotton Fiber			Composition of the Cuticle(%)
	Typical(%)	Low (%)	High(%)	
Cellulose	94.0	88.0	96.0	
Protein	1.3	1.1	1.9	30.4
Pectin	0.9	0.7	1.2	19.5
Waxes and Oils	0.6	0.4	1.0	17.4
Minerals	1.2	0.7	1.6	6.5
Maleic, citric and other organic acids	0.8	0.5	1.0	
Total sugars	0.3			
Cutin				8.7

2.1.4.1 Oils and Waxes

Cotton fiber contains approximately 0.5% of oils and waxes. The wax of cotton fiber is chemically complex containing acids and alcohols of high molecular weight [12]. Analyses of cotton [1] indicate that it contains all the even-number of carbon primary alcohols and the largest amount is n-triacotanol ($C_{30}H_{61}OH$). It also contains all the even-number of fatty acids from C_{24} to C_{34}. The one occurring in the largest amount is n-tetracosanoic acid ($C_{23}H_{47}COOH$). Small amounts of fatty acids such as palmitic, stearic, and oleic are found in waxes. The properties of cotton waxes from the extraction of Texas cotton with hot benzene are as follows [13]:

Melting point	68 to 71°C
Specific gravity	0.959
Saponification value	70.6
Acetyl value	73.1
Iodine value	24.5

Percentage of fatty acids	25.0
Percentage of unsaponifiable matter	69.0

2.1.4.2 Pectins

Pectin content in mature cotton fibers is about 0.6 to 1.2% depending on the method of determination. It is difficult to extract pectin quantitatively from the fiber. The amount of pectins on fiber can be roughly indicated by an estimation of uronic acids [5].

Pectins are high-molecular weight carbohydrates with chain structures similar to cellulose and consist of chains of α-1,4-linked D-galacturonic acid units shown in Figure 2.6. Cellulose breaks down into glucose but pectins decompose to give galactose, several pentoses, poly-galacturonic acid and methyl alcohol. Available evidence [14] indicates that pectins may occur in cotton fiber in the form of insoluble calcium, magnesium, and ion salts of the poly-galacturonic acid. It is insoluble in water but soluble in alkaline solutions.

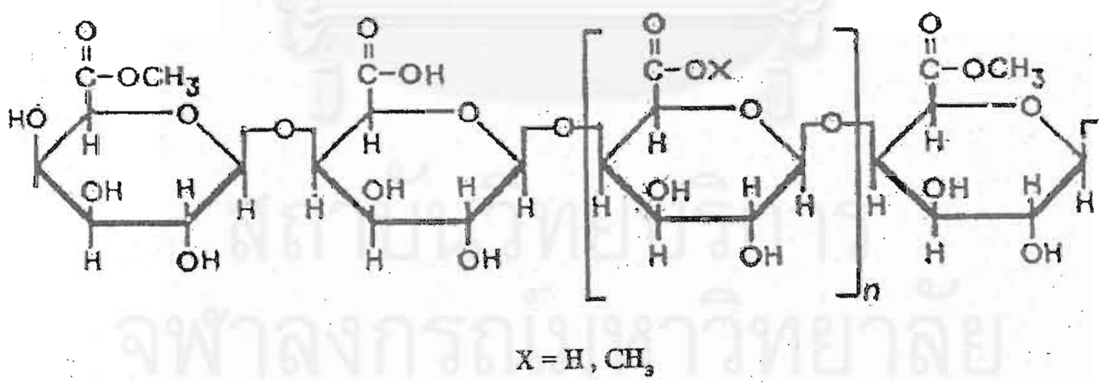


Figure 2.6 Molecular structure of pectins [15].

2.1.4.3 Minerals

Cotton may contain between 1 and 1.8 percent of mineral matter. Its quantity and composition vary according to the nature of the soil on which the plant was cultivated. Silicon is always present and other elements such as iron, aluminium, calcium and magnesium are also found. When cotton is ashed, all the metallic organic salts appear as carbonates. Analysis of the ash shows the presence of the following [16]:

Potassium carbonate	44.8%
Potassium chloride	9.9%
Potassium sulphate	9.3%
Calcium sulphate	9.0%
Calcium carbonate	10.6%
Magnesium sulphate	8.4%
Ferric oxide	3.0%
Aluminium oxide	5.0%

The carbonates of potassium and calcium are not in that state originally, but are products of the combustion of organic salts of those metals.

2.1.4.4 Nitrogen Compounds

Cotton contains approximately 1% of nitrogen impurities. Unless removed, they can produce undesirable effects in the finished material. These compounds consist essentially of degraded products of the protoplasm, which cell contained when it was still living and growing. They are the composition of the outer primary wall. Their exact identity has not been established but it is reasonable to assume that they are protein and polypeptides left in lumen after cell dies.

2.1.4.5 Coloring Matter

When waxes and nitrogen impurities have been removed, cotton still has a yellowish or brown discoloration. This is caused by the natural coloring matter, which can only be removed effectively by oxidizing agent in bleaching step. It presents only traces and its composition has not been established with certainty. It may be related to the pigments of cotton flowers. The nature of the pigment responsible for coloring is not known.

2.1.4.6 Ash

Cotton taken directly from the gin shows approximately 2 to 3% ash content. Analysis of the ash shows that it consists mainly of magnesium, calcium, or potassium carbonates, phosphates, sulfates, or chloride with the carbonates predominating.

2.2 Polyester Fiber

Polyester is formed by the interaction of small bifunctional molecules containing hydroxyl or carboxyl groups, commonly by the esterification of a dibasic organic acid with a dihydric alcohol. The investigations in this field reported in the published papers of Carothers of Du Pont were extented during the period 1939 to 1941 by Whinfield and Dickson [17]. The simplest and important of these was poly(ethylene terephthalate) commonly known as PET, and the inventors gave the name Terylene to this substance and the fiber made from it. Commercial production of PET fiber began in the United States in 1953 [6].

PET fibers are now manufactured throughout the world and are marketed under many tradenames. Modified forms of the polymer are also produced as well as some other polyester fibers based on different di-acids or diols. The information given as following refers principally to fibers spun from the unmodified homopolyester of ethylene glycol and terephthalic acid.

2.2.1 Production of Polyester Fiber

PET is the most widely used polyester fiber. It may be produced by a stepwise polycondensation reaction between ethylene glycol and terephthalic acid, as shown in Figure 2.7. However, a purer product is achieved by reacting the dimethyl ester of therephthalic acid with ethylene glycol.

After polymerization, the polymer is extruded in the form of ribbon and then cut into chips. The chips are dried and conveyed to a hopper, from which they are fed to the melt spinning tank (Diagram 2.1). The melted substrate is fed through the spinneret and solidifies into fiber form upon contact with air. The filaments are drawn to increase crystallinity, and wound on bobbins.

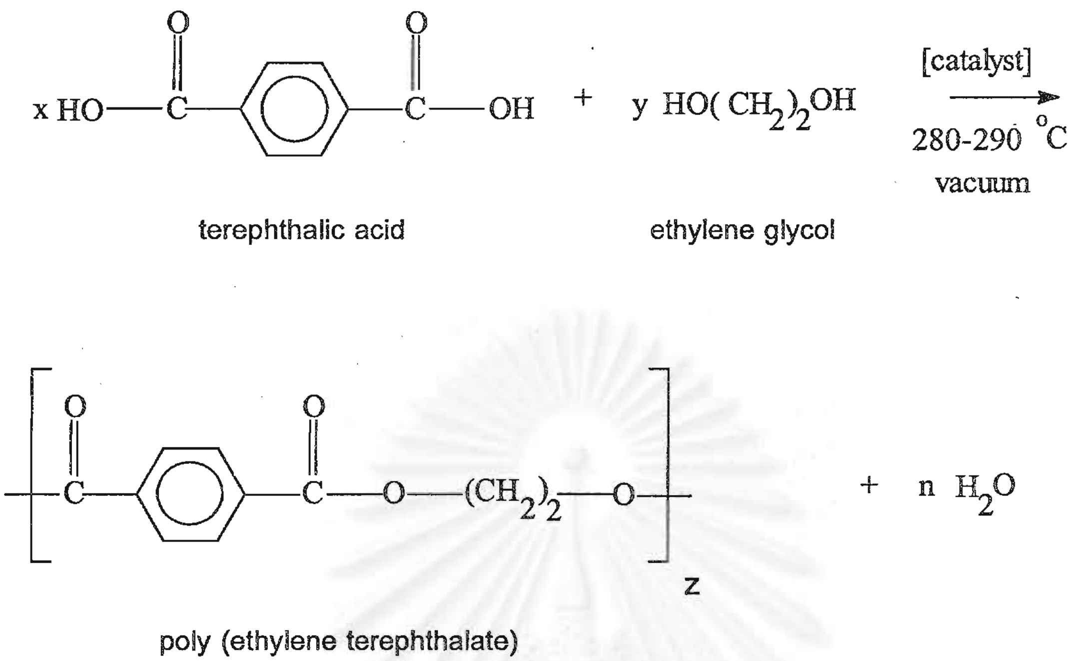


Figure 2.7 Chemical reactions in polyester formation [18].

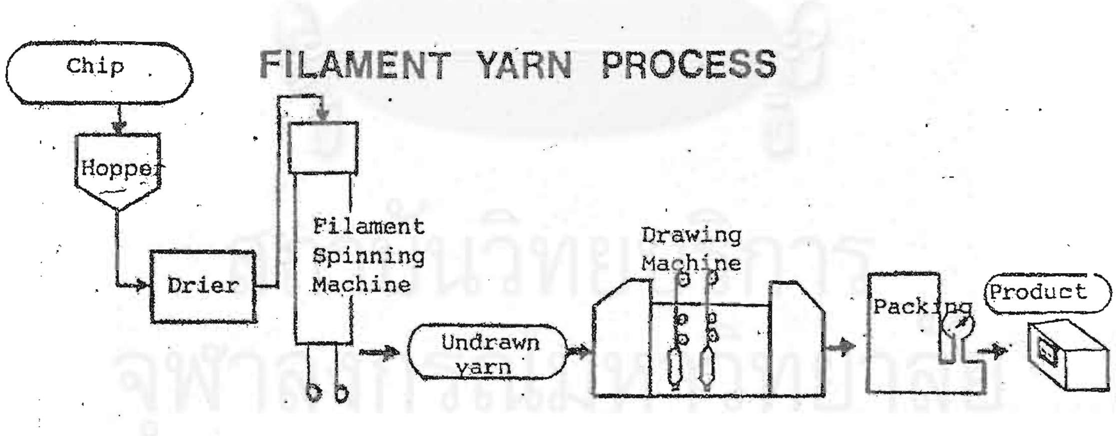


Diagram 2.1 Filament yarn process of polyester [19].

2.2.2 Molecular Structure of Polyester fiber

Polyester fiber consists of organic molecular units linked through ester groups. Its repeating unit along the chain is 10.75 Å and the successive ester groups are essentially in the trans configuration of each other (Figure 2.8) [6]. The properties of polyester are determined, to a great extent, by the aromatic rings and ester groups that make up the molecule. The ring structures are much more hydrophobic than the linear hydrocarbons in the polyamides. In addition, the ester groups are not as polar as amide groups. Both of these factors indicate that polyester should have a very low affinity for water; in fact the standard moisture regain for polyester is less than 1 percent. In addition, the smooth surface of the fibers reduces the wicking action of water over the surface. Thus, polyester fabrics, particularly those made from untextured filament yarns, are somewhat uncomfortable in a lack of moisture absorbency, unfamiliar skin contact sensations and static-related problems [20].

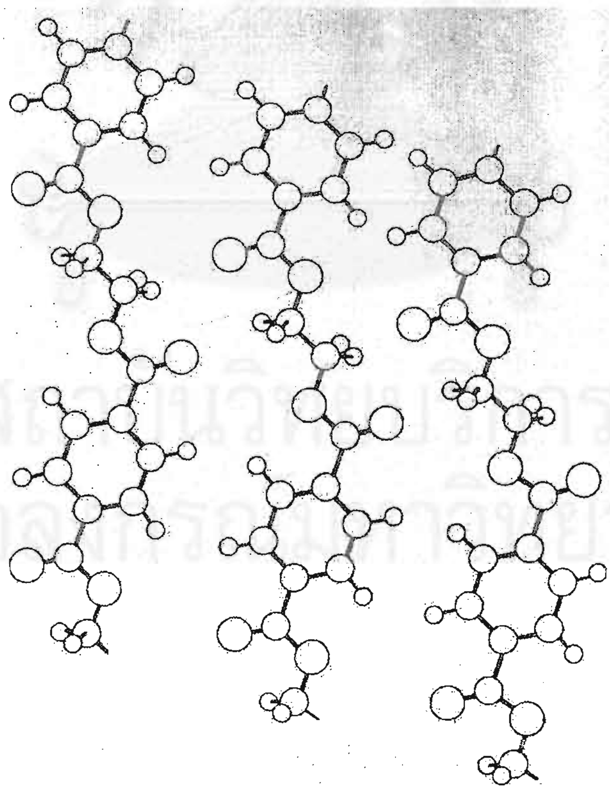


Figure 2.8 The crystal structure of PET (top view) [6].

2.2.3 Properties of Polyester Fiber

The properties of polyester fiber are listed in Table 2.3

Table 2.3 Properties of polyester fiber [6].

Physical Properties	
Tenacity(g/den):	4.5 to 5.0. As high as 8 for high-tenacity fibers.
Stretch and elasticity:	20 to 30% elongation at break; 97 to 100% recovery at 2% elongation.
Resiliency:	Excellent to good wrinkle recovery.
Abrasion resistance:	Exceptionally good.
Dimensional stability:	If properly heat-set will not shrink or stretch.
Moisture regain:	0-0.4% at standard conditions.
Specific gravity:	1.38
Chemical Properties	
Effects of bleaches:	Not affected by oxidizing or reducing bleaches.
Acids and alkalies:	Good resistance to almost all common acids. Hot concentrated sulfuric acid will cause deterioration.
Organic solvents:	Not affected by organic solvents.
Sunlight and heat:	Good resistance to sunlight, if behind glass. Prolonged exposure causes deterioration. Low temperatures should be used for ironing.
Resistance to stains:	Good resistance to water-borne stains. Oil-borne stains may be difficult to remove.
Dyeability:	Wide range of shade can be produced which have good to excellent colorfastness in water and fair to good fastness in light.

Table 2.3 (Continued)

	Disperse and azoic dyes, and some pigments are used.
Biological Properties	
Effects of fungi and molds:	Resists mildew.
Effects of insects:	Do not damage.
Flammability Behavior	Burns slowly; will shrink away from flame, yet exhibit melt drip.
Electrical and Thermal Conductivity	Will not conduct electricity.

2.3 Polyester/Cotton Blends

In an attempt to improve the consumer acceptance of polyester in apparel, the Du Pont Company developed techniques for blending polyester and cotton in the same yarn; 65 percent polyester and 35 percent cotton. The product was improved in comfort and contained better dyeability, while maintaining the wrinkle resistance and dimensional stability of 100 percent polyester [6]. Cotton contributes a high level of moisture absorption to the blend as well as better hand. The major disadvantage of the blend is that cotton absorbs water-borne stains, while polyester absorbs oil-borne stains. The blended fabrics are more difficult to clean than either 100 percent polyester or 100 percent cotton.

Under competitive pressure from the polyester manufactures, cotton producers found that suitable fabrics may be produced from a wide range of polyester/cotton blend ratios. Today the most popular ratios for apparel are polyester/cotton: 65/35, 50/50, and 35/65 blends, although blends as high as 80/20 or as low as 20/80 have proved useful.

2.4 Nylon Fiber

Nylon fiber is a fiber-forming substance. It consists of long polyamide chains.

In 1927, the Du Pont Company created a research group, under the direction of Wallace Hume Carothers, to study high-molecular-weight materials.

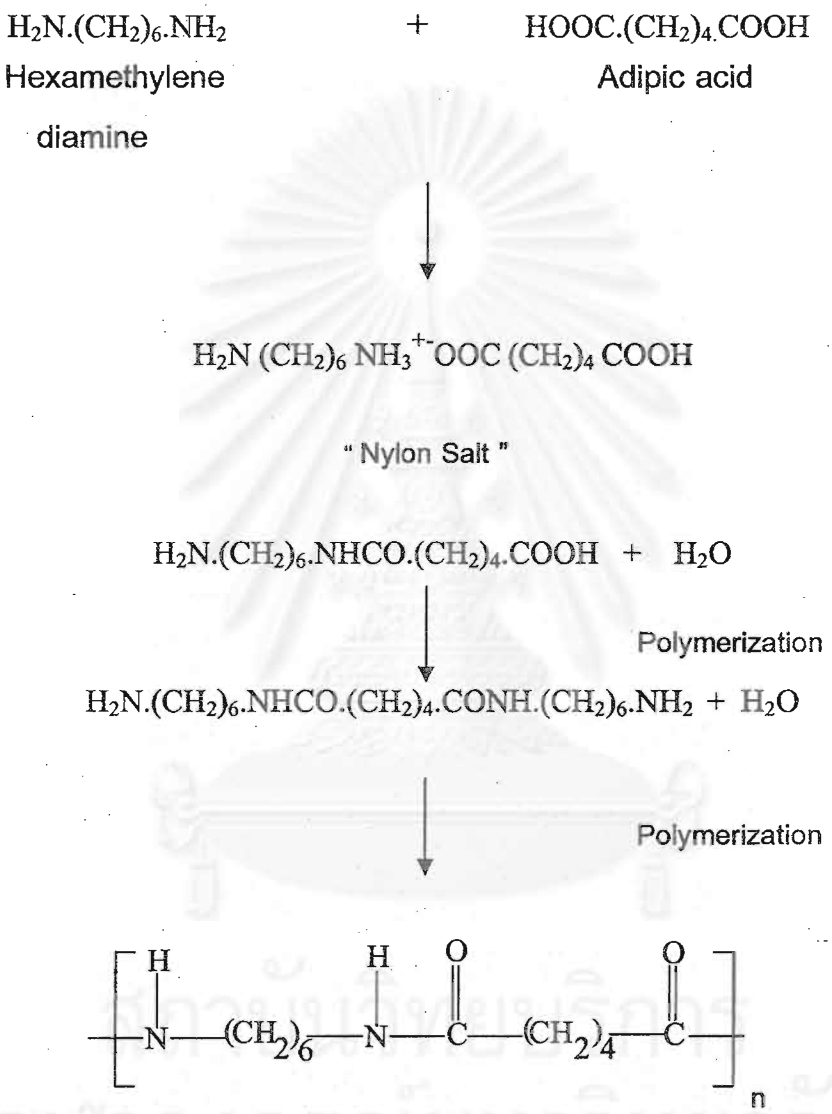
By 1930, Carothers and his associates had performed a feat that had eluded researchers for centuries. They had created a textile fiber. Over the next nine years, they had developed the processes, techniques, and equipment for manufacturing a synthetic polyamide in commercial quantities. They also gave it the name **nylon**. Accompanied by a well-planned marketing and advertising campaign, knitted nylon hosiery for women was introduced to the public in early 1940 [6].

The nylon production for textiles is confined to two major polymer types, namely nylon 6, 6 and nylon 6.

2.4.1 Production of Nylon Fiber

2.4.1.1 Nylon 6, 6

The original and still the most important polyamide is nylon 6, 6. It is produced from hexamethylene diamine and adipic acid by a stepwise polycondensation reaction (Figure 2.9). Hexamethylene diamine and adipic acid are reacted to form *nylon salt*. The salt is then polymerized under N_2 pressure and high temperatures to form long-chain molecules of polymer. During polymerization, water always is eliminated. After polymerization, the polymer is extruded from the reactor, cooled, and broken into flakes. In the process known as melt spinning, the flakes from many batches are blended and remelted. The molten material is extruded through a spinneret into a stream of cold air. The hot nylon cools to form filaments, which are stretched to orient the molecules and increase crystallinity, and wound on bobbins. The filament may also be cut to staple length and spun into yarn.



nylon 6, 6

n= degree of polymerization

Figure 2.9 The formation of nylon 6, 6 from hexamethylene diamine and adipic acid [16].

2.4.1.2 Nylon 6

Another commercially important nylon is nylon 6. It is more widely used in Europe than in the United States. Nylon 6 has properties similar to those of nylon 6, 6. It is manufactured by polymerizing *caprolactam*, extruding the polymer to form a flake, blending and remelting the flake, and extruding the hot melt into a cold air stream. The preparation of nylon 6 is shown in Figure 2.10.

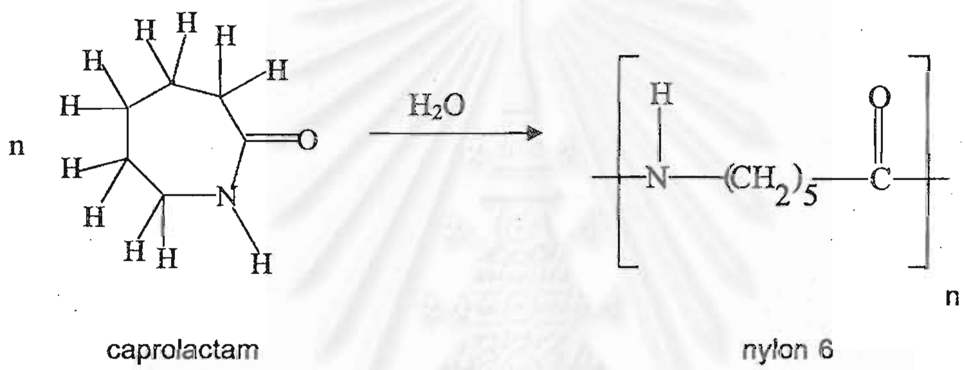


Figure 2.10 The preparation of nylon 6. The 7-membered rings of caprolactam open and polymerize to form linear polymer chains [6].

2.4.2 Molecular Structure of Nylon Fiber

Nylon 6,6 (Figure 2.11) and nylon 6 are composed of linear hydrocarbons held together by amide links. The hydrocarbon portions are *hydrophobic* ; that is, they reject water. The amide groups are *hydrophilic*. The hydrophobic portion of the molecule predominates, so that nylons have a rather low moisture regain. This is a liability as far as comfort is concerned, but is an advantage in that nylons are not readily stained by water-borne materials such as fruit juices or coffee.

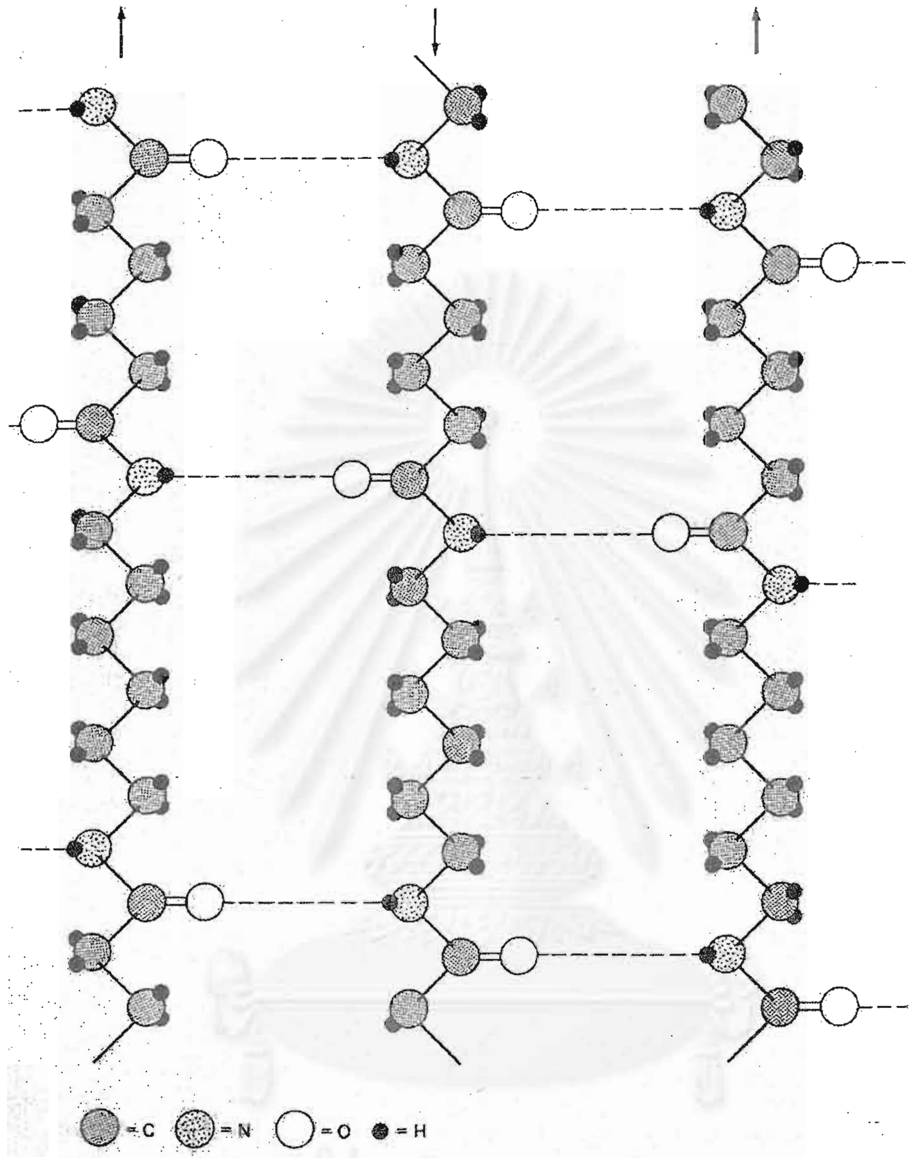


Figure 2.11 Crystal structure and hydrogen bonding in nylon 6, 6 [6].

2.4.3 Properties of Nylon Fiber

The properties of nylon fiber are listed in Table 2.4

Table 2.4 Properties of nylon fiber [6].

Physical Properties	
Tenacity(g/den):	4.6 to 5.8; high-tenacity nylon may be as 9.
Stretch and elasticity:	High elasticity and good elongation. 30% elongation at break, 100% recovery at 2% elongation.
Resiliency :	Good; good wrinkle-resistant properties.
Abrasion resistance:	Excellent; good resistance to flexing.
Dimensional stability:	Can be heat-set to maintain shape. Will maintain shape if heat-set temperature is not exceeded 150°C.
Moisture regain:	4.2 to 5% at standard conditions. Nylon 6 is slightly higher than nylon 6, 6.
Specific gravity:	1.14
Chemical Properties	
Effects of bleaches:	Not affected by oxidizing and reducing bleaches but may be harmed by chlorine and strong oxidizing bleaches.
Acid and alkalies:	Weakened by strong acids; not affected by alkalies.
Solvent:	Resists dry-cleaning solvents and reagents used in spot and stain removers.
Sunlight and heat:	Loses strength on prolonged exposure to sunlight. Good resistance to heat.
Dyeability:	Acid, direct, vat, disperse, basic, and

Table 2.4 (continued)

	metalized dyes are used.
Biological Properties	
Effects of fungi and molds:	Resistant to mold and fungi.
Effects of insects:	Not attacked by insects.
Flammability Behavior	Burns slowly, self-extinguishing. Melts and drips.
Electrical and Thermal Conductivity	Fibers have low electrical and thermal conductivity. May develop a static charge, particularly under conditions of low humidity.

2.5 Enzymes

Enzymes, like proteins, are produced from reacting various amino acids and polymerizing into long polypeptide chains $[\text{-NH-R-CONH-R-CO-}]_n$. These amino acids react to each other and eliminate water. As the polymeric chain length increases, ionic and other interactions eventually cause the complex molecule. Enzymes can be found in all living cells, where they perform a vital function by controlling the metabolic processes, whereby nutrients are converted into energy and new cells. Moreover, enzymes take part in the breakdown of materials into simpler compounds.

Enzymes are bio-catalyst, and by their mere presence, and without being consumed in the process, enzymes can speed up the chemical process that would otherwise run very slowly. Catalyst is a substance that enhances the rate of chemical reaction but is not permanently alerted by reactions. Catalyst performs this feat because it decreases the activation energy required for a chemical reaction to happen or provides an alternative reaction pathway that requires less energy [21]. Figure 2.12 shows a transition state occurring at the apex of both reaction pathways. During any chemical reaction, reactants with sufficient energy attain transition state configuration. For biochemical systems, this occurs when the substrate binds to the enzyme.

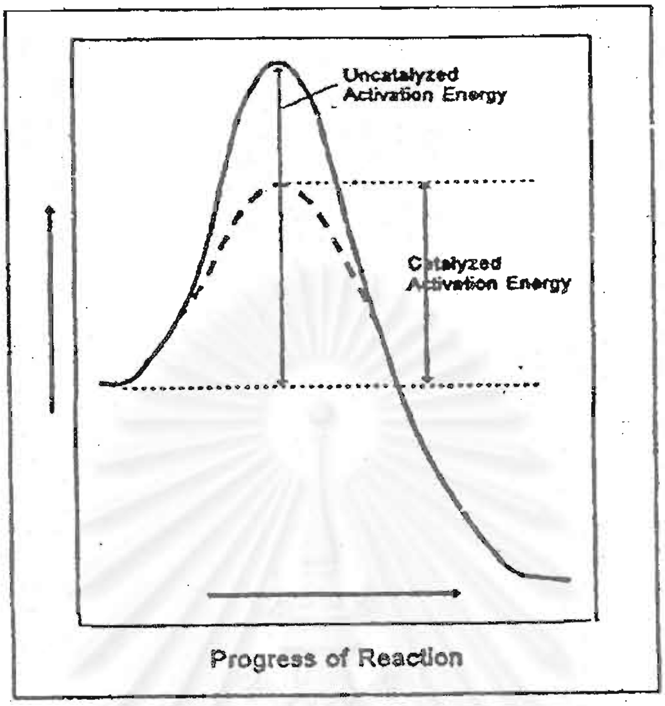


Figure 2.12 Activation energy for a given reaction in the presence and in the absence of catalyst [21].

After the reaction is complete, enzyme is released and is ready to start another reaction. In principle, this could go on forever. But in practically, most catalysts have a limited stability. Over a period of time, they lose their activity and are not usable again. Generally, most enzymes are used only once and discarded after they have completed their job.

Enzyme's functions are very specific in comparison to inorganic catalysts such as acids, bases, metals and metal oxides. Enzyme can break down particular compounds. In some cases, their action is limited to specific bonds in the compounds with which they react. The molecule that an enzyme acts on is known as its substrate, which is converted into a product.

Enzymes work at atmospheric pressure and in mild conditions with respect to temperature and pH. Most enzymes function optimally at a temperature of 30°C-70°C and at pH near the neutral point (pH 7). Due to their efficiency, specific action, mild work conditions and high biodegradability, enzymes are very well suited for a wide range of industrial applications.

Nowadays, special enzymes have been developed to work at higher temperatures for specific applications.

2.5.1 Mechanism for Enzyme Action

An enzyme has a quite specific three-dimensional shape. This shape and other factors, such as the location of the active site on the enzyme, control the specificity of the molecule. An enzyme is absorbed onto a given substrate surface in "lock-and-key" fashion [22] (Figure 2.13). At the surface of the substrate, the enzyme serves to accelerate the reaction of the substrate and the environment before converting into products. Since enzymes are catalysts, they themselves are not changed by the reaction that the substrate undergoes. After the reaction has taken place, the enzyme is released to be reabsorbed onto another substrate surface. The process continues until the enzyme is poisoned by a chemical bogie or inactivated by extremes of temperature, pH, or by other negative conditions in the processing environment.

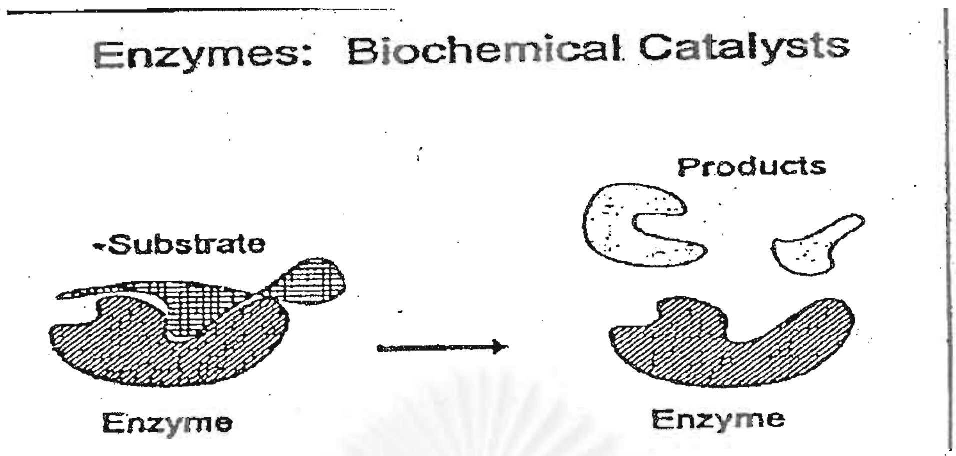


Figure 2.13 "Lock-and-Key" mechanism for enzyme action. Enzyme is absorbed at a substrate, followed by releasing the reaction products and enzyme[22].

2.5.2 Classification of Enzymes

An international classification has been established to define six major classes of enzyme function according to the type of chemical reaction it catalyzes [23-25].

The following are the six major enzyme categories:

EC.1 ***Oxidoreductases*** catalyze oxidation-reduction reactions. Subclasses of this group include dehydrogenase, oxidases, oxygenases, reductases, peroxidases and hydroxylases.

EC.2 ***Transferases*** catalyze transfers of groups such as amino, carboxyl, carbonyl, methyl, acyl (RC=O), glycosyl, or phosphoryl. Common trivial names for the transferases often include the prefix "trans". Examples include transcarboxylases, transmethyl-lases, and transaminases.

EC.3 ***Hydrolases*** catalyze cleavage of bonds between a carbon atom and some atoms by addition of water. The hydrolases include the esterases, phophatases, and peptidases.

EC.4 ***Lyases*** catalyze breakage of carbon-carbon, carbon-surfur, and certain carbon-nitrogen bonds. Decarboxylases, dehydratases, deaminases, and systhases are examples of lyases.

EC.5 ***Isomerases*** catalyze racemization of optical or geometric isomers and certain intramolecular oxidation-reduction reactions. Epimerases catalyze the inversion of asymmetric carbon atom. Mutases catalyze the intramolecular transfer of functional groups.

EC.6 ***Ligases*** catalyze bond formation between two substrate molecules. The energy for these reactions is frequently derived from the hydrolysis of adenosine triphosphate. The names of many ligases include the term synthetase. Several other ligases are called carboxylases.

Each enzyme is individuated by four numbers: the first indicates the reaction catalyzed (class), the second is the function involved, the third gives more details on the reaction catalyzed indicating or the group acceptor or the substrate, and the fourth is the serial number of the enzyme in its subclass.

2.5.3 Lipases

Lipases are produced by numerous bacteria and fungi. The natural substrates of lipases are triglycerides of long-chain fatty acids. Lipases catalyze the hydrolysis of fats at the interface between the insoluble substrate and the aqueous phase in which the enzymes are soluble. Lipases attack the ester bonds in these fats, regenerates water soluble glycerol and water insoluble fatty acids, and converts to water soluble salts by the addition of alkali.

Lipases cleave triglycerides into free fatty acids, produce intermediate 1,2- or 2,3-diglycerides, hydrolyze into 2-monoglycerides in step II and hydrolyze to free glycerol and fatty acids in step III. The hydrolysis reaction is shown in Figure 2.14.

Lipid hydrolysis depends on different parameters such as pH, temperature, water content, and the phase boundary area. The optimum pH of most lipases lie between 7.5 to 9.0 [26].

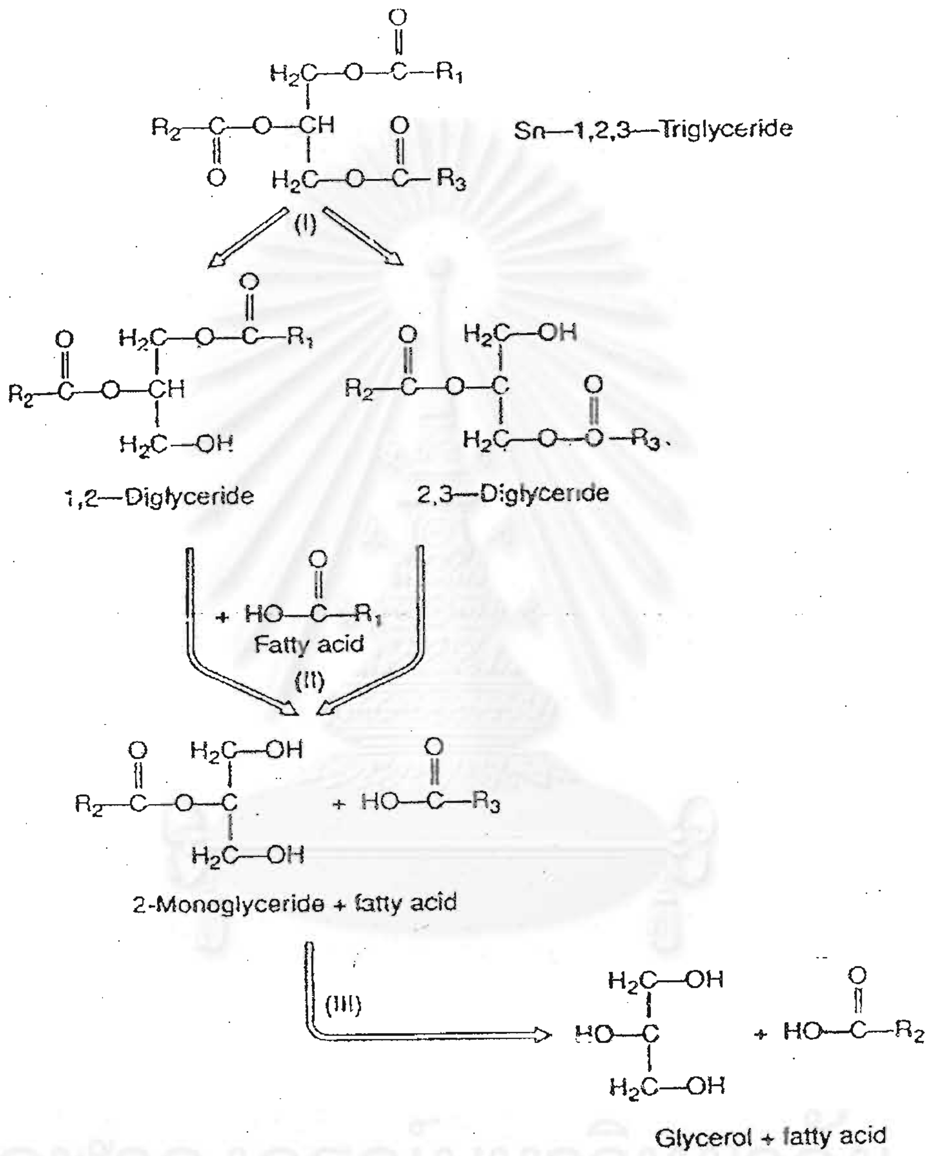


Figure 2.14 Reaction steps of lipase in triglyceride hydrolysis [26].

2.5.4 Proteases

Proteases or proteolytic enzymes catalyze the hydrolysis of proteins and peptides. There are two kinds of proteases, the proteinases (endopeptidases) and the peptidases (exopeptidases). Proteinases act on the interior peptide bonds of proteins and peptides. They include pepsin, trypsin, and chymotrypsin from animals and papaine from papaya. Peptidases (exopeptidases) act on peptide bonds adjacent to the free amino acid or carboxyl group [9].

2.5.5 Cellulases

Cellulases are multi-component enzyme systems commonly produced by soil-dwelling fungi and bacteria. These fungi and bacteria produce cellulases to reduce cellulose to glucose to use as food. There are three types of cellulase components act in degrading cellulose to glucose; endo-cellulases, exo-cellulases, and β-glucosidases. The current proposed mechanism of cellulase action is shown in Figure 2.15.

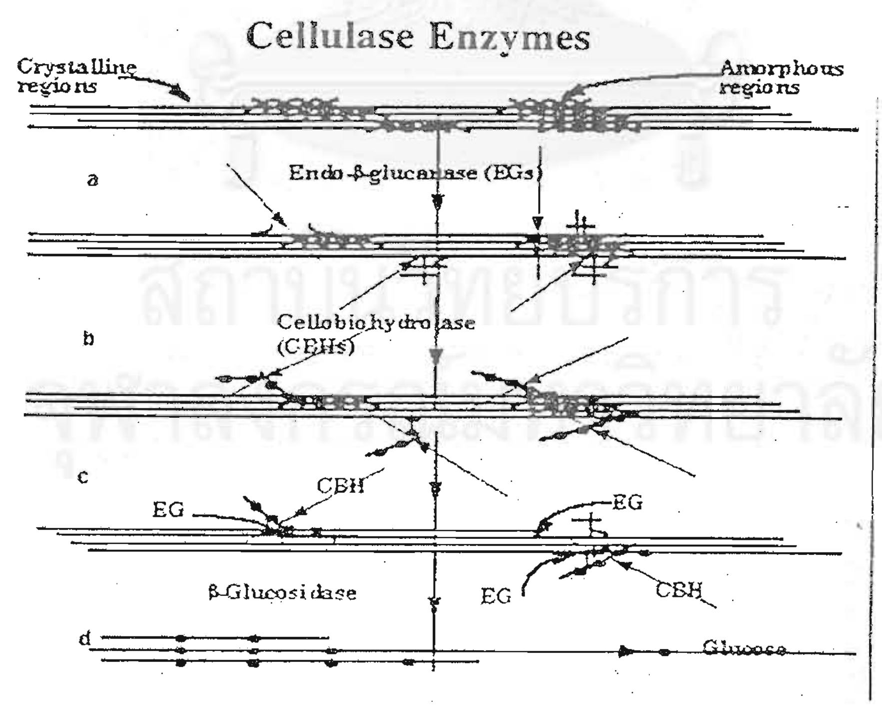


Figure 2.15 Schematic representation of synergistic action of cellulase on cellulose [27].

Endoglucanases or endo-celulases hydrolyze cellulose polymers randomly along the chains to form chain ends, preferentially attacking noncrystalline regions. Cellobiohydrolases or exo-cellulases attack the polymer chain ends and produce primarily cellobioses. Coupled with the binding domains associated with the enzyme, exo-cellulases may assist in degradation of cellulose by disrupting the crystalline cellulose structure which makes the region more susceptible to subsequent hydrolysis by endo-cellulases. β-glucosidases hydrolyze small chain oligomers, such as cellobioses, into glucoses.

Most commercial cellulases are derived from the fungal *Trichoderma* and *Penicillium* species. They may be produced both in powder forms and as concentrated liquids 25% for active in brine for stability. The optimum activity of cellulase from fungi is at pH 4.5-5 and temperature 45 ^{o}C.

2.6 Preparation Processes

Preparation processes of cotton and other fibers are necessary for removing impurities from the fibers and for improving their aesthetic appearance and processability prior to dyeing, printing and/or mechanical and chemical finishing.

The need for good preparation has long been appreciated, but the developments taken place in dyeing processes, particularly continuous pad dyeing, have accentuated the importance of the correct preparation of cotton. Over long continuous runs, the fabric must be evenly treated to have excellent absorbency, low residual size and wax content, whiteness appropriate to the color to be dyed and minimal fiber degradation. This technical standard must be met against economic constraints relating to the cost of chemical, labor, power and water.

Among these preparation processes, scouring is one of the most important process for all kinds of fibers. Without this step, the fibers will not absorb water, dyes, and chemicals adequately. In general, scouring is conducted at a high temperature in an aqueous solution containing detergent, chelating agent, and alkali. Some solvents can be used for scouring as well, and the process was called “Solvent Scouring”.

2.6.1 Conventional Scouring

Scouring is almost invariably the first wet process (except desizing for only woven fabric) applied to textile materials. The main purpose of scouring any substrate is to remove oils both from natural and spinning, weaving and knitting operations, fats, soluble impurities and any particulate or solid dirt adhering to the fiber. The process consists essentially of treatment with a detergent, with or without the addition of alkali depending on types of fibers.

An effective removal of impurities in cotton, particularly waxes, is achieved by boiling cotton in 3-6% sodium hydroxide solution or less frequently in dilute solutions of calcium hydroxide (lime) or sodium carbonate(soda ash). The proper choice of textile auxiliaries in the alkaline bath is essential for good scouring. These include sequestering or chelating agents such as ethylenediaminetetraacetic acid (EDTA) to remove inorganic substances or heavy metal in hard water, and surfactants such as the anionic sodium lauryl sulfate that serves as a detergent, dispersing agent, and emulsifying agent to remove unsaponifiable waxes.

Synthetic fibers are scoured with milder chemicals such as anionic or nonionic detergents with trace amounts of sodium carbonate or ammonia. Scouring temperature for these fibers is generally 50-100 ^{o}C.

Cotton/synthetic fiber blends (such as cotton/polyester) require alkaline concentrations and conditions intermediate between those for cotton and for synthetics [28].

2.6.2 Enzymatic Scouring

Csiszer, Szakacs, and Rusznak [29] studied the removal of seed coat fragment in spinning blow room waste, by consecutive cellulase treatment and traditional pad-steam scouring. They found that the compact and resistant structure of lignocellulose in seed-coat was loosened by the complex action of enzymes. When it was attacked by aqueous sodium hydroxide, the tiny fibers that attach the seed coat fragment to the fabric were hydrolyzed by the enzyme, facilitating the removal of those impurities from the fabric surface. Approximately 80% of seed coat fragments were dissolved.

Li and Hardin [11, 30] proposed the action of pectinase and cellulase enzymes on structure changes in surfaces of cotton observed from staining tests and microscopy observations. The pectinase enzymes could destroy the cuticle structure by digesting the inner layer pectins in the cuticle and cellulases could destroy the cuticle structure by digesting the primary wall of cellulose immediately under the cuticle. In addition to the research they also studied the factors; surfactants, agitation, and selection of enzymes [31] which effected on an enzymatic scouring. They concluded that nonionic surfactants were compatible with enzymes because they did not interfere with the three-dimensional structure of enzymes. Mechanical agitation could increase apparent enzyme activity and efficiency in scouring, but should be cautious when using machanical agitation with high shear forces because enzymes could be denatured.

Buschle-Diller and El Mogahzy [32] presented the effect of cotton yarn scourings with pectinase and cellulase enzymes, with 1,1,1-trichloroethylene and with caustic soda. They proposed that all three scouring methods increased yarn absorbency. Caustic scouring gave yarn the highest degree of whiteness but left them with the most sensitive to oxidative damage during subsequent bleaching . Solvent extraction method increased yarn tenacity.

Pectinase/cellulase scouring yielded very soft yarn with moderate yarn tensile strength and fiber deterioration.

Buschle-Diller et. al. [33,34] studied bioscouring of cotton using pectinase alone and combination with lipase, cellulase and xylanase. They concluded that the water absorbency of textile material was improved if used pectinase combination with lipase and cellulase. And their latest research [35] concerned a combination of all three preparatory processes including desizing, scouring and bleaching. They used glucose wastes from the desizing bath to react with the glucose oxidase enzyme in order to produce hydrogen peroxide for the bleaching step. They found that the whiteness of the enzymatically bleached goods was closed to those of fabrics bleached conventionally with hydrogen peroxide.

Hsieh and Hartzell [10] investigated four kinds of enzymes, i.e., pectinase, cellulase, protease, and lipase, for their effectiveness in improving the water absorbency and retention properties of cotton fabrics. Cellulase was the only enzyme to produce detectable improvements in water wettability of greige cotton. It was able to gain access to cellulose removing the hydrophobic noncellulosic component from the fabric surface. But when they combined cellulase with pectinase, both water contact angle and water retention values fall within the range of commercially scoured fabrics. They also used pectinase combined with a 100 ^{o}C water pretreatment that resulted in the same wetting properties with cellulase treatment.

In addition to this research Hsieh et. al. [36] studied the effective of the enzymatic hydrolysis to improve the wetting and absorbency of polyester fabrics. By using lipase enzymes, the water wetting and retention of polyester fabrics improved more than the alkaline hydrolysis.

Yachmenev, Blanchard and Lambert [37] used the ultrasound energy in the reaction chamber during cellulase treatment of cotton fabric. They found that using ultrasonic energy could provide significant saving of processing time and cellulase enzyme concentration needed for the process, and obtain better uniformity of the treatment.

In this study, commercial enzymes lipase, protease, and cellulase were used to investigate the effects of the enzymatic scouring on cotton, CVC, T/C, polyester and nylon fabrics compared with the conventional scouring process. The primary focus was to determine the appropriate enzymatic scouring formulations for the fabrics.

CHAPTER 3

Experimental

3.1 Materials

3.1.1 Fabric Samples:

:Greige cotton fabric, single jersey knit, yarn count 50/1, weighs 1.1614 g/100 cm^2

:Greige CVC (Cotton 55%:Polyester 45%) fabric, single jersey knit, yarn count 40/1, weighs 1.9404 g/100 cm^2

:Greige T/C (Cotton 35%:Polyester 65%) fabric, interlock knit, yarn count 45/1, weighs 1.7825 g/100 cm^2

:Greige polyester fabric, twill weave, weighs 2.0808 g/100 cm^2

:Greige nylon fabric, plain weave, weighs 0.5967 g/100 cm^2

Greige samples were tested for the strength, the extractable materials, the water absorbency, and the weight according to the test procedures outlined in section 3.4.

3.1.2 Enzymes:

Table 3.1 Enzymes used in this experiment.

Enzyme	EC. Number	Source	Activity	Company
Lipase	EC.3.1.1.3	Pancreas	15 units/g	Tokyo Chemical Industry, Japan
Protease	EC.3.4.23.6	Aspergillus oryzae	14,000 units/g	Tokyo Chemical Industry, Japan
Cellulase	EC.3.2.1.4	Aspergillus niger	25,000 units/g	Tokyo Chemical Industry, Japan

3.1.3 Reagent Grade Chemicals:

Table 3.2 Chemicals used in this experiment.

Chemical	Company
Sodium hydroxide pellets 98%	EKA Chemicals
Glacial acetic acid	Merck, Germany
Potassium hydrogen phosphate powder	APS Ajax Finechem, Australia
Disodium hydrogen phosphate powder	APS Ajax Finechem, Australia
Womine TE (Wetting agent)	Tokai Seiyu, Japan
Sodium acetate powder	APS Ajax Finechem, Australia
Sodium chloride powder	APS Ajax Finechem, Australia
Sodium hydrosulphite powder	APS Ajax Finechem, Australia
Sodium carbonate powder	SEELZE-HANNOVER, Germany
1,1,2,2 - Tetrachloroethylene	MAY & BAKER, England

3.1.4 Dyes:

Table 3.3 Dyestuffs used in this experiment.

Dye	Company
Methylene blue	Nacali Tesque, Inc., Japan
Benzopurpurine 4B	Tokyo Chemical Industry, Japan
Remazol® Red RGB	Dystar, Germany
Dianix® Red CC	Dystar, Germany
C.I. Disperse Red 60	Ciba
Kayanol Milling Red BW	Metro company Ltd.

3.2 Equipment

1. pH meter, Denver Instrument, Model 215
2. Labolatory dyeing machine & steel pots, Ahiba Polymat®
3. Labolatory dyeing machine, UGOLINI, Model B.M.R.
4. Macbeth reflectance spectrophotometer, COLOR-EYE® 7000
5. UV-Visible spectrophotometer, JENWAY, Model 6405
6. Shaker bath, Gallenkamp, Model No. 900032
7. Soxhlet extraction assembly
8. Sample cutter, diameter 11.3 cm., Jen-Haur Co., Ltd.
9. Stop watch, Alba, Cal. SW01
10. Bursting strength tester, Yasuda, Mullen type
11. Tensile strength tester, LR 100K, LLOYD Instrument
12. Balance, Mettler Toledo, Model AB 204
13. Infrared Moisture Balance, Model AD-4715
14. Stiffness tester, Shirley, Model No.248
15. Scanning Electron Microscope, JEOL, Model JSM-5410LV

3.3 Fabric Scouring Procedures

The following experiment was conducted in order to study the effectiveness of the enzymatic scouring on various fabrics using lipase, protease, and cellulase enzymes and to compare the scouring results with the results from the conventional scouring process. Fabrics were first prewashed, then scoured, and finally tested for properties using the procedures shown as follows.

3.3.1 Prewashing

Before scouring, all greige fabrics were prewashed in a boiling water for 30 minutes in order to remove the water soluble materials depositing on the fabric surface. They were then rinsed in water and air dried. The prewashed fabrics were tested for the water absorbency according to the test procedure outline in section 3.4.1 in order to determine whether a scouring was needed. The fabric with an inadequate absorbency was further scoured using the following procedures.

3.3.2 Scouring

3.3.2.1 Conventional Scouring

Prewashed cotton, CVC and T/C fabrics were scoured in solutions containing a wetting agent "Womine TE" and a scouring agent sodium hydroxide in the Ahiba Polymat® Laboratory dyeing machine (see Figure 3.1) at a liquor ratio of 20:1. The temperature was raised to 80°C over 12 minutes (4°C/min) and hold at this temperature for 60 minutes. The fabrics were then removed from the machine, washed in water at 80°C for 20 minutes, rinsed until a neutral pH, and air dried.

Prewashed polyester fabric was scoured in the solution containing Womine TE and sodium carbonate at a liquor ratio of 20:1, at 80°C for 30 minutes in the dyeing machine mentioned earlier. Then it was washed in water at 80°C for 20 minutes, rinsed and air dried.

Prewashed nylon fabric was scoured in a solution containing only Womine TE at a liquor ratio of 20:1, at $80^{\circ}C$ for 30 minutes in the dyeing machine. Then it was rinsed and air dried.

The scouring formulations (see Table 3.4) and conditions used in this experiment were based on the industrial guideline and on the theoretical data. The conventional scouring procedure is shown in Diagram 3.1.

Figure 3.1 Laboratory dyeing machine, Ahiba Polymat®.

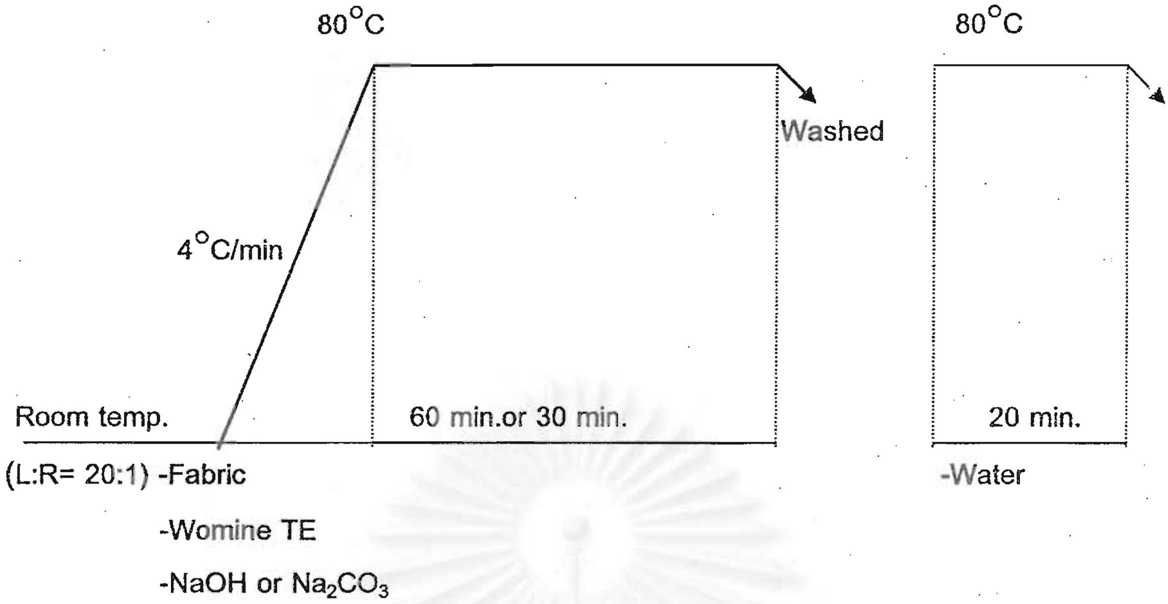


Diagram 3.1 The conventional scouring procedure.

Table 3.4 The conventional scouring formulations.

Fabric	Sodium hydroxide (%o.w.f.)*	Sodium carbonate (g/l)	Womine TE (g/l)
Cotton fabric	3.0	–	3.0
CVC fabric	0.5	–	3.0
T/C fabric	0.5	–	3.0
Polyester fabric	–	3.0	3.0
Nylon fabric	–	–	3.0

* of weight of fabric

The conventional scoured fabrics were tested for the water absorbency, the fabric weight loss, the fabric whiteness, the dye absorption, and the appearance of the fiber surface according to the test procedures shown in section 3.4.

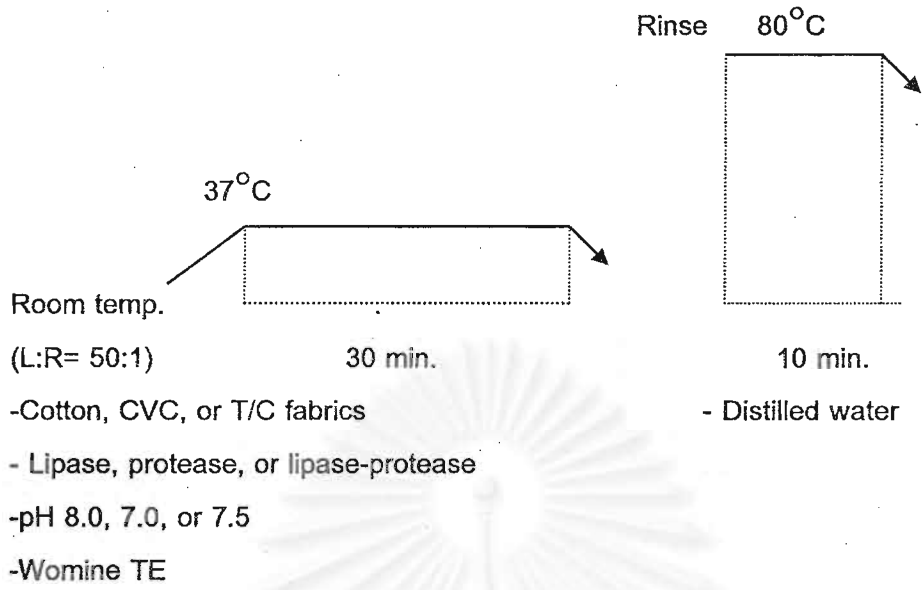
3.3.2.2 Enzymatic Scouring

Three kinds of enzymes, lipase, protease, and cellulase were used as scouring agents for the enzymatic scouring. Prewashed fabrics were scoured in solutions containing Womine TE and enzymes at a liquor ratio of 50:1 at pH, temperatures and times indicated in Table 3.5.

Cotton, CVC, and T/C fabrics were one-step scoured with lipase/nonionic wetting agent; with protease/nonionic wetting agent; with lipase/protease/nonionic wetting agent; and with cellulase/nonionic wetting agent. They were also scoured using two-steps scouring with lipase/nonionic wetting agent and then with cellulase/nonionic wetting agent; with protease/nonionic wetting agent and then with cellulase/nonionic wetting agent; and with lipase-protease/ nonionic wetting agent and then with cellulase/nonionic wetting agent. Polyester fabric was scoured with enzyme lipase and nonionic wetting agent. Nylon fabric was not scoured with any enzyme because it could be completely scoured using only a nonionic wetting agent in the conventional scouring process.

The enzymatic scouring conditions and procedures for each fabric are shown in Diagrams 3.2-3.3 and Table 3.5. After each step of scouring, the fabric was removed from the enzyme solution and placed in a boiling water for 10 minutes in order to terminate the enzyme function.

The amount of enzymes and nonionic wetting agent "Womine TE" used was varied for each fabric to achieve an adequate absorbency.



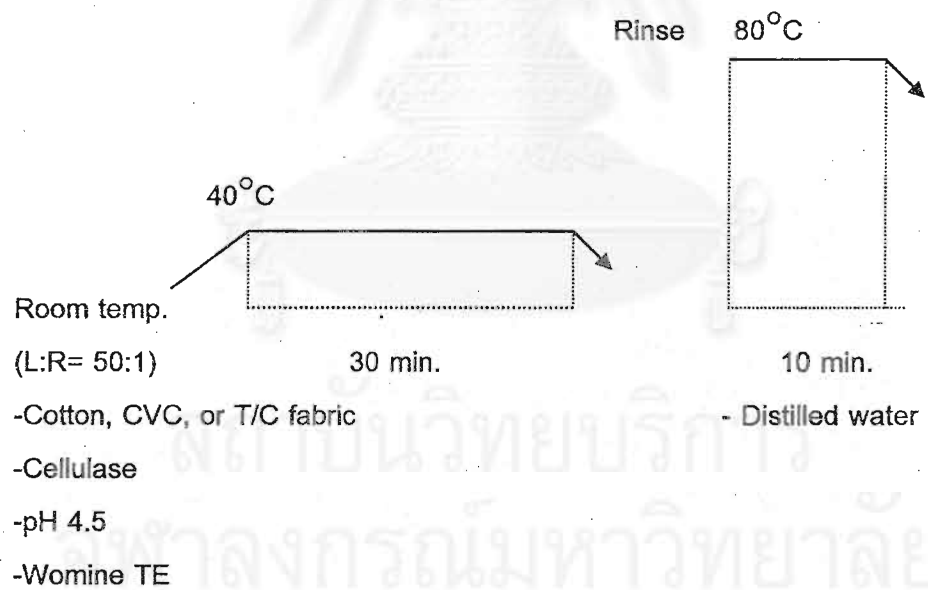


Diagram 3.2 The enzymatic scouring procedures for cotton, CVC, and T/C fabrics .

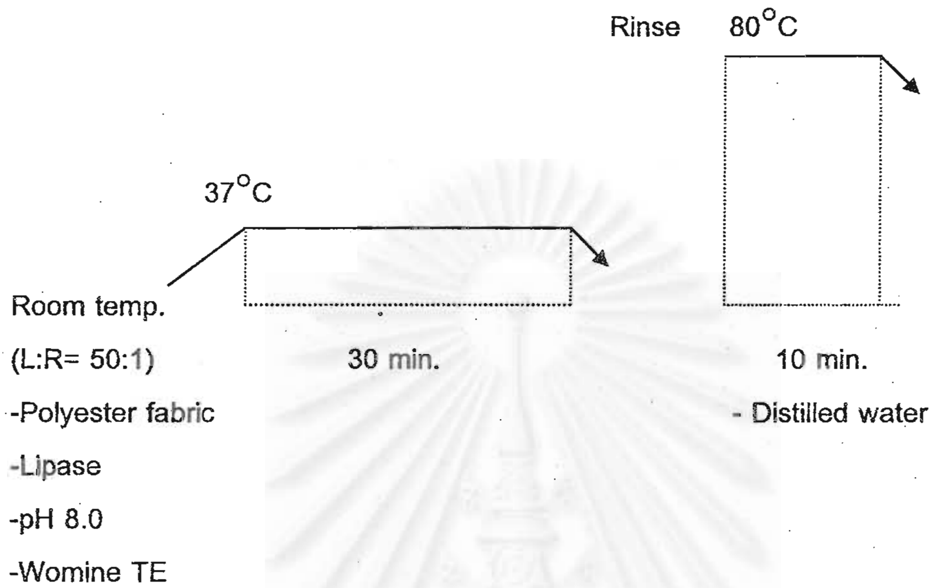


Diagram 3.3 The enzymatic scouring procedure for polyester fabrics.

Table 3.5 The enzymatic scouring formulations using various enzymes.

Fabric	Trial	Enzyme			Womine TE (g/l)	Condition		
		Step	Type	g/l		Temp. (°C)	pH	Time (min.)
Cotton fabric	1	1	Lipase	0.50	1	37	8.0	30
	2	1	Lipase	0.50	1	37	8.0	30
		2	Cellulase	0.50	1	40	4.5	30
	3	1	Protease	0.50	1	37	7.0	30
	4	1	Protease	0.50	1	37	7.0	30
		2	Cellulase	0.50	1	40	4.5	30
	5	1	Cellulase	0.50	1	40	4.5	30
	6	1	Lipase	0.25	1	37	7.5	30
		1	Protease	0.25				
	7	1	Lipase	0.25	1	37	7.5	30
		1	Protease	0.25				
		2	Cellulase	0.50	1	40	4.5	30
CVC fabric	8	1	Lipase	0.50	1	37	8.0	30
	9	1	Lipase	1.00	1	37	8.0	30
	10	1	Lipase	2.00	1	37	8.0	30
	11	1	Lipase	3.00	1	37	8.0	30
	12	1	Lipase	4.00	1	37	8.0	30
	13	1	Lipase	5.00	1	37	8.0	30
	14	1	Lipase	10.00	1	37	8.0	30
	15	1	Lipase	0.50	1	37	8.0	30
		2	Cellulase	0.50	1	40	4.5	30
	16	1	Lipase	1.00	1	37	8.0	30
		2	Cellulase	0.50	1	40	4.5	30
	17	1	Lipase	2.00	1	37	8.0	30
		2	Cellulase	0.50	1	40	4.5	30

Table 3.5 (continued)

Fabric	Trial	Enzyme			Womine TE (g/l)	Condition		
		Step	Type	g/l		Temp. (°C)	pH	Time (min.)
CVC fabric	18	1	Protease	0.50	1	37	7.0	30
	19	1	Protease	1.00	1	37	7.0	30
	20	1	Protease	2.00	1	37	7.0	30
	21	1	Protease	3.00	1	37	7.0	30
	22	1	Protease	4.00	1	37	7.0	30
	23	1	Protease	5.00	1	37	7.0	30
	24	1	Protease	10.00	1	37	7.0	30
	25	1	Protease	0.50	1	37	7.0	30
		2	Cellulase	0.50	1	40	4.5	30
	26	1	Protease	1.00	1	37	7.0	30
		2	Cellulase	0.50	1	40	4.5	30
	27	1	Protease	2.00	1	37	7.0	30
		2	Cellulase	0.50	1	40	4.5	30
	28	1	Lipase	0.25	1	37	7.5	30
		1	Protease	0.25				
	29	1	Lipase	0.50	1	37	7.5	30
		1	Protease	0.50				
	30	1	Lipase	1.00	1	37	7.5	30
		1	Protease	1.00				
	31	1	Lipase	1.50	1	37	7.5	30
		1	Protease	1.50				
	32	1	Lipase	2.00	1	37	7.5	30
		1	Protease	2.00				
	33	1	Lipase	2.50	1	37	7.5	30
		1	Protease	2.50				
	34	1	Lipase	5.00	1	37	7.5	30
		1	Protease	5.00				

Table 3.5 (continued)

Fabric	Trial	Enzyme			Womine TE (g/l)	Condition		
		Step	Type	g/l		Temp. (°C)	pH	Time (min.)
CVC fabric	35	1	Lipase	0.25	1	37	7.5	30
		1	Protease	0.25				
		2	Cellulase	0.50	1	40	4.5	30
	36	1	Lipase	0.50	1	37	7.5	30
		1	Protease	0.50				
		2	Cellulase	0.50	1	40	4.5	30
	37	1	Lipase	1.00	1	37	7.5	30
		1	Protease	1.00				
		2	Cellulase	0.50	1	40	4.5	30
	38	1	Cellulase	0.50	1	40	4.5	30
	39	1	Cellulase	1.00	1	40	4.5	30
T/C fabric	40	1	Lipase	0.50	1	37	8.0	30
	41	1	Lipase	1.00	1	37	8.0	30
	42	1	Lipase	5.00	1	37	8.0	30
	43	1	Lipase	10.00	1	37	8.0	30
	44	1	Lipase	0.50	1	37	8.0	30
		2	Cellulase	0.50	1	40	4.5	30
	45	1	Lipase	1.00	1	37	8.0	30
		2	Cellulase	0.50	1	40	4.5	30
	46	1	Lipase	5.00	1	37	8.0	30
		2	Cellulase	0.50	1	40	4.5	30
	47	1	Lipase	10.00	1	37	8.0	30
		2	Cellulase	0.50	1	40	4.5	30
	48	1	Lipase	0.50	1	37	8.0	30
		2	Cellulase	1.00	1	40	4.5	30

Table 3.5 (continued)

Fabric	Trial	Enzyme			Womine TE (g/l)	Condition		
		Step	Type	g/l		Temp. (°C)	pH	Time (min.)
T/C fabric	49	1	Lipase	0.50	1	37	8.0	30
		2	Cellulase	2.00	1	40	4.5	30
	50	1	Lipase	0.50	1	37	8.0	30
		2	Cellulase	5.00	1	40	4.5	30
	51	1	Lipase	0.50	1	37	8.0	30
		2	Cellulase	10.00	1	40	4.5	30
	52	1	Lipase	10.00	1	37	8.0	30
		2	Cellulase	10.00	1	40	4.5	30
	53	1	Protease	0.50	1	37	7.0	30
	54	1	Protease	1.00	1	37	7.0	30
	55	1	Protease	5.00	1	37	7.0	30
	56	1	Protease	10.00	1	37	7.0	30
	57	1	Protease	0.50	1	37	7.0	30
		2	Cellulase	0.50	1	40	4.5	30
	58	1	Protease	1.00	1	37	7.0	30
		2	Cellulase	0.50	1	40	4.5	30
	59	1	Protease	5.00	1	37	7.0	30
		2	Cellulase	0.50	1	40	4.5	30
	60	1	Protease	10.00	1	37	7.0	30
		2	Cellulase	0.50	1	40	4.5	30
	61	1	Protease	0.50	1	37	7.0	30
		2	Cellulase	1.00	1	40	4.5	30
	62	1	Protease	0.50	1	37	7.0	30
		2	Cellulase	2.00	1	40	4.5	30
	63	1	Protease	0.50	1	37	7.0	30
		2	Cellulase	5.00	1	40	4.5	30

Table 3.5 (continued)

Fabric	Trial	Enzyme			Womine TE (g/l)	Condition		
		Step	Type	g/l		Temp. (°C)	pH	Time (min.)
T/C fabric	64	1	Protease	0.50	1	37	7.0	30
		2	Cellulase	10.00	1	40	4.5	30
	65	1	Protease	10.00	1	37	7.0	30
		2	Cellulase	10.00	1	40	4.5	30
	66	1	Lipase	0.25	1	37	7.5	30
		1	Protease	0.25				
	67	1	Lipase	0.50	1	37	7.5	30
		1	Protease	0.50				
	68	1	Lipase	2.50	1	37	7.5	30
		1	Protease	2.50				
	69	1	Lipase	5.00	1	37	7.5	30
		1	Protease	5.00				
	70	1	Lipase	0.25	1	37	7.5	30
		1	Protease	0.25				
		2	Cellulase	0.50	1	40	4.5	30
	71	1	Lipase	2.50	1	37	7.5	30
		1	Protease	2.50				
		2	Cellulase	0.50	1	40	4.5	30
	72	1	Lipase	5.00	1	37	7.5	30
		1	Protease	5.00				
		2	Cellulase	0.50	1	40	4.5	30
	73	1	Lipase	0.25	1	37	7.5	30
		1	Protease	0.25				
		2	Cellulase	1.00	1	40	4.5	30

Table 3.5 (continued)

Fabric	Trial	Enzyme			Womine TE (g/l)	Condition		
		Step	Type	g/l		Temp. (°C)	pH	Time (min.)
T/C fabric	74	1	Lipase	0.25	1	37	7.5	30
		1	Protease	0.25				
		2	Cellulase	2.00	1	40	4.5	30
	75	1	Lipase	0.25	1	37	7.5	30
		1	Protease	0.25				
		2	Cellulase	5.00	1	40	4.5	30
	76	1	Lipase	0.25	1	37	7.5	30
		1	Protease	0.25				
		2	Cellulase	10.00	1	40	4.5	30
	77	1	Lipase	5.00	1	37	7.5	30
		1	Protease	5.00				
		2	Cellulase	10.00	1	40	4.5	30
T/C fabric, (prewashed with 1 g/l wetting agent)	78	1	Lipase	0.50	1	37	8.0	30
	79	1	Protease	0.50	1	37	7.0	30
	80	1	Lipase	0.25	1	37	7.5	30
		1	Protease	0.25				
	81	1	Cellulase	0.50	1	40	4.5	30
	82	1	Lipase	0.50	1	37	8.0	30
		2	Cellulase	0.50	1	40	4.5	30
	83	1	Protease	0.50	1	37	7.0	30
		2	Cellulase	0.50	1	40	4.5	30
	84	1	Lipase	0.25	1	37	7.5	30
		1	Protease	0.25				
		2	Cellulase	0.50	1	40	4.5	30

Table 3.5 (continued)

Fabric	Trial	Enzyme			Womine TE (g/l)	Condition		
		Step	Type	g/l		Temp. (°C)	pH	Time (min.)
polyester fabric	85	1	Lipase	0.50	1	37	8.0	30
	86	1	Lipase	1.00	1	37	8.0	30
	87	1	Lipase	5.00	1	37	8.0	30
	88	1	Lipase	10.00	1	37	8.0	30
polyester fabric (prewashed with 1 g/l wetting agent)	89	1	Lipase	0.50	1	37	8.0	30
	90	1	Lipase	1.00	1	37	8.0	30
	91	1	Lipase	1.00	3	37	8.0	30
	92	1	Lipase	1.00	5	37	8.0	30
	93	1	Lipase	3.00	1	37	8.0	30
	94	1	Lipase	5.00	1	37	8.0	30
	95	1	Lipase	3.00	3	37	8.0	30

3.4 Test Procedures

3.4.1 Water Absorbency of Fabrics

After a complete scouring, the fabric is required to absorb water immediately or within 3 seconds and to absorb evenly all over the fabric. In this work after each scouring attempt, the fabric was first test for its water absorbency in order to determine whether the scouring formulation was appropriate. The right formulation was the one being used to scour a fabric and obtain a clean fabric with an immediate water absorbency or 0 wetting time. The absorbency test was conducted using the AATCC Test Method 79-2000 "Absorbency of Bleached Textiles [38]". A drop of water is allowed to fall onto the surface of the test specimen. The time required for the specula reflection of the water drop to disappear is measured and recorded as a wetting time. The fabric that absorbs water within three seconds or less and absorbs evenly all over the fabric is generally considered to have an adequate absorbency.

3.4.2 Fabric Weight

Fabric weight was determined by cutting the greige sample using a standard circular cutter and weighing the cut sample. The fabric weight was reported as mass in grams per unit area. The test was conducted 3 times on each sample and the sample weights were averaged.

3.4.3 Fabric Weight Loss

Fabric weight loss was measured in order to determine for the amount of materials being removed from the fabric by means of scouring. The fabric was weighed at 105°C both before and after each scouring using an Infrared moisture balance. Each fabric was tested 3 times and the weights were averaged.

3.4.4 Extractable Materials in Greige Cotton and Cotton Blends

The extractable materials in greige cotton and cotton blends were determined using the AATCC Test Method 97-1999 " Extractable Content of Greige and/or Prepared Textiles [39] ". The test was conducted in order to measure the amount of materials depositing on the greige cotton and cotton blends fabric. The fabric was first extracted with water and then with a solvent.

The fabric was weighed at 105°C before a water extraction using an Infrared moisture balance. The fabric was then immersed in 200 ml of distilled water at 82 $\pm 3^{\circ}C$ for 2 hours. The fabric was rinsed twice with 25 ml. of distilled water in a Buchner funnel secured in a filtration flask, air dried, and weighed again.

The fabric was weighed before a solvent extraction. The fabric was then extracted with 1,1,2,2- tetrachloroethylene 12-16 times in a Soxhlet extractor, removed and evaporated the solvent.

% extractable materials in greige fabrics were calculated using the following equation:

$$E= [(B - A)/ I] * (100) \qquad \text{(equation 3.1)}$$

Where

E is the material extracted by water or organic solvent, %

B is the mass of the specimen before the particular extraction, g

A is the mass of the specimen after the particular extraction, g

I is the mass of the oven-dried specimen before the first extraction, g

The test was conducted three times and the % extractable materials were averaged.

3.4.5 Presence of Pectins on Cotton and Cotton Blends

Cellulosic samples were tested for the presence of pectins by measuring the absorption of methylene blue onto the samples. This method is based on the interaction between the cationic dye of methylene blue and the carboxylate anion of the pectins on samples. The higher the dyes absorb onto the substrate, the higher the pectins present on the substrate.

A calibration curve indicating the relationship of the concentrations of methylene blue solution and its light absorbance was constructed using the following procedure. Various concentrations of the methylene blue solutions (0.0005, 0.001, 0.002, 0.003, 0.004, 0.005, 0.006, and 0.007 g/l) were prepared. Each solution was analyzed for its light absorbance at wavelenght 662 nm. using a spectrophotometer (see Figure 3.3). Then the calibration curve between the concentration of the methylene blue solution and the light absorbance was established for further uses.

Unscoured and scoured cotton, CVC and TC substrates were immersed in solutions containing 0.5 g/l methylene blue in the laboratory dyeing machine at a liquor ratio of 30:1 at 70°C for 8 hours. The solution after dyeing was diluted 40 times with distilled water. Then it was measured for a maximum light absorbance at wavelength 662 nm., and the concentration of the solution after dyeing was determined from the calibration curve. Finally, the concentration of methylene blue solution on each substrate was calibrated. The test was carried three times and the data were averaged.

3.4.6 Fabric Whiteness

The whiteness of unscoured and scoured fabrics was measured based on CIE Ganz using the Macbeth reflectance spectrophotometer (see Figure 3.2). Each fabric was measured eight times and the data were averaged.

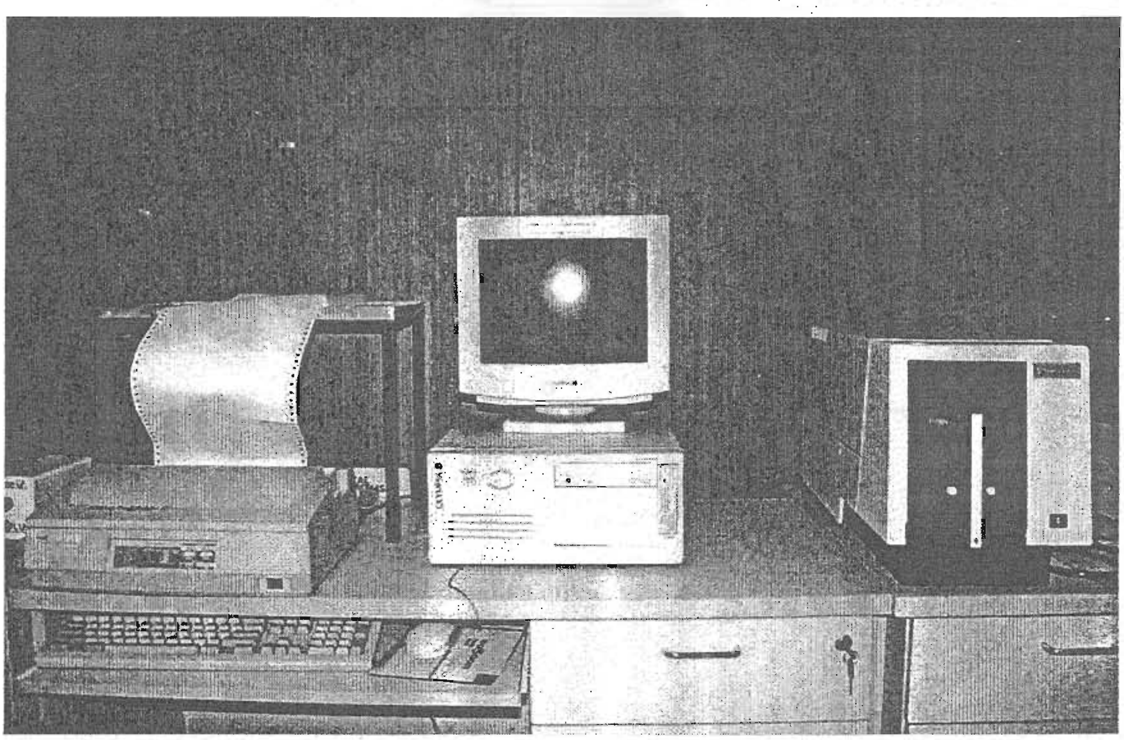


Figure 3.2 Macbeth reflectance spectrophotometer, Color-eye 7000.

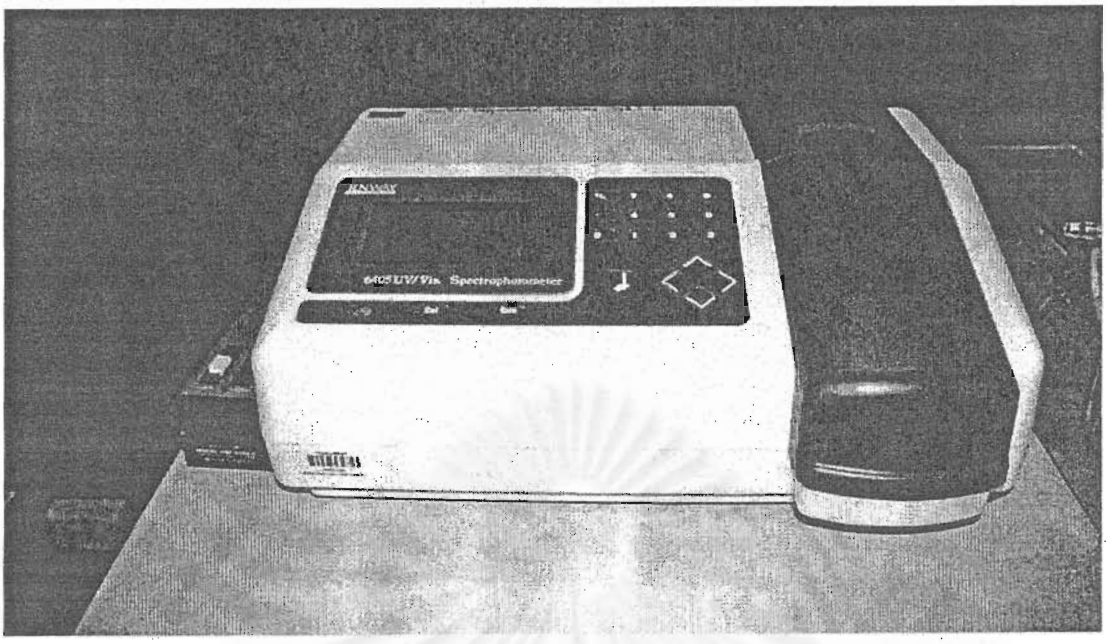


Figure 3.3 UV-visible spectrophotometer, JENWAY 6405.

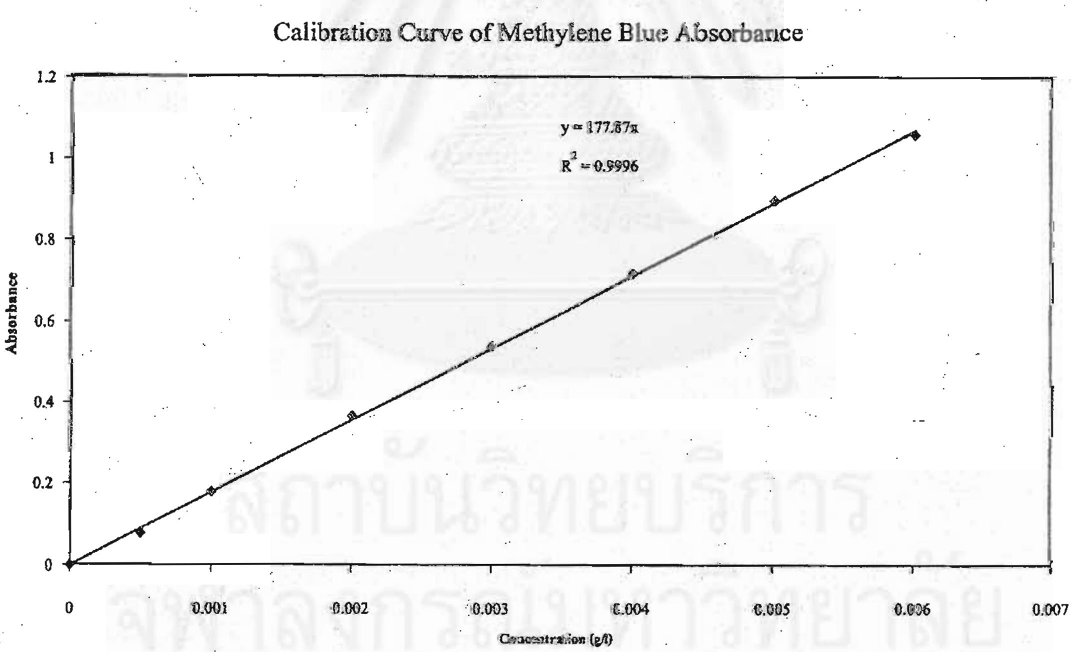


Figure 3.4 Concentration & absorbance calibration curve of standard methylene blue solution.

3.4.7 Dye Absorption Measurements

Scoured fabrics were dyed in order to observe the dyeability of the fabric after passing various scouring processes. Scoured cotton fabrics were dyed with direct dye, Benzopurpurine 4B 1% o.w.f. in the laboratory dyeing machine at a liquor ratio of 30:1. The dyeing process was commenced at room temperature. Then the temperature was raised to 95°C (5°C/minute) and maintained at this temperature for 45 minutes. The dye fabrics were then removed from dye solution, rinsed thoroughly in running tap water, squeezed and air dried. The dyeing process is illustrated in Diagram 3.4.

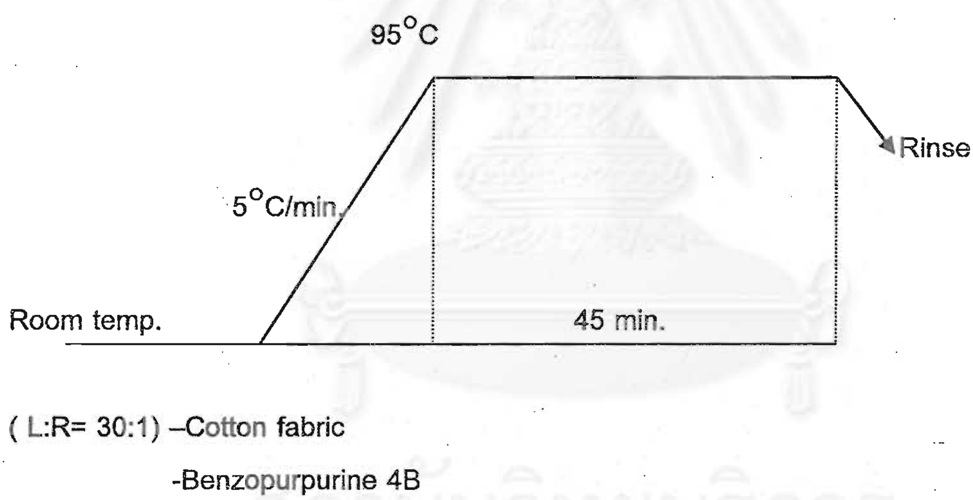


Diagram 3.4 The dyeing process for cotton fabric.

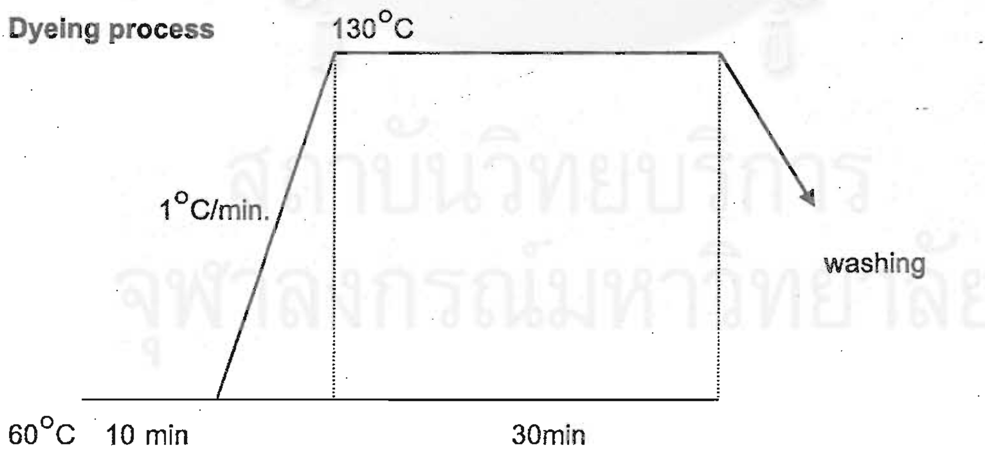
To dye the scoured CVC and T/C fabrics, a reactive dye (Remazol®) and a disperse dye (Dianix®) were used. The dye concentration is shown in Table 3.6.

Table 3.6 The dye concentrations used for dyeing CVC and T/C fabrics.

Substrate	**Dye**	
	Disperse (Dianix®) (%o.w.f.)	Reactive (Remazol®)(%o.w.f.)
CVC	1	3
TC	2	3

CVC and T/C fabrics were first dyed in the solutions containing disperse dye and 1 g/l wetting agent at a liquor ratio of 20:1 at pH 4-5. The temperature of dyeing process was commenced at 60°C for 10 minutes, then raised to 130°C (1°C/minute) and maintained at this temperature for 30 minutes (see Diagram 3.5). After dyeing, the fabrics were washed in water containing 2 g/l wetting agent at 60°C for 15 minutes. The unfixed disperse dye was removed from the fabric using the reduction clear process shown in Diagram 3.6. This process required 2 g/l sodium hydrosulfite, 2 g/l sodium hydroxide and 1 g/l wetting agent, and it was conducted at 80°C for 15 minutes. Then the fabrics were washed and air dried, ready for the reactive dyeing.

Dyeing process



(L:R= 20:1) -CVC or T/C fabric

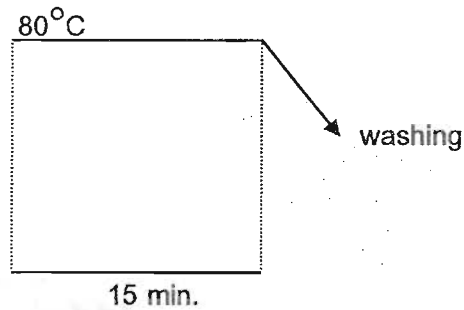
-pH 4-5

-Wetting agent 1 g/l

-Dianix

Diagram 3.5 The disperse dyeing process for CVC and T/C fabrics.

Reduction clear process



(L:R= 20:1)-$Na_2S_2O_4$ 2 g/l

-NaOH 2 g/l

-Wetting agent 1 g/l

Diagram 3.6 The reduction clear process for CVC and T/C fabrics after disperse dyeing.

CVC and T/C fabrics were further dyed at a liquor ratio of 10:1 in the solution containing reactive dye, sodium chloride, sodium carbonate and sodium hydroxide as shown in Diagram 3.7. The dyeing temperature was commenced at room temperature for 80 minutes, then raised 1°C/minute to 50°C and maintained at this temperature for 75 minutes.

First NaCl was added at room temperature together with the fabric, next 10 minutes the reactive dye (Remazol®) was added, after 30 minutes sodium carbonate and sodium hydroxide were added. Then the temperature was raised from room temperature to 50°C. After 10 minutes, sodium hydroxide was added and maintained at this temperature for another 65 minutes. After dyeing, the dyed fabrics were removed from the dye solution, rinsed thoroughly in cool water, hot water, and washed with 1g/l wetting agent at 100°C for 10 minutes. Finally rinse again with cool water and hot water, respectively.

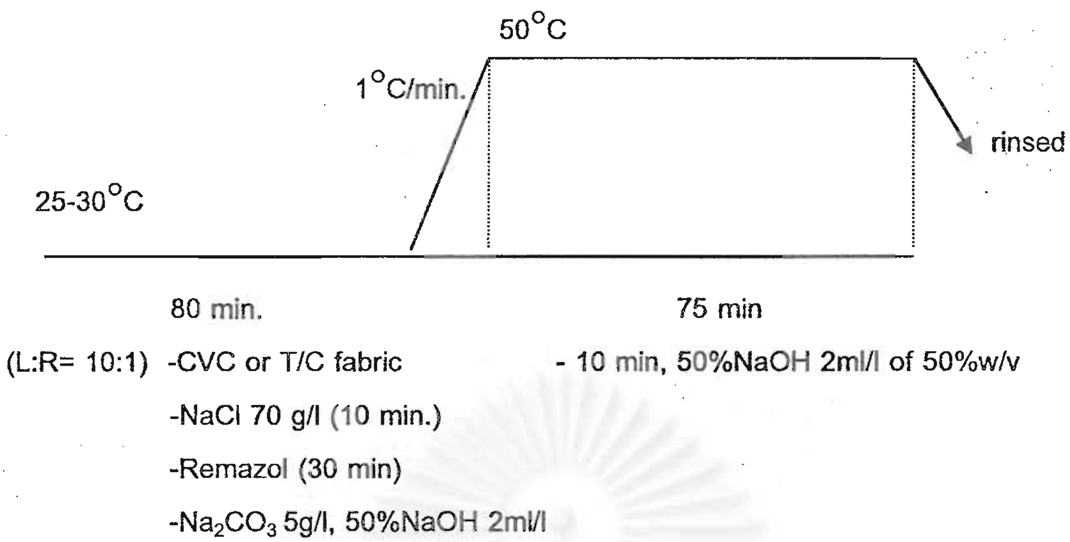
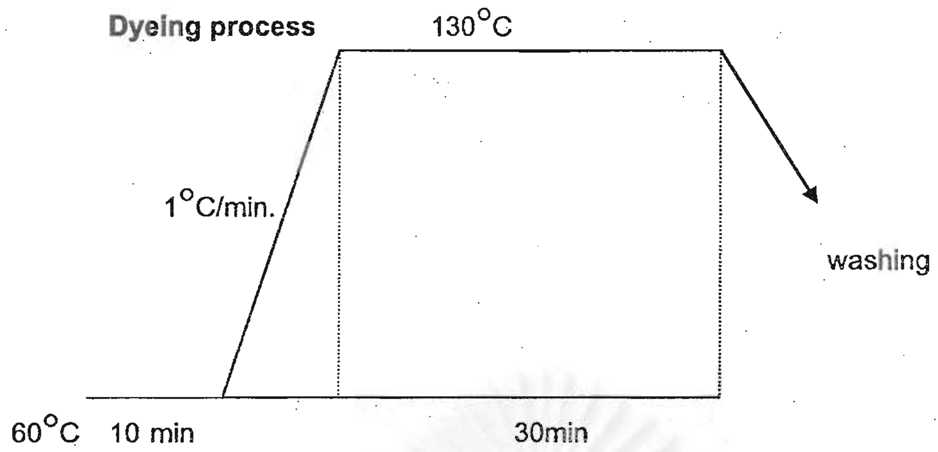


Diagram 3.7 The reactive dyeing process for CVC and T/C fabrics.

Scoured polyester fabrics were dyed in the solutions containing disperse dye and 1 g/l wetting agent at a liquor ratio of 20:1 at pH 4-5. The temperature of dyeing process was commenced at 60°C for 10 minutes, then raised to 130°C (1°C/minute) and maintained at this temperature for 30 minutes (see Diagram 3.8). After dyeing, the fabrics were washed in water containing 2 g/l wetting agent at 60°C for 15 minutes. The unfixed disperse dye was removed from the fabric using the reduction clear process shown in Diagram 3.9. This process required 2 g/l sodium hydrosulfite, 2 g/l sodium hydroxide and 1 g/l wetting agent, and it was conducted at 80°C for 15 minutes. Then the fabrics were washed and air dried.



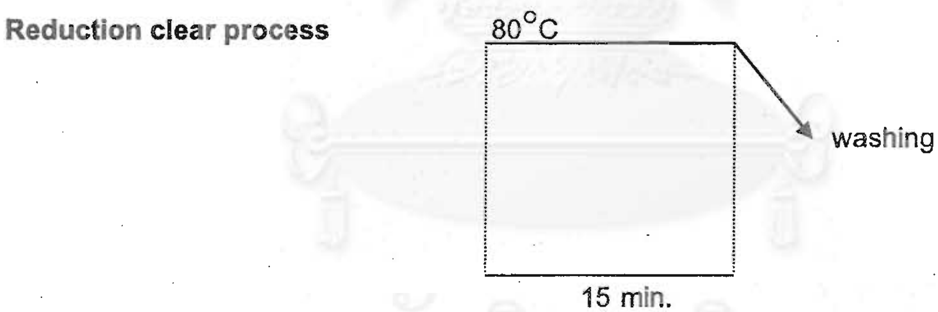
(L:R= 20:1)- Polyester fabric

-pH 4-5

-Wetting agent 1 g/l

-Dianix

Diagram 3.8 The dyeing process for polyester fabric.



(L:R= 20:1)-$Na_2S_2O_4$ 2g/l

-NaOH 2 g/l

-Wetting agent 1 g/l

Diagram 3.9 The reduction clear process for polyester fabric after disperse dyeing.

Scoured nylon fabrics were dyed with an acid dye, milling acid dye 2% o.w.f. in the laboratory dyeing machine at a liquor ratio of 20:1 and at pH 4-5. The dyeing process was commenced at 40°C for 10 minutes, raised to 100°C (2°C/minute), and maintained at this temperature for 45 minutes. The dyed fabrics were then removed from the dye solution, washed with 2 g/l wetting at 50°C for 15 minutes. Finally they were rinsed thoroughly in running tap water, squeezed, and air dried. The dyeing process is illustrated in Diagram 3.10.

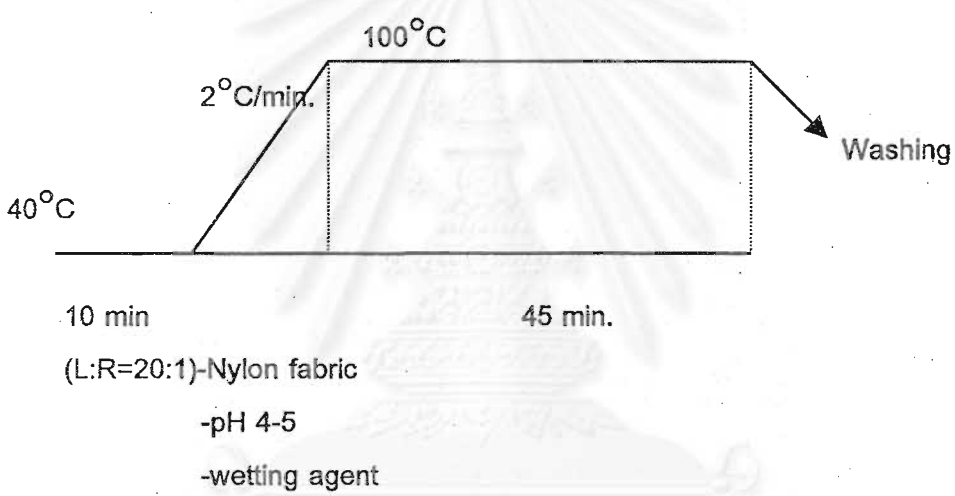


Diagram 3.10 The dyeing process for nylon fabric.

After dyeing, all dyed samples were measured for the color strength (K/S) at a specific wavelength between 520-570nm using the Macbeth® reflectance spectrophotometer. The apparent color strength can be expressed as K/S using the Kubelka-Munk equation as follows.

$$K/S = \frac{(1-R)^2}{2R} \qquad \text{(equation 3.2)}$$

where K is the absorption coefficient

S is the scattering coefficient

R is the reflectance of dyed fabric at the maximum absorption wavelength of dye

Within the same dyeing process, the fabric with higher K/S value showed darker shade than the fabric with lower K/S value. Each fabric was tested for three times and the data were averaged.

3.4.8 Fabric Strength

Knitted fabrics were tested for bursting strength using the Standard for Method of Testing for Textiles, Volume 19, "Diaphragm Bursting Strength and Bursting Distension Tester Method [40]". Bursting strength is the maximum pressure (kg/m^2) of the fluid that pushed the test fabric until break down. The testing was conducted by laying the fabric on the diaphragm that has a fluid pressure control in the bursting strength machine. Then the fabric was locked with the covering and when started, the machine supplied the pressure that could break the fabrics. Each fabric was tested for ten times and the data were averaged.

Woven fabrics were tested for tensile strength using ASTM D 5035-95, "Tensile Properties of Woven Fabric: Revealed Strip Test-1R [41]". A test specimen was clamped in a tensile testing machine and force was applied to the stretch specimen until it broke. The breaking force and the elongation of the test specimen were read from the machine. The test was conducted for five times at the warp direction and eight times at the weft direction and the data were averaged.

3.4.9 Stiffness Testing

Polyester fabric was tested for its stiffness using the stiffness tester according to the standard test method of ASTM D1388 – 96, "Stiffness of

Fabrics [42]". The test was conducted in order to observe whether the enzymatic treatment on polyester fabric can soften the fabric.

A specimen was laid on the stiffness tester, then it was slided at a specific rate in a direction parallel to the long dimension of the apparatus. The length of the overhang was measured when the tip of the specimen was depressed under its own mass to the point where the line joining the top to the edge of the platform made a 41.5^{o} with the horizontal. From this measured length, the bending length and flexural rigidity were calculated by equation 3.3.

$$G = W.c^{3} \qquad \text{(equation 3.3)}$$

where G is the flexural rigidity, mg.cm

W is the fabric weight in mass per unit area, mg/cm^{2}

c is the bending length, cm

3.4.10 Scanning Electron Microscope

Fabrics were examined for the appearance of the fiber surface using the Scanning Electron Microscope (SEM). The samples were prepared by mounting on SEM stubs, and coating with gold in the nanometer level by sputter-coater. The gold was inonized during scanning using the Argon gas in the vacumm condition.

CHAPTER 4

Results and Discussion

4.1 Greige Fabrics

Greige fabrics were tested for various properties according to the test procedures outlined in section 3.4 and their properties are shown in Table 4.1.

Table 4.1 Properties of various greige fabrics.

Fabric Property	Cotton fabric	CVC fabric	T/C fabric	Polyester fabric	Nylon fabric
Weight (g/100 cm^2)	1.1614	1.9404	1.7825	2.0808	0.5967
Water absorbency	Did not absorb water				
Whiteness	-5.998	1.015	19.967	66.450	71.576
Water soluble extractable material (%)	2.460	1.430	0.980	—	—
Solvent soluble extractable material(%)	0.320	0.210	0.160	—	—
MB(g) on substrate(kg) (presence of pectin)	10.580	8.760	6.600	—	—
Bursting strength(kg/cm^2)	6.030	10.690	11.310	—	—
Breaking load in warp direction(N)	—	—	—	688.280	412.580
Breaking load in weft direction(N)	—	—	—	310.860	330.500
Stiffness in warp direction(mg.cm.)	—	—	—	452.440	328.980
Stiffness in weft direction(mg.cm.)	—	—	—	358.300	160.680

MB = Methylene blue

All greige fabrics did not absorb water due to the hydrophobic substances coated on the fiber surface. Only polyester and nylon fabrics contained high whiteness. Extractable materials in cotton fabric were higher than in cotton/polyester blends and so did the methylene blue content or the presence of pectins.

4.2 Scoured Cotton Fabrics

Prewashed cotton fabric was scoured using the conventional and the enzymatic processes. It was then tested for the water absorbency, the fabric strength, the weight loss, the presence of pectins, the whiteness, the dye absorption, and the appearance of the fiber surface. The results are shown as follows.

4.2.1 Water Absorbency of the Conventional Scoured Cotton Fabrics

In general, the first priority required in a scoured fabric is the fabric must be uniformly wet with water within 3 seconds at room temperature. For this work, a more rigorous standard was used to signify an adequate absorbency. The scoured fabric would have an adequate absorbency only when it absorbed water immediately and uniformly once water was dropped on the fabric surface. Table 4.2 shows the water absorbency of the conventional scoured cotton fabric.

Table 4.2 Water absorbency of the conventional scoured cotton and the recommended scouring formulation.

Fabric	Sodium hydroxide (% owf)	Womine TE (g/l)	Temp. (°C)	Time (minute)	Water absorbency*
Cotton fabric	3	3	80	60	A

*A= Absorbed immediately

After scouring the cotton fabric using the formulation in Table 4.2, the fabric showed an adequate absorbency. It absorbed water instantaneously once water was applied onto the fabric surface. This is because most hydrophobic substrates coated on the fiber surface were removed by scouring and the fiber was left with a hydrophilic character.

4.2.2 Water Absorbency of the Enzymatic Scoured Cotton Fabric

In this experiment the fabric was enzymatic scoured using lipase, protease, and cellulase individually in one step scouring and all together in one step and two steps scouring. It was then tested for its water absorbency and the result is shown in Table 4.3.

Table 4.3 Water absorbency of the enzymatic scoured cotton fabric and the scouring formulations.

Fabric	Trial	Enzyme			Condition**			Water absorbency*
		Step	Type	g/l	Temp. (°C)	pH	Time (min.)	
Cotton fabric	1	1	Lipase	0.50	37	8.0	30	D
	2	1	Lipase	0.50	37	8.0	30	A
		2	Cellulase	0.50	40	4.5	30	
	3	1	Protease	0.50	37	7.0	30	D
	4	1	Protease	0.50	37	7.0	30	A
		2	Cellulase	0.50	40	4.5	30	
	5	1	Cellulase	0.50	40	4.5	30	C
	6	1	Lipase	0.25	37	7.5	30	D
		1	Protease	0.25				
	7	1	Lipase	0.25	37	7.5	30	A
		1	Protease	0.25				
		2	Cellulase	0.50	40	4.5	30	

*A= Absorbed immediately B= Absorbed within 1-3 seconds

C= Absorbed in 1 minute D= Stayed as water drop

**1 g/l wetting agent was added in every steps

The water absorbency results shown in Table 4.3 indicate that scouring cotton fabric using lipase (trial 1), protease (trial 3), or cellulase (trial 5) alone, or using a lipase/protease combination (trial 6) could not generate a complete scouring result. The scoured fabric did not absorb water instantaneously. But once the fabric was scoured first with lipase and then with cellulase (trial 2), with protease and then with cellulase (trial 4), or with a lipase/protease combination and then with cellulase (trial 7), the scoured fabric showed an adequate absorbency. Lipase, protease, and cellulase were less effective for cotton scouring when each enzyme was used alone and so did a combination use of lipase and protease.

Scouring with lipase or protease or lipase/protease, and followed with cellulase could accomplish a perfect scouring result. Lipase could have catalyzed the hydrolysis of the hydrophobic substrates, such as fats and oils located on the fiber surface, and turned them into small water soluble molecules, moving away from the fiber. Protease could also catalyze the hydrolysis of the protein compounds and convert them into small amino acids dissolving in water. Both enzymes could remove only a few parts of the impurities causing some cracks on the hydrophobic layers. Anyway the fabric still could not absorb water instantaneously. These cracking areas might be too small for water molecules to penetrate immediately once water was dropped on the fiber surface. Water would need times longer than a few minutes to completely penetrate into the fiber.

As cellulase was applied at the second step of scouring, water could have carried cellulase through the cracking area to the cellulose layers and catalyzed the hydrolysis of cellulose into glucose and small sugar molecules, dissolving in water. Hydrolysis of cellulose could lead to a loss of some cotton fibers and cause more departure of the impurities. All these reactions finally had made cotton fabric being able to absorb water adequately.

4.2.3 Strength and Weight Loss of the Conventional and the Enzymatic Scoured Cotton Fabrics

Scoured cotton fabrics were tested for the fabric bursting strength and the fabric weight loss (%) and the results are shown in Table 4.4 and Figures 4.1 and 4.2.

Table 4.4 % Weight loss and bursting strength of unscoured and scoured cotton fabrics.

Scouring Procedure*	% Weight loss	Bursting Strength	
		(kg/cm^2)	% change**
No scouring	0.00	6.03	0.00
With NaOH	1.08	6.26	+3.81
With Lipase/Cell	0.85	6.15	+1.99
With Protease/Cell	0.83	6.36	+5.47
With Li+Pro/Cell	0.78	6.34	+5.14

* Li = Lipase Pro = Protease Cell = Cellulase

** + = strength increased - = strength decreased

Results in Table 4.4 and Figure 4.1 indicate that the conventional scoured fabric lost 1 % of fabric weight after scouring while the enzymatic scoured fabrics lost 0.8 %. In general, cotton fibers contain approximately 6% of impurities. From this work, it was found that 0.8-1% of impurities and a few fibers were removed in order to acquire an adequate absorbency of the fabric.

Sodium hydroxide scouring process could remove more impurities and fibers from the fabric than the enzymatic scouring process but both processes produced scoured fabrics with an adequate absorbency. Various enzymatic scouring processes could help removing approximately the same amount of impurities and fibers from the fabric, especially when the fabric was scouring with lipase/cellulase or with protease/cellulase. This result could mean that the oils/fats and proteins, hydrolyzed by the catalysis of lipase and protease respectively, could exist in a mixture form coating on the fiber rather than individual layers of them and this could support this experiment that either lipase or protease could be used to scour in the first step, then to follow with the cellulase scouring in the second step.

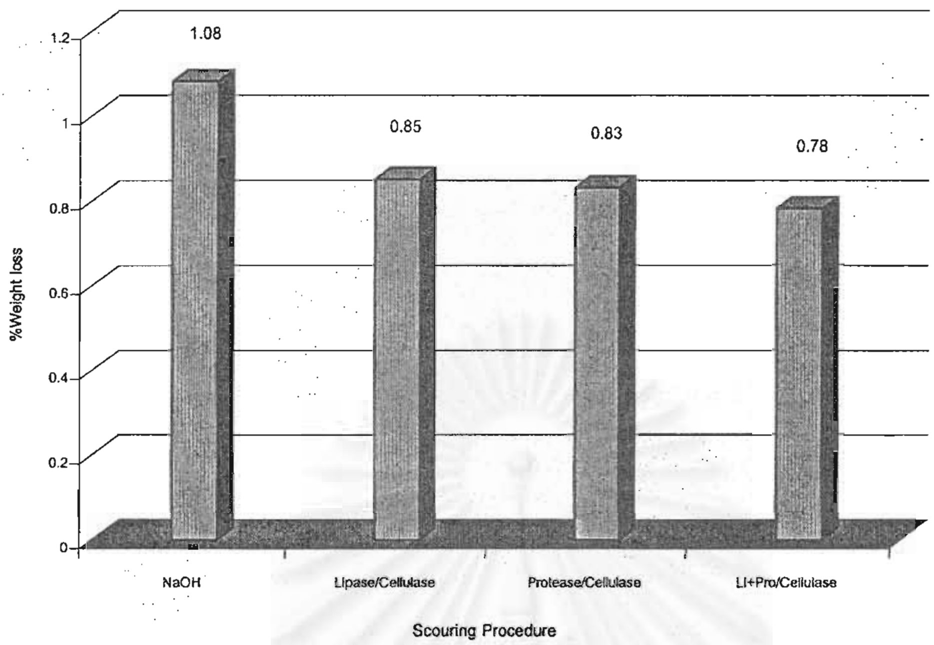


Figure 4.1 % Weight loss of scoured cotton fabrics, compared with unscoured.

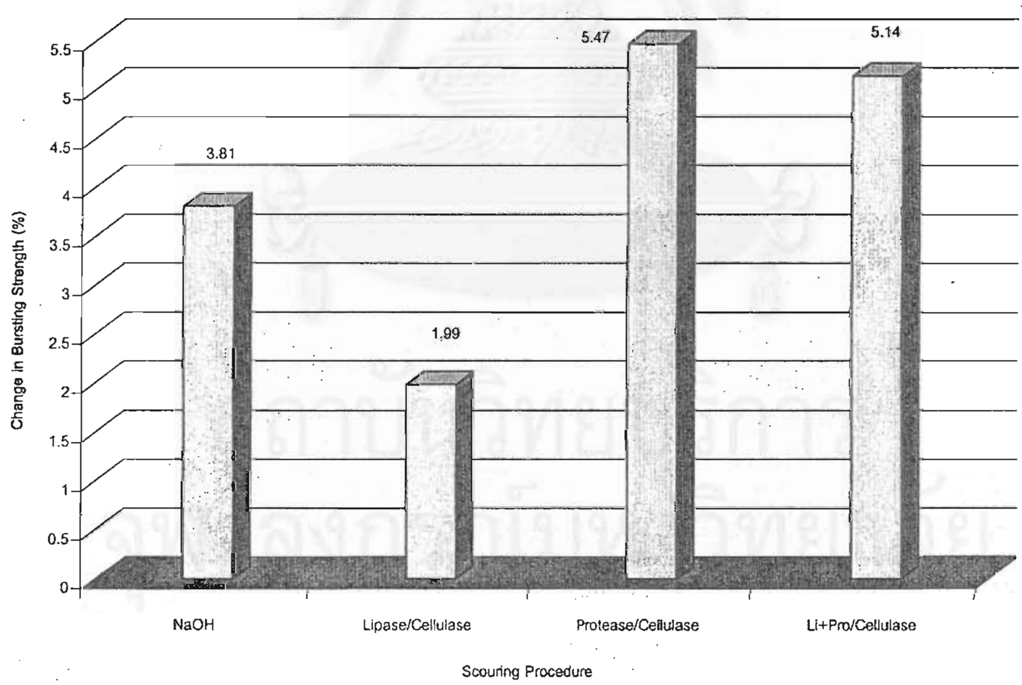


Figure 4.2 % Change in bursting strength of scoured cotton fabrics, compared with unscoured.

When a combination use of lipase and protease was utilized in the first scouring step followed with cellulase in the second step, this process produced a scoured fabric with a slight lower weight loss than the former two enzymatic scouring processes. This could be because the amount of lipase and protease each used was 50% lower than they were used in the former two processes and thus decreased the amount of substrates removing from the fabric.

Table 4.4 and Figure 4.2 show that all scoured fabrics gained strength after scouring although they lost 0.8-1% weight of impurities and fibers. According to Buschle-Diller, and Price [32, 43], an increase of the fabric strength after scouring could be due to an increase of the interfiber friction after the removal of the waxy substances. Results shown in Figure 4.2 indicate that the protease/cellulase and lipase+protease/cellulase scoured fabrics contained the highest % strength increase, followed by the sodium hydroxide scoured fabrics, and the lipase/cellulase scoured fabric, respectively. This could be due to the differences of the interfiber friction within the fabrics.

4.2.4 Presence of Pectins and Whiteness of the Conventional and the Enzymatic Scoured Cotton Fabrics.

Scoured cotton fabrics were tested for the precence of pectins and the whiteness and the results are shown in Table 4.5 and Figure 4.3.

The whiteness of fabrics shown in Table 4.5 indicates that all scoured fabrics contained whiteness higher than the unscoured fabric. The sodium hydroxide scoured fabric had its whiteness at 22.7 while the enzymatic scoured fabrics had their whiteness 17.3, 17.7, and 18.1. This means that the sodium hydroxide scouring process could remove the natural color substances from the fibers more than the enzymatic scouring process and various enzymatic scouring processes provided a similar capability of removing the natural color substances from the fibers. It is also reasonable to conclude that these color substances could exist in many places on the fiber, such as in the oils/fats/waxes and in the protein areas because any scouring agent used in this experiment could remove more or less natural color substances from the fibers.

Table 4.5 Presence of pectins (methylene blue content) and whiteness of unscoured and scoured cotton fabrics.

Scouring Procedure*	Whiteness	MB content** (g/kg)	%Reduction of MB content after scouring
No scouring	-5.998	10.58	0.00
With NaOH	22.712	7.75	26.75
With Li/Cell	17.335	8.41	20.51
With Pro/Cell	18.113	8.45	20.13
With Li+Pro/Cell	17.666	8.02	24.20

* Li = Lipase Pro= Protease Cell= Cellulase

**MB = methylene blue

Table 4.5 also shows the methylene blue content on the unscoured and scoured cotton fabrics and the %reduction of methylene blue content on scoured cotton fabrics compared with unscoured is shown in Figure 4.3. To determine the presence of pectins on cotton fabric, the fabric was dyed in a solution of methylene blue and the amount of methylene blue on the fabric was measured. The fabric containing a high amount of methylene blue on means the fabric also contain a high amount of pectins.

Results in Table 4.5 and Figure 4.3 indicate that unscoured fabric had the highest amount of methylene blue, in other words, it contained the highest amount of pectins. After the fabric was scoured, the presence of pectins decreased more by the sodium hydroxide scouring process than by the enzymatic scouring process. Sodium hydroxide could help removing more pectins from the fabric than the enzymes. In this experiment, although pectinase enzyme was not used in the enzymatic scouring process but the enzymes used lipase/protease/cellulase could also help removing pectins from the fabric. Pectins could exist in a mixing from or in a matrix form attached with oils/fats/waxes and proteins, and could be removed together with the oils/fats/waxes and protein when the enzymatic scouring was conducted.

Using a combination lipase and protease in the first scouring step and cellulase in the second scouring step could help removing pectins a slightly better than using a single enzyme in the first step of scouring and cellulase in the second step.

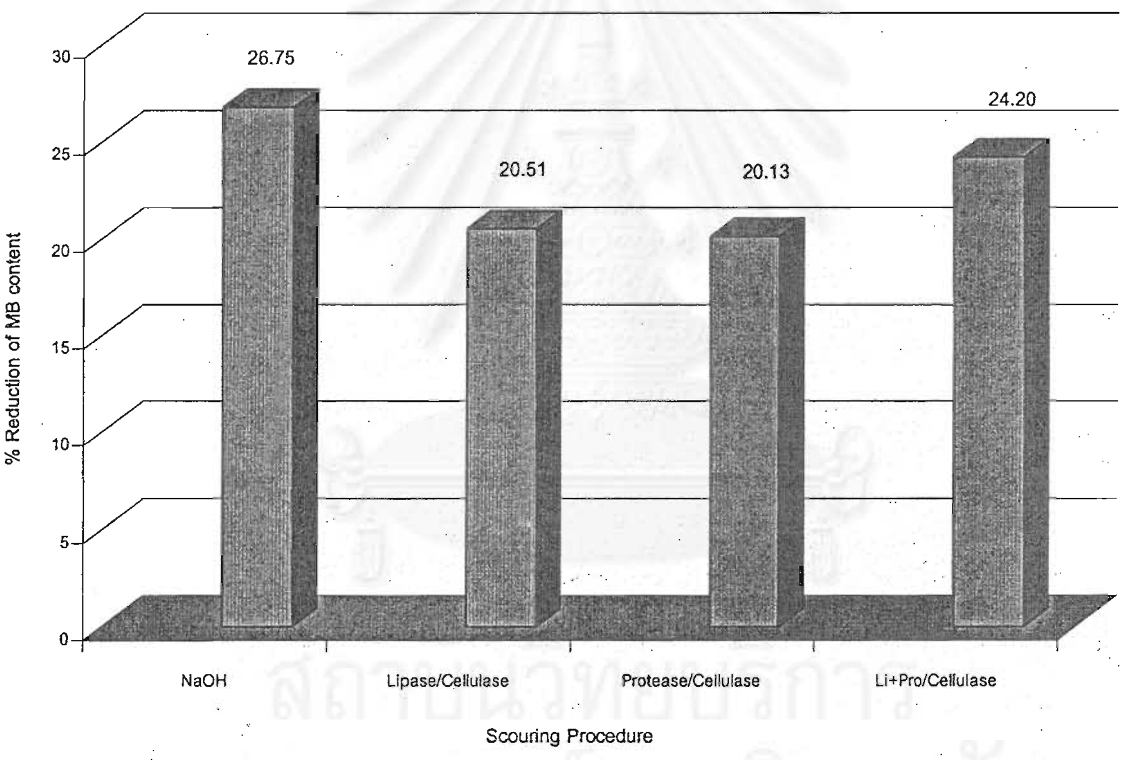


Figure 4.3 %Reduction of methylene blue content on scoured cotton fabrics, compared with unscoured.

4.2.5 Dye Absorption of the Conventional and the Enzymatic Scoured Cotton Fabrics

Table 4.6 and Figure 4.4 show the dye absorption result of the scoured cotton fabrics in terms of the fabrics color strength or K/S value. The dyed fabric containing a high color strength also show a high dye absorption. In this experiment, the sodium hydroxide scoured fabric had a slightly higher color strength (7.9) than the enzymatic scoured fabrics (6.5-6.7). In general, the differences of the color strength within 1-2 numbers are insignificant. Therefore in this case, all scouring processes produced fabrics with same dyeability, especially the enzymatic scouring processes.

Table 4.6 Color strength of scoured knitted cotton fabric.

Scouring Procedure*	K/S
With NaOH	7.867
With Li/Cell	6.710
With Pro/Cell	6.514
With Li+Pro/Cell	6.531

* Li = Lipase   Pro= Protease   Cell= Cellulase

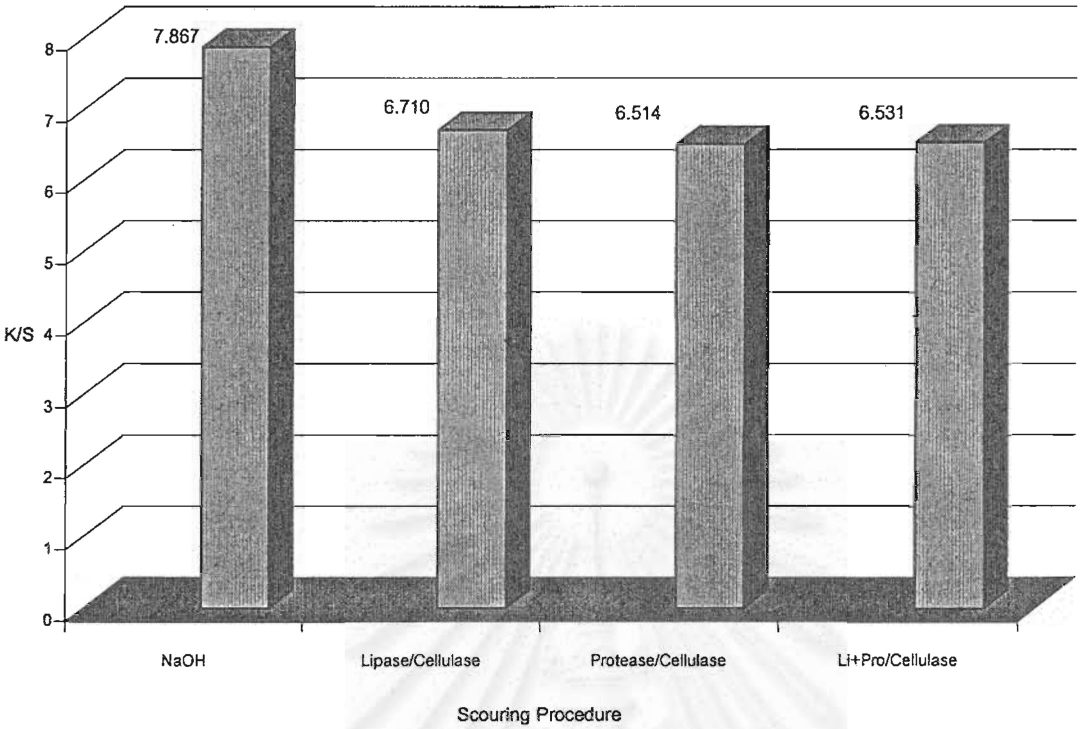


Figure 4.4 Color strength of scoured cotton fabrics.

4.2.6 Fiber Surface Morphology of the Conventional and the Enzymatic Scoured Cotton Fabrics

Unscoured and scoured cotton fabrics were observed for the appearance of the fiber surface using the SEM technique and the result is shown in Figure 4.5 (a) to (f). Before scouring, cotton fibers had a rough surface with no crack mark and no fibril protruding from the fiber. There were some substances covered the fiber surface. After scouring with sodium hydroxide, most substances covering the fiber were removed. There were some crack marks shown on the fiber and some fibrils protruding from the fiber surface. Less fibrils were shown on the fiber when the enzymatic scouring process was conducted, but more substances covering the fiber were left and the fiber showed some cracks. Sodium hydroxide had performed very well in term of removing the substances covering the fiber although leaving more fibrils on the fiber. The appearances of the fiber surface of all three enzymatic scoured cotton fabrics were very similar as mention earlier.

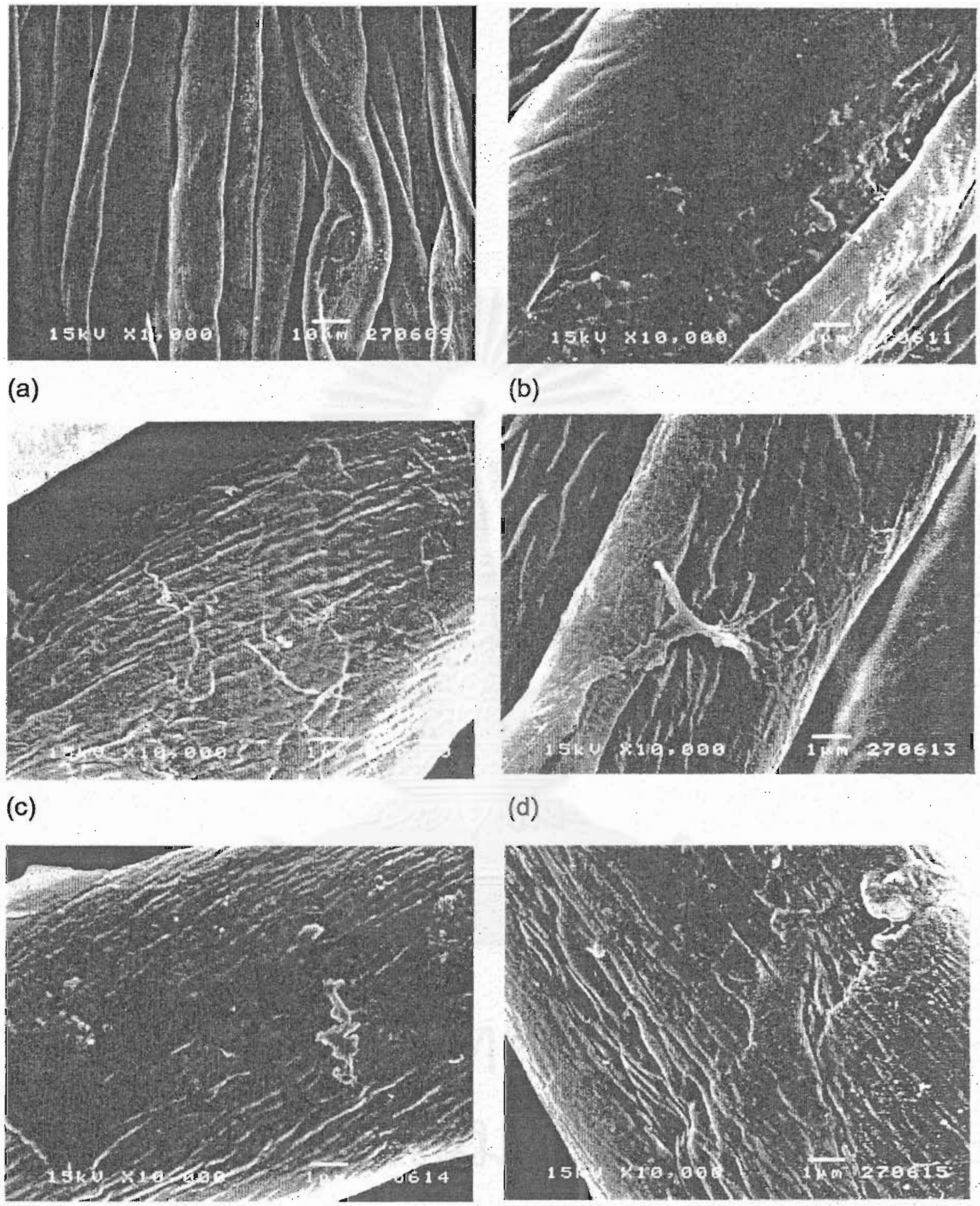


Figure 4.5 SEM micrographs of greige (X1,000) and scoured cotton fibers (x10,000) (a) greige cotton, (b) greige cotton, (c) sodium hydroxide scoured cotton, (d) lipase/cellulase scoured cotton, (e) protease/cellulase scoured cotton, (f) lipase+protease/cellulase scoured cotton.

4.2.7 Conclusions of the Conventional and the Enzymatic Scouring of Cotton Fabric

Both conventional scouring using sodium hydroxide and enzymatic scouring using lipase, protease, and cellulase enzymes could produce good quality scoured cotton fabrics with an adequate absorbency, a loss of 0.8-1.0 %fabric weight, increases of strength and whiteness, and a good dyeability.

4.3 Scoured CVC (Cotton/Polyester; 55/45) Fabric

Prewashed CVC fabric was scoured using the conventional and the enzymatic processes. It was then tested for properties the same way as conducted on the cotton fabric. The results are shown as follows.

4.3.1 Water Absorbency of the Conventional Scoured CVC Fabric

Table 4.7 shows the water absorbency of the conventional scoured CVC fabric. After scouring using the formulation in Table 4.7, the fabric showed an adequate absorbency. Most hydrophobic substances coated on the fiber could have been removed properly. These substances included the natural substances from cotton fiber and the synthetic oils from polyester and may also from cotton.

Table 4.7 Water absorbency of the conventional scoured CVC fabric and the recommended scouring formulation.

Fabric	Sodium hydroxide (% owf)	Womine TE (g/l)	Temp. ($^{\circ}C$)	Time (minute)	Water absorbency*
CVC fabric	0.5	3	80	60	A

*A= Absorbed immediately

4.3.2 Water Absorbency of the Enzymatic Scoured CVC Fabric

Prewashed CVC fabric was enzymatic sscoured using the same scouring procedure as conducted on the cotton fabric. Table 4.8 shows the water absorbency results of the scoured fabric and the scouring formulations. Scouring CVC fabric using lipase (trials 1-7), protease (trials 11-17), or a lipase/protease combination (trials 21-27) could not generate a complete scouring result. The scoured fabric did not absorb water instantaneously. But once the fabric was scoured first with lipase and then with cellulase (trials 8-10), with protease and then with cellulase (trials 18-20), or with a lipase/protease combination and then with cellulase (trials 28-30), the scoured fabric showed an adequate absorbency. All these results were very similar to those from the enzymatic scoured cotton fabric. The only difference was that CVC fabric could be effectively scoured using cellulase alone (trial 32). Lipase and protease were less effective than the cellulase for the CVC scouring when each enzyme was used alone and so did a combination use.

Table 4.8 Water absorbency of the enzymatic scoured CVC fabric and the scouring formulations.

Fabric	Trial	Enzyme			Condition**			Water Absorbency*
		Step	Type	g/l	Temp. (°C)	pH	Time (min.)	
CVC fabric	1	1	Lipase	0.5	37	8	30	D
	2	1	Lipase	1.0	37	8	30	C
	3	1	Lipase	2.0	37	8	30	B
	4	1	Lipase	3.0	37	8	30	B

Table 4.8(continued)

Fabric	Trial	Enzyme			Condition**			Water Absorbency*
		Step	Type	g/l	Temp. (°C)	pH	Time (min.)	
CVC fabric	5	1	Lipase	4.0	37	8	30	C
	6	1	Lipase	5.0	37	8	30	C
	7	1	Lipase	10.0	37	8	30	C
	8	1	Lipase	0.5	37	8	30	A
		2	Cellulase	0.5	40	4.5	30	
	9	1	Lipase	1.0	37	8	30	A
		2	Cellulase	0.5	40	4.5	30	
	10	1	Lipase	2.0	37	8	30	A
		2	Cellulase	0.5	40	4.5	30	
	11	1	Protease	0.5	37	7	30	C
	12	1	Protease	1.0	37	7	30	C
	13	1	Protease	2.0	37	7	30	B
	14	1	Protease	3.0	37	7	30	B
	15	1	Protease	4.0	37	7	30	B
	16	1	Protease	5.0	37	7	30	B
	17	1	Protease	10.0	37	7	30	B
	18	1	Protease	0.5	37	7	30	A
		2	Cellulase	0.5	40	4.5	30	
	19	1	Protease	1.0	37	7	30	A
		2	Cellulase	0.5	40	4.5	30	
	20	1	Protease	2.0	37	7	30	A
		2	Cellulase	0.5	40	4.5	30	

Table 4.8(continued)

Fabric	Trial	Enzyme Step	Enzyme Type	Enzyme g/l	Condition** Temp. (°C)	Condition** pH	Condition** Time (min.)	Water Absorbency*
CVC fabric	21	1	Lipase	0.25	37	7.5	30	C
		1	Protease	0.25				
	22	1	Lipase	0.5	37	7.5	30	D
		1	Protease	0.5				
	23	1	Lipase	1.0	37	7.5	30	C
		1	Protease	1.0				
	24	1	Lipase	1.5	37	7.5	30	C
		1	Protease	1.5				
	25	1	Lipase	2.0	37	7.5	30	C
		1	Protease	2.0				
	26	1	Lipase	2.5	37	7.5	30	C
		1	Protease	2.5				
	27	1	Lipase	5.0	37	7.5	30	C
		1	Protease	5.0				
	28	1	Lipase	0.25	37	7.5	30	A
		1	Protease	0.25				
		2	Cellulase	0.5	40	4.5	30	
	29	1	Lipase	0.5	37	7.5	30	A
		1	Protease	0.5				
		2	Cellulase	0.5	40	4.5	30	
	30	1	Lipase	1.0	37	7.5	30	A
		1	Protease	1.0				
		2	Cellulase	0.5	40	4.5	30	
	31	1	Cellulase	0.5	40	4.5	30	C
	32	1	Cellulase	1.0	40	4.5	30	A

*A= Absorbed immediately B= Absorbed within 1-3 second C= Absorbed in 1 minute D= Stayed as water drop

**1 g/l wetting agent was added in every steps

4.3.3 Strength and Weight Loss of the Conventional and the Enzymatic Scoured CVC Fabrics

Table 4.9 and Figure 4.6 indicate that the scoured fabrics lost only 0.35- 0.55% of fabric weight after scouring. Both conventional and enzymatic scouring equally removed impurities from the fabrics. This could be because the CVC fabric contained less impurities to be removed than the cotton fabric and thus lost a smaller weight. The cellulase scoured fabric lost the highest weight due to the highest hydrolysis of the cellulose, compared with other scoured fabrics.

The bursting strength of scoured fabrics shown in Table 4.9 and Figure 4.7 illustrates that most scoured fabrics gained strength after scouring except the cellulase scoured fabric which lost only 0.53%. the reason of strength increase could be the same as for the scoured cotton fabric, but for the strength decrease could be due to the hydrolysis of the cellulose. The strength increase of scoured CVC fabric was lower than of scoured cotton fabric and this could be because of the lower interfiber friction between cotton fibers and polyester fibers in CVC fabric than that among cotton fibers in cotton fabric.

Table 4.9 % Weight loss and bursting strength of unscoured and scoured CVC fabrics.

Scouring Procedure*	% Weight loss	Bursting Strength	
		(kg/cm^2)	% change**
No scouring	0.00	10.69	0.00
With NaOH	0.50	10.91	2.06
With Li/Cell	0.51	10.81	1.12
With Pro/Cell	0.38	10.83	1.31
With Li+Pro/Cell	0.35	10.70	0.09
With Cell	0.55	10.63	-0.53

*Li = Lipase Pro = Protease Cell = Cellulase

** + = strength increased - = strength decreased

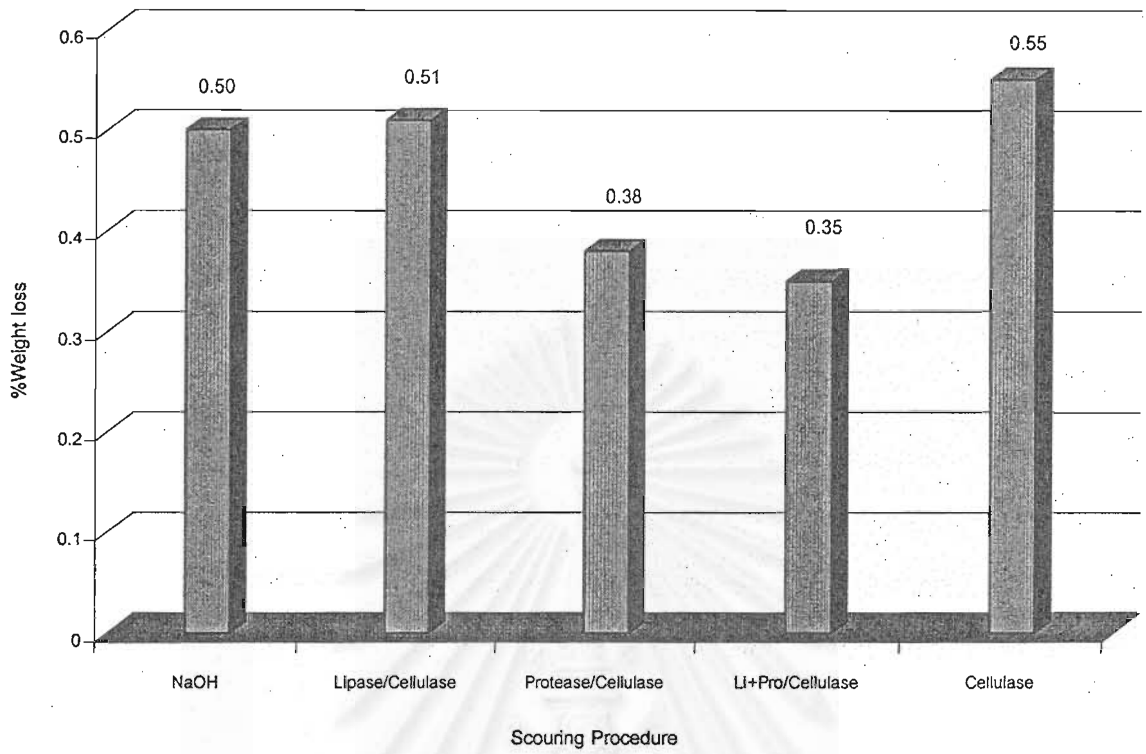


Figure 4.6 %Weight loss of scoured CVC fabrics, compared with unscoured.

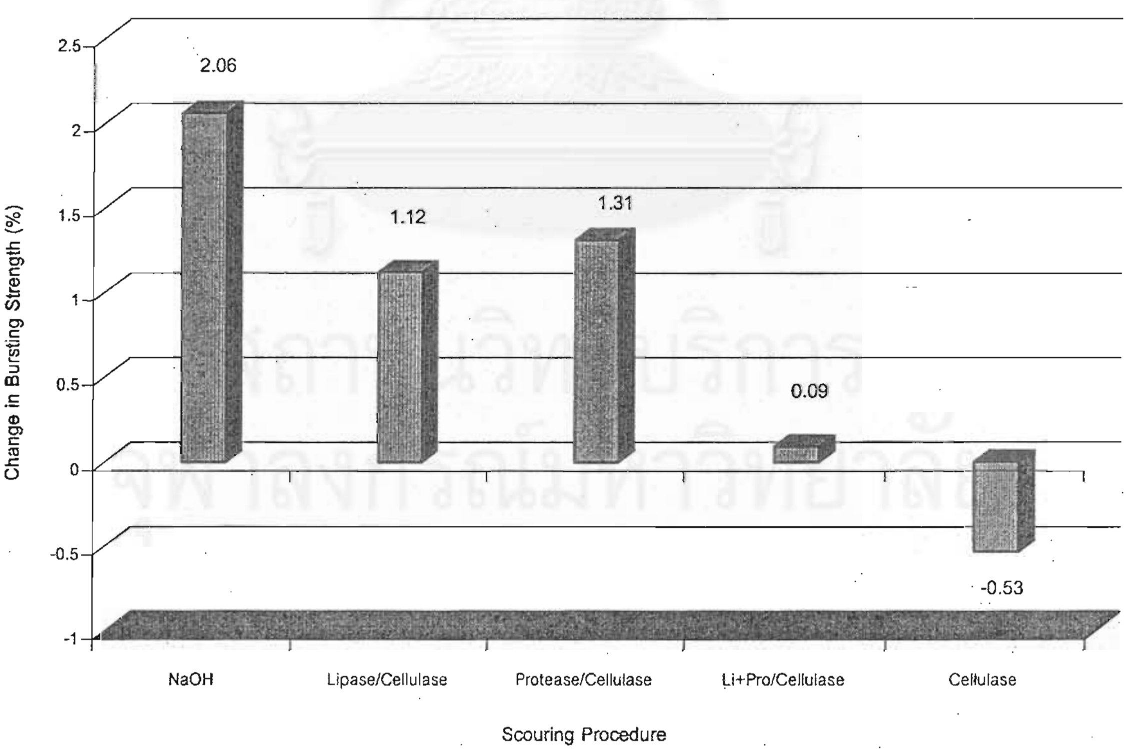


Figure 4.7 %Change in bursting strength of scoured CVC fabrics, compared with unscoured.

4.3.4 Presence of Pectins and Whiteness of the Conventional and the Enzymatic Scoured CVC Fabrics

Scoured CVC fabrics were tested for the presence of pectins and the whiteness and the results are shown in Table 4.10 and Figure 4.8.

After scouring, all CVC fabrics had higher whiteness than the unscoured. Sodium hydroxide scouring process produced whiter fabric than the enzymatic scouring process, as also took place in the cotton scouring. All enzymatic scoured fabrics contained a similar whiteness between 26.4 -28.2. Scoured CVC fabrics had higher whiteness than the scoured cotton fabrics. This could be because the CVC fabric consisted of 55% cotton and 45% polyester, and thus there were less natural color substances to be removed from cotton and more synthetic white color substances (TiO_2) inside the polyester.

The methylene blue contents of various scoured CVC fabrics shown in Table 4.10 and Figure 4.8 between 5.79-6.67 which was lower than the unscoured. This means that pectins were removed from the fabric after each scouring procedure. Cellulase scouring process could remove the highest amount of pectins from the fabric and this could be the reason that the cellulase scoured fabric lost the highest weight and lost strength.

Table 4.10 Presence of pectins (methylene blue content) and whiteness of unscoured and scoured CVC fabrics.

Scouring Procedure*	Whiteness	MB** Content (g/kg)	%Reduction of MB Content after scouring
No scouring	1.015	8.76	0.00
With NaOH	35.698	6.30	28.08
With Li/Cell	26.962	6.23	28.88
With Pro/Cell	28.207	6.36	27.40
With Li+Pro/Cell	26.392	6.67	23.86
With Cellulase	27.003	5.79	33.90

*Li = Lipase Pro= Protease Cell= Cellulase

**MB = methylene blue

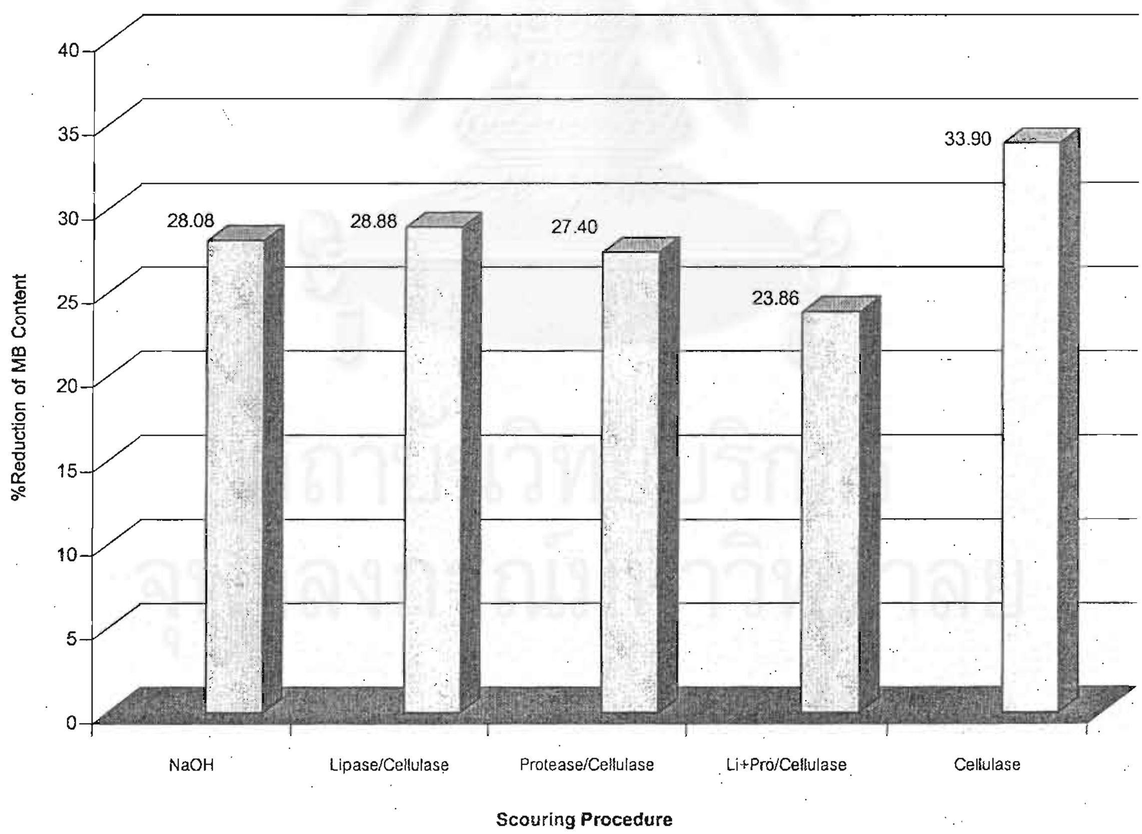


Figure 4.8 %Reduction of methylene blue content on scoured CVC fabrics, compared with unscoured.

4.3.5 Dye Absorption of the Conventional and Enzymatic Scoured CVC Fabrics

Scoured CVC fabrics were dyed with disperse and reactive dyes and the result is shown in Table 4.11 and Figure 4.9. All scoured fabrics show similar color strength between 25.5-26.4 which is insignificant. Both sodium hydroxide scouring process and enzymatic scouring process could effectively scour CVC fabrics to acquire an equal dyeability.

Table 4.11 Color strength of scoured knitted CVC fabric.

Scouring Procedure*	K/S
With NaOH	25.638
With Li/Cell	25.516
With Pro/Cell	26.354
With Li+Pro/Cellulase	26.107
With Cellulase	26.430

* Li = Lipase Pro= Protease Cell= Cellulase

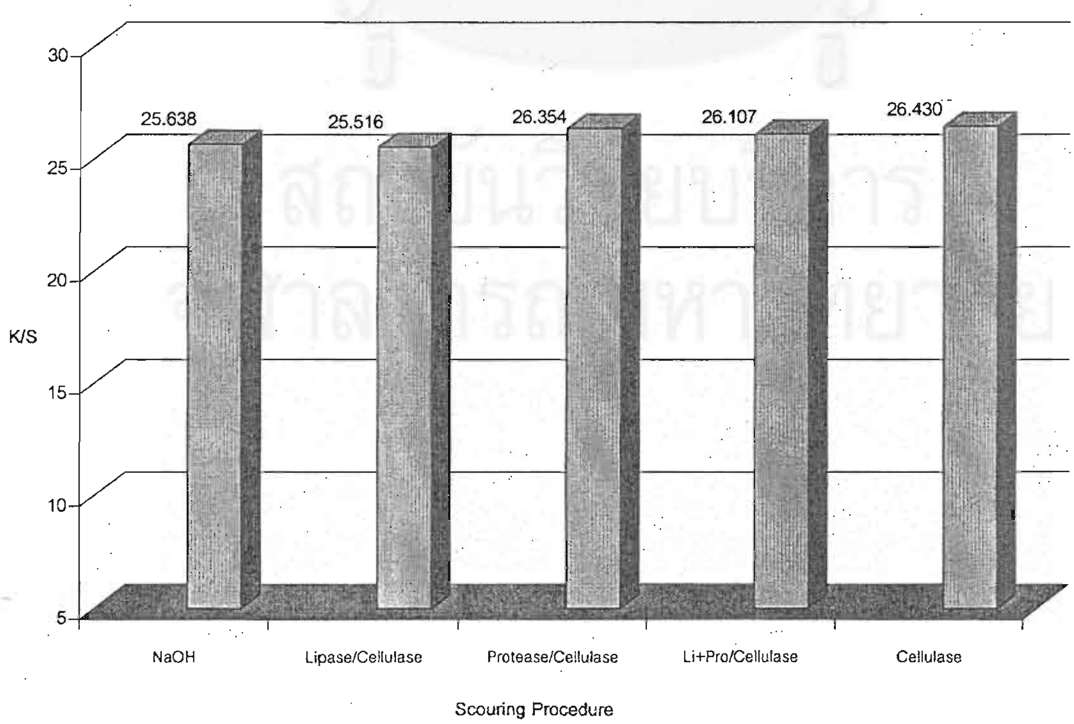


Figure 4.9 Color strength of scoured CVC fabrics.

4.3.6 Fiber Surface Morphology of the Conventional and the Enzymatic Scoured CVC Fabrics

Figure 4.10 shows pictures of cotton/polyester fibers in CVC fabrics both before and after scouring. Unscoured fibers (Picture a) show impurities on the fiber surface. Sodium hydroxide scoured fibers in Picture (b) still had impurities and a few crack marks on the surface. Scouring CVC fabric using lipase, protease, and cellulase enzymes produced some fibrils on the fiber surface in which cellulase scouring alone could produce the highest amount of fibrils. There were still some impurities left on the fiber surface after any enzymatic scouring.

4.3.7 Conclusions of the Conventional and the Enzymatic Scouring of CVC Fabric

Both conventional scouring using sodium hydroxide and enzymatic scouring using lipase, protease, and cellulase enzymes could produce good quality scoured CVC fabric with an adequate absorbency, a very low weight loss (0.35-0.55%), an increase of strength (except the cellulase scoured), an increase of whiteness, and a good dyeability.

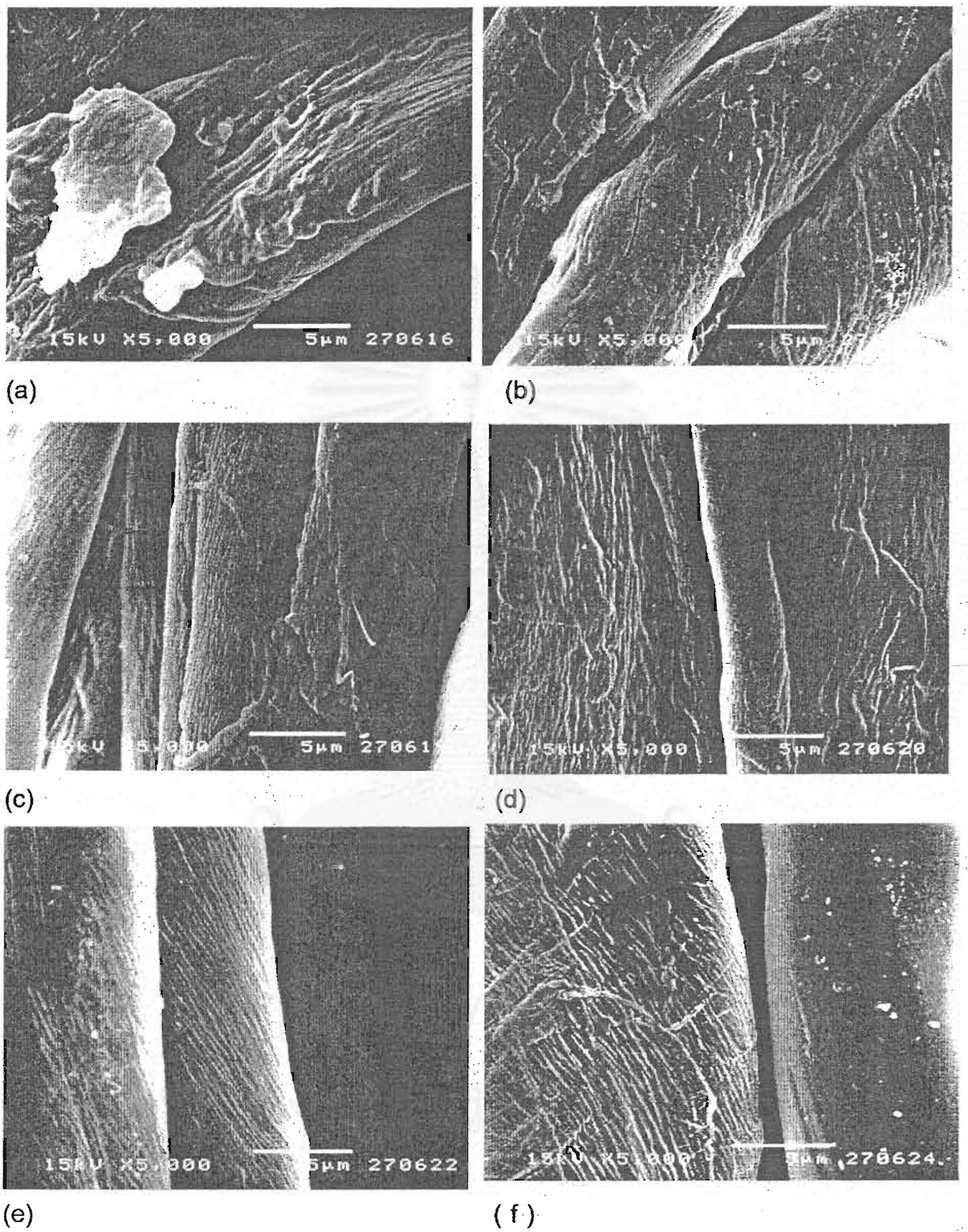


Figure 4.10 SEM micrographs of greige (x 5,000) and scoured CVC [cotton / polyester; 55/45] fiber(x 5,000): (a) greige CVC, (b) Sodium hydroxide scoured CVC (c) lipase/cellulase scoured CVC, (d) protease/cellulase scoured CVC (e) lipase+protease/cellulase scoured CVC (f) cellulase scoured CVC.

4.4 Scoured T/C (Cotton/Polyester; 35/65) Fabric

Prewashed T/C fabric was scoured using the conventional and the enzymatic processes. It was then tested for properties the same way as conducted on the cotton and the CVC fabrics. The results are shown as follows.

4.4.1 Water Absorbency of the Conventional Scoured T/C Fabric

Table 4.12 shows the water absorbency of the conventional scoured T/C fabric. Prewashed T/C fabric was scoured using the same conventional scouring procedure as conducted on the CVC fabric and the scoured fabric had an adequate absorbency.

Table 4.12 Water absorbency of the conventional scoured T/C fabric and the recommended scouring formulation.

Fabric	Sodium hydroxide (% owf)	Womine TE (g/l)	Temp. ($^{\circ}C$)	Time (minute)	Water absorbency*
T/C fabric	0.5	3	80	60	A

*A= Absorbed immediately

4.4.2 Water Absorbency of the Enzymatic Scoured T/C Fabric

Prewashed T/C fabric was enzymatic scoured using the same scouring procedure as conducted on the cotton and the CVC fabrics. Table 4.13 shows the water absorbency results of the scoured fabric and the scouring formulations. One step scouring with lipase, protease, cellulase, or a combination lipase and protease could not scour the T/C fabric to acquire an adequate absorbency, and so did the two steps scouring with lipase then cellulase, protease then cellulase, or lipase and protease then cellulase. The enzyme concentration had been increased from 0.5 g/l to 10 g/l but still was not able to gain water absorbency of the scoured T/C fabric (trials 1-38). This could be because T/C fabric had a polyester content of 65% and a cotton content of 35%, and thus could contain a high amount of the synthetic oils on the fibers. These oils could have hindered the activity of the enzyme and thus made an ineffective scouring. If this is true, these oils should have been removed before conducting an enzymatic scouring on the T/C fabric. Therefore, greige T/C fabric was prewashed in a solution containing 1 g/l nonionic wetting agent in order to remove those synthetic oils. Then it was enzymatic scoured using the same procedure as the previous experiment. The water absorbency result indicates that only the two steps scouring mentioned previously could scour the T/C fabric to acquire an adequate water absorbency (trials 43-45).

Table 4.13 Water absorbency of the enzymatic scoured T/C fabric and the scouring formulations.

Fabric	Trial	Enzyme			Condition**			Water Absorbency*
		Step	Type	g/l	Temp. (°C)	pH	Time (min.)	
T/C fabric	1	1	Lipase	0.5	37	8	30	D
	2	1	Lipase	1.0	37	8	30	C
	3	1	Lipase	5.0	37	8	30	C
	4	1	Lipase	10.0	37	8	30	C
	5	1	Lipase	0.5	37	8	30	C
		2	Cellulase	0.5	40	4.5	30	
	6	1	Lipase	1.0	37	8	30	C
		2	Cellulase	0.5	40	4.5	30	
	7	1	Lipase	5.0	37	8	30	B
		2	Cellulase	0.5	40	4.5	30	
	8	1	Lipase	10.0	37	8	30	B
		2	Cellulase	0.5	40	4.5	30	
	9	1	Lipase	0.5	37	8	30	B
		2	Cellulase	1.0	40	4.5	30	
	10	1	Lipase	0.5	37	8	30	B
		2	Cellulase	2.0	40	4.5	30	
	11	1	Lipase	0.5	37	8	30	B
		2	Cellulase	5.0	40	4.5	30	
	12	1	Lipase	0.5	37	8	30	B
		2	Cellulase	10.0	40	4.5	30	
	13	1	Lipase	10.0	37	8	30	B
		2	Cellulase	10.0	40	4.5	30	
	14	1	Protease	0.5	37	7	30	C
	15	1	Protease	1.0	37	7	30	C
	16	1	Protease	5.0	37	7	30	C

Table 4.13 (continued)

Fabric	Trial	Enzyme			Condition**			Water Absorbency*
		Step	Type	g/l	Temp. (°C)	pH	Time (min.)	
T/C fabric	17	1	Protease	10.0	37	7	30	C
	18	1	Protease	0.5	37	7	30	C
		2	Cellulase	0.5	40	4.5	30	
	19	1	Protease	1.0	37	7	30	C
		2	Cellulase	0.5	40	4.5	30	
	20	1	Protease	5.0	37	7	30	B
		2	Cellulase	0.5	40	4.5	30	
	21	1	Protease	10.0	37	7	30	B
		2	Cellulase	0.5	40	4.5	30	
	22	1	Protease	0.5	37	7	30	B
		2	Cellulase	1.0	40	4.5	30	
	23	1	Protease	0.5	37	7	30	B
		2	Cellulase	2.0	40	4.5	30	
	24	1	Protease	0.5	37	7	30	B
		2	Cellulase	5.0	40	4.5	30	
	25	1	Protease	0.5	37	7	30	B
		2	Cellulase	10.0	40	4.5	30	
	26	1	Protease	10.0	37	7	30	B
		2	Cellulase	10.0	40	4.5	30	
	27	1	Lipase	0.25	37	7.5	30	C
		1	Protease	0.25				
	28	1	Lipase	0.5	37	7.5	30	D
		1	Protease	0.5				
	29	1	Lipase	2.5	37	7.5	30	C
		1	Protease	2.5				
	30	1	Lipase	5.0	37	7.5	30	C
		1	Protease	5.0				

Table 4.13 (Continued)

Fabric	Trial	Enzyme			Condition**			Water Absorbency*
		Step	Type	g/l	Temp. (°C)	pH	Time (min.)	
T/C fabric	31	1	Lipase	0.25	37	7.5	30	B
		1	Protease	0.25				
		2	Cellulase	0.5	40	4.5	30	
	32	1	Lipase	0.5	37	7.5	30	C
		1	Protease	0.5				
		2	Cellulase	0.5	40	4.5	30	
	33	1	Lipase	2.5	37	7.5	30	C
		1	Protease	2.5				
		2	Cellulase	0.5	40	4.5	30	
	34	1	Lipase	5.0	37	7.5	30	B
		1	Protease	5.0				
		2	Cellulase	0.5	40	4.5	30	
	35	1	Lipase	0.25	37	7.5	30	B
		1	Protease	0.25				
		2	Cellulase	1.0	40	4.5	30	
	36	1	Lipase	0.25	37	7.5	30	B
		1	Protease	0.25				
		2	Cellulase	2.0	40	4.5	30	
	37	1	Lipase	0.25	37	7.5	30	B
		1	Protease	0.25				
		2	Cellulase	5.0	40	4.5	30	
	38	1	Lipase	0.25	37	7.5	30	B
		1	Protease	0.25				
		2	Cellulase	10.0	40	4.5	30	

Table 4.13 (Continued)

Substrates	Trial	Enzyme			Condition			Water Absorbency*
		No.	Type	g/l	Temp. (°C)	pH	Time (min.)	
TC fabric (prewashed with1 g/l wetting agent)	39	1	Lipase	0.5	37	8	30	C
	40	1	Protease	0.5	37	7	30	C
	41	1	Lipase	0.25	37	7.5	30	C
		1	Protease	0.25				
	42	1	Cellulase	0.5	40	4.5	30	C
	43	1	Lipase	0.5	37	8	30	A
		2	Cellulase	0.5	40	4.5	30	
	44	1	Protease	0.5	37	7	30	A
		2	Cellulase	0.5	40	4.5	30	
	45	1	Lipase	0.25	37	7.5	30	A
		1	Protease	0.25				
		2	Cellulase	0.5	40	4.5	30	

*A= Absorbed immediately B= Absorbed within 1-3 seconds

C= Absorbed in 1 minute D= Stayed as water drop

**1 g/l wetting agent was added in every steps

4.4.3 Strength and Weight Loss of the Conventional and the Enzymatic Scoured T/C Fabrics

Scoured T/C fabrics were tested for the fabric bursting strength and the fabric weight loss and the results are shown in Table 4.14 and Figures 4.11-4.12.

Table 4.14 and Figure 4.11 indicate that the scoured T/C fabrics lost only 0.36-0.64% of fabric weight after scouring, approximately the same weight loss as the scoured CVC fabrics (0.35-0.55%) and the reason for this low weight loss could be the same as for the scoured CVC fabrics. Unexpectedly, the sodium hydroxide scoured fabric lost the least weight at 0.36% while the enzymatic scoured fabrics lost more weight at 0.50-0.64%.

The bursting strength of scoured T/C fabrics shown in Table 4.14 and Figure 4.12 illustrates that all scoured T/C fabrics gained strength after scouring. The reason for the increase of strength has been explained earlier in sections 4.2.3 and 4.3.3.

Table 4.14 % Weight loss and bursting strength of unscoured and scoured T/C fabrics.

Scouring Procedure*	% Weight loss	Bursting Strength	
		(kg/cm^2)	% change**
No scouring	0.00	11.31	0.00
With NaOH	0.36	11.62	2.74
With Li/Cell	0.50	11.46	1.33
With Pro/Cell	0.64	11.36	0.44
With Li+Pro/Cell	0.60	11.55	2.12

*Li = Lipase Pro = Protease Cell = Cellulase

** + = strength increased - = strength decreased

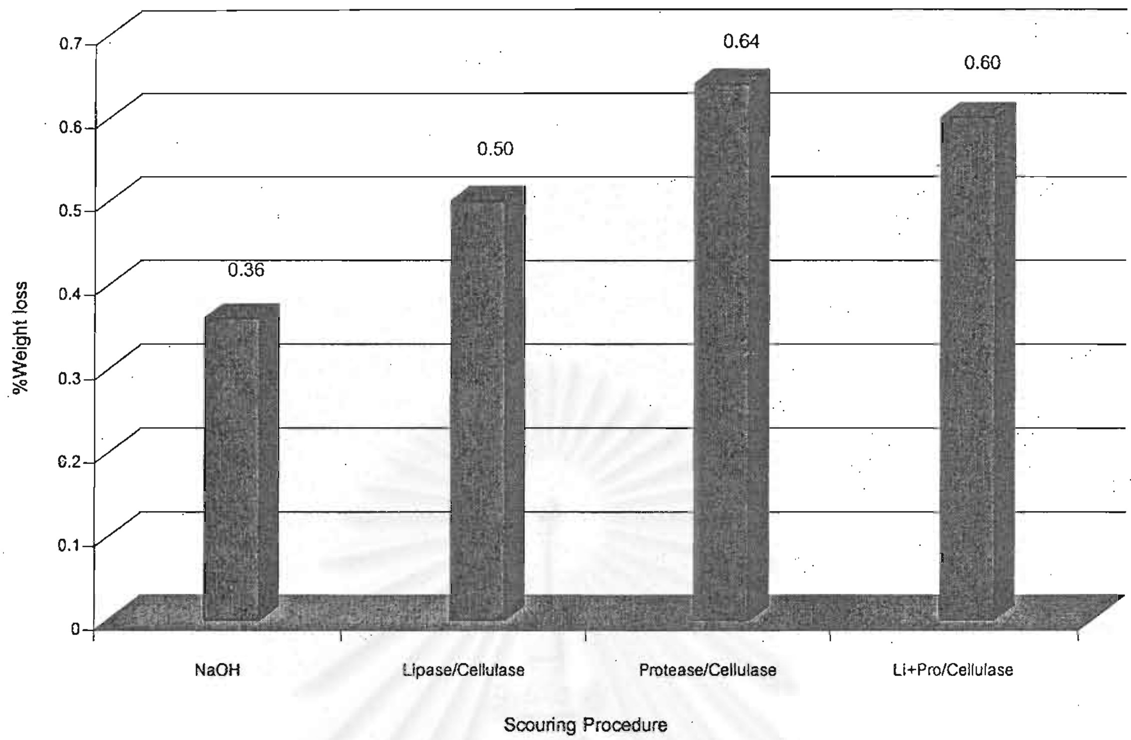


Figure 4.11 %Weight loss of scoured T/C fabrics, compared with unscoured.

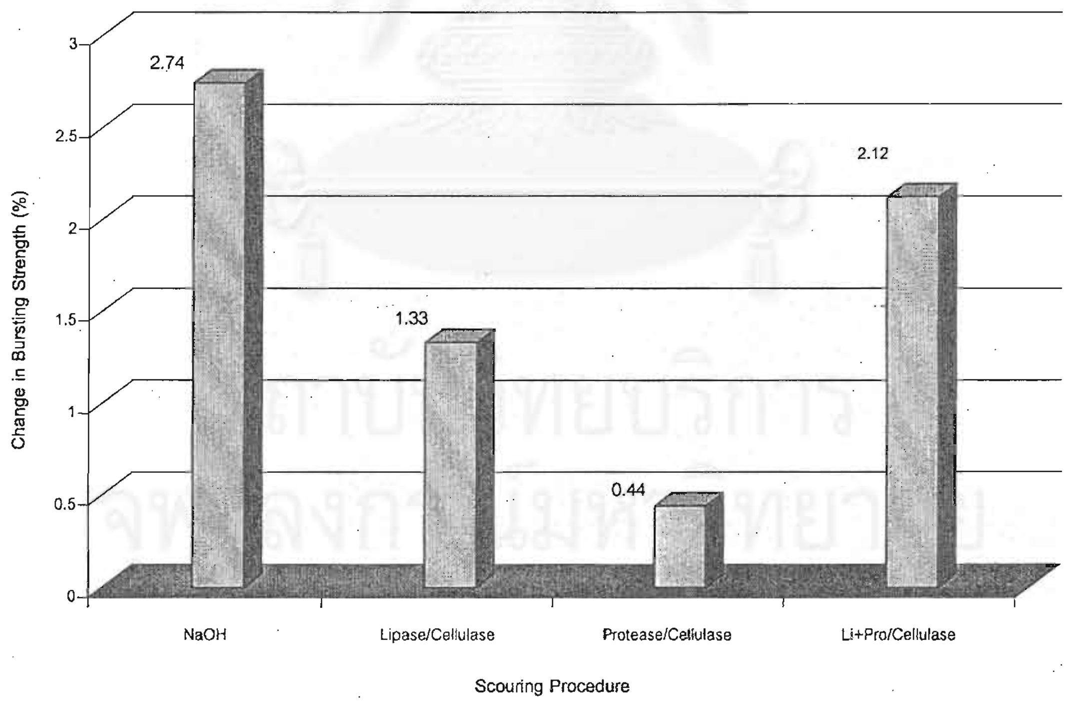


Figure 4.12 Change in bursting strength of scoured T/C fabrics, compared with unscoured.

4.4.4 Presence of Pectins and whiteness of the Conventional and the Enzymatic Scoured T/C Fabrics

Scoured T/C fabrics were tested for the presence of pectins and the whiteness and the results are shown in Table 4.15 and Figure 4.13.

After scouring, all scoured fabrics gained in whiteness. Both sodium hydroxide scouring and enzymatic scouring produced fabrics with approximately the same whiteness. The T/C fabrics had higher whiteness than the CVC fabrics because T/C fabrics contained 20% polyester higher than the CVC fabrics and thus consisted more TiO_2 as white pigment than the CVC fabrics.

The methylene blue contents of various scoured T/C fabrics shown in Table 4.15 and Figure 4.13 were between 4.64-5.63 which was lower than the unscoured. Pectins were removed from the fabric after scouring. The enzymatic scouring process could remove more pectins from the T/C fabric than the sodium hydroxide scouring process and this could be the reason that the enzymatic scoured T/C fabrics lost more weight than the enzymatic scoured CVC fabrics.

Table 4.15 Presence of pectins (methylene blue content) and whiteness of unscoured and scoured T/C fabric.

Scouring Procedure*	Whiteness	MB** Content(g/kg)	%Reduction of MB Content after scouring
No scouring	19.967	6.60	0.00
With NaOH	38.36	5.63	14.70
With Li/Cell	39.083	5.11	22.58
With Pro/Cell	37.979	4.64	29.70
With Li+Pro/Cell	39.918	5.17	21.67

* Li = Lipase Pro= Protease Cell= Cellulase

**MB = methylene blue

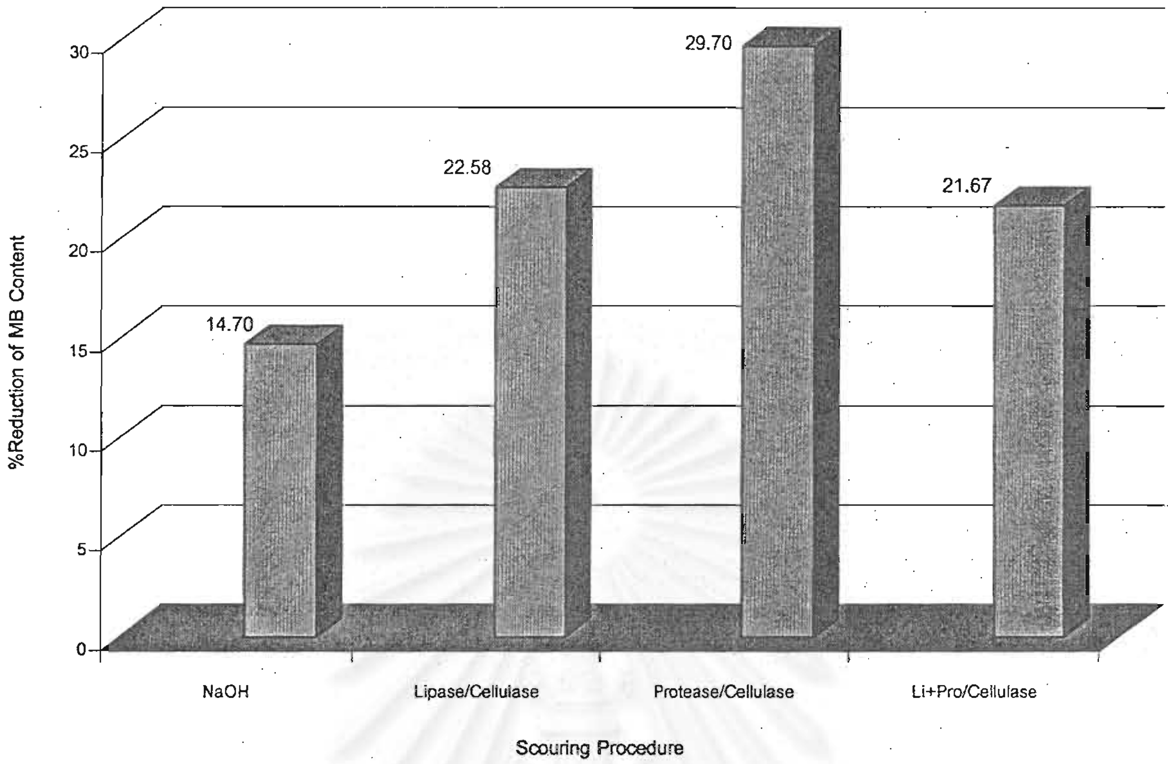


Figure 4.13 %Reduction of methylene blue content on scoured T/C fabrics, compared with unscoured.

4.4.5 Dye Absorption of the Conventional and the Enzymatic Scoured T/C Fabrics

Scoured T/C fabrics were dyed with disperse and reactive dyes and the result is shown in Table 4.16 and Figure 4.14. All scoured fabrics show similar color strength between 26.9-28.5 which is insignificant. Both sodium hydroxide scouring process and enzymatic scouring process produced scoured T/C fabrics with equal dyeability.

Table 4.16 Color strength of scoured knitted T/C fabric.

Scouring Procedure*	K/S
With NaOH	28.515
With Li/Cell	27.743
With Pro/Cell	26.943
With Li+Pro/Cell	27.405

* Li = Lipase   Pro= Protease   Cell= Cellulase

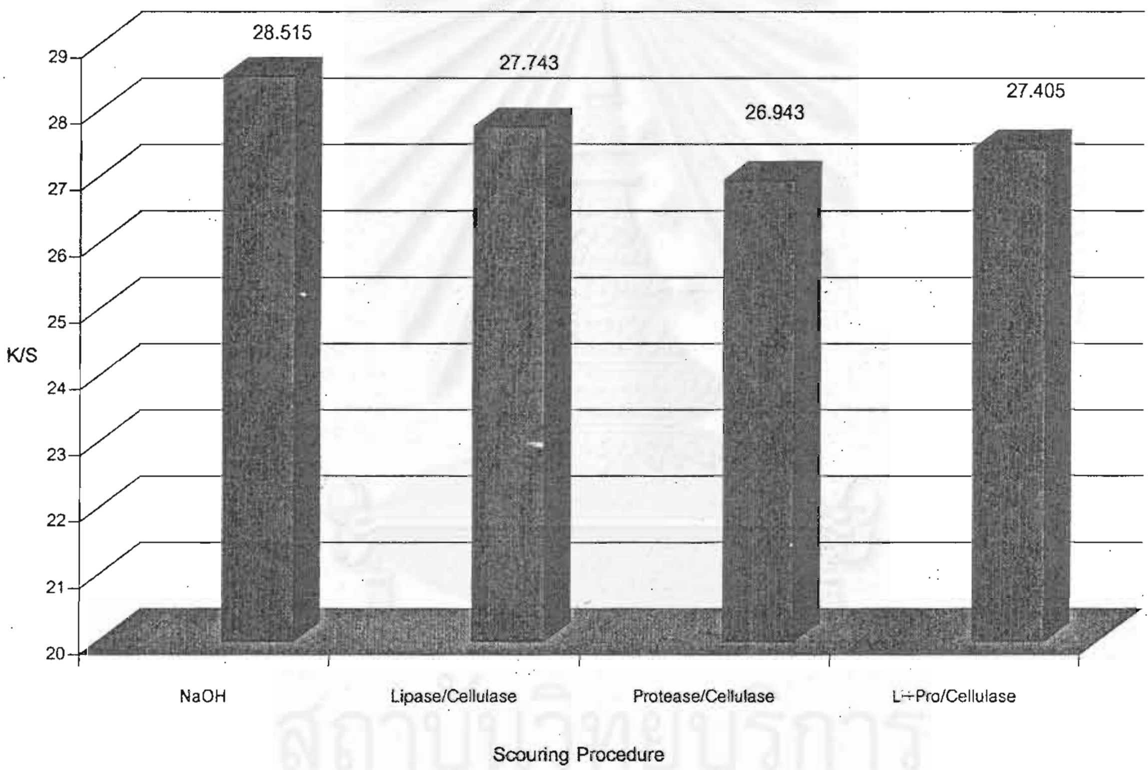


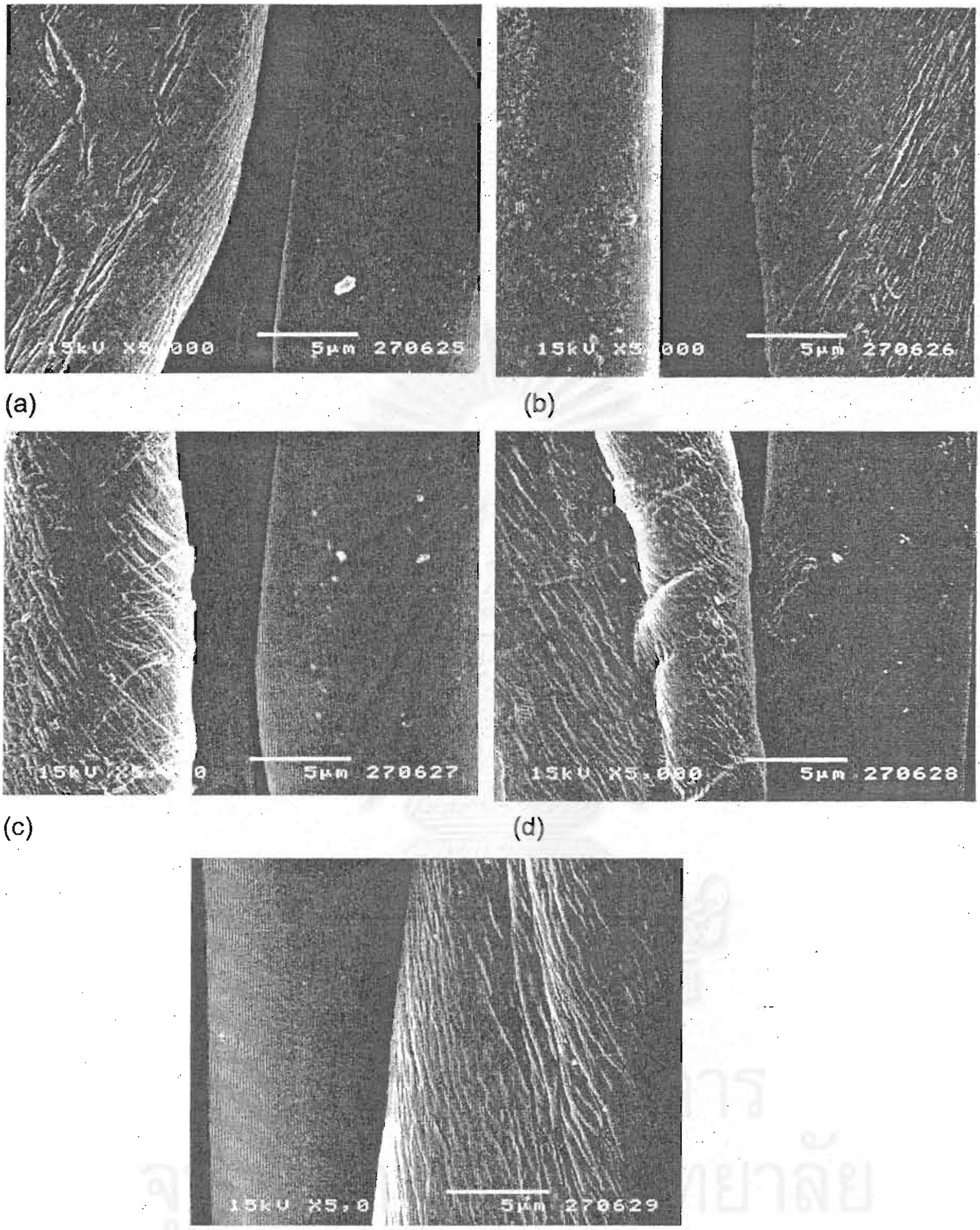
Figure 4.14 Color strength of scoured T/C fabrics.

4.4.6 Fiber Surface Morphology of the Conventional and the enzymatic Scoured T/C Fabrics

Figure 4.15 shows pictures of cotton/polyester fibers in T/C fabrics both before and after scouring. Unscoured fibers (picture a) show impurities on the fiber surface. All scoured fibers still had impurities on the fibers (pictures b-e) and some fibrils protruded from the cotton fibers.

4.4.7 Conclusions of the Conventional and the Enzymatic Scouring of T/C Fabric

To successfully scour T/C fabric using enzymes, the fabric was needed to be prewashed with a wetting agent in order to remove the oils coated on the fibers before conducting an enzymatic scouring. Both conventional scouring using sodium hydroxide and enzymatic scouring using lipase, protease, and cellulase enzymes could produce good quality scoured T/C fabric with an adequate absorbency, a very low weight loss, an increase of strength, an increase of whiteness and a good dyeability.



(a) (b) (c) (d) (e)

Figure 4.15 SEM micrographs of greige (x 5,000) and scoured T/C [cotton / polyester; 35/65] fibers (x 5,000): (a) greige T/C, (b) sodium hydroxide scoured T/C, (c) lipase/cellulase scoured T/C, (d) protease/cellulase scoured T/C, (e) lipase+protease/cellulase scoured T/C.

4.5 Scoured Polyester Fabric

Prewashed polyester fabric was scoured using the conventional and the enzymatic processes. It was then tested for the wetness, the fabric weight loss, the fabric breaking load, the fabric stiffness, the dye absorption, the whiteness, and the appearance of the fiber surface. The results are shown as follows.

4.5.1 Wetness of the Conventional Scoured Polyester Fabric

Table 4.17 shows the wetness of the conventional scoured polyester fabric. Prewashed polyester fabric was scoured using sodium carbonate and the scoured fabric had an adequate wetted.

Table 4.17 Wetness of the conventional scoured polyester fabric and the recommended scouring formulation.

Fabric	Sodium carbonate (g/l)	Womine TE (g/l)	Temp. ($^{\circ}C$)	Time (minute)	Wetness*
Polyester fabric	3	3	80	30	A

*A= wetted immediately

4.5.2 Wetness of the Enzymatic Scoured Polyester Fabric

Prewashed polyester fabric was first scoured using lipase enzyme at concentrations of 0.5-10 g/l at appropriate pH and temperature for 30 minutes. The wetness of the scoured fabrics indicates an inadequate wetness (trials 1-4 in Table 4.18). This could be because the synthetic oils coated on the polyester fiber hindered the activity of the lipase enzyme and thus produced an ineffective scouring. Therefore, polyester fabric was prewashed with a solution containing 1 g/l nonionic wetting agent before it was scoured with lipase enzyme. Only the fabric scoured with 3 g/l lipase and 3 g/l nonionic wetting agent provided an adequate wetness (trial 11 in Table 4.18). To successfully scour the polyester fabric using lipase enzyme, higher concentrations of lipase and wetting agent were needed than those needed for scouring cotton, CVC or T/C fabric. This could be because it was more difficult to hydrolyze the ester bond in polyester than to hydrolyze the ester bond in oils/fats from cotton fiber. After the polyester chains were was hydrolyzed at the ester bonds, the fiber in that area would be weaken and easily broken. Anyway, the fiber surface could be opened and could absorb more water.

Table 4.18 Wetness of the enzymatic scoured polyester fabric and the scouring formulations.

Fabric	Trial	Enzyme		Womine TE (g/l)	Condition			Wetness*
		Type	g/l		Temp. (°C)	pH	Time (min.)	
polyester fabric	1	Lipase	0.5	1	37	8	30	B
	2	Lipase	1	1	37	8	30	B
	3	Lipase	5	1	37	8	30	B
	4	Lipase	10	1	37	8	30	B
polyester fabric (prewashed with1 g/l wetting agent)	5	Lipase	0.5	1	37	8	30	B
	6	Lipase	1	1	37	8	30	B
	7	Lipase	1	3	37	8	30	B
	8	Lipase	1	5	37	8	30	C
	9	Lipase	3	1	37	8	30	B
	10	Lipase	5	1	37	8	30	B
	11	Lipase	3	3	37	8	30	A

*A= wetted immediately B= wetted within 1-3 seconds

C= wetted in 1 minute D= Stayed as water drop

4.5.3 Strength and Weight Loss of the Conventional and the Enzymatic Scoured Polyester Fabrics

Scoured polyester fabrics were tested for the fabric breaking load and the fabric weight loss and the results are shown in Table 4.19 and Figures 4.16-4.17.

After scouring, polyester fabrics lost 0.26% of fabric weight from lipase scouring and lost 0.32% of fabric weight from sodium carbonate scouring. The only impurity on the polyester fiber was oils and these oils could be removed from the fiber by scouring with either lipase or with sodium carbonate.

The strength of the scoured polyester fabric was determined by measuring the fabric breaking load due to its weaving structure. Table 4.19 and Figure 4.17 indicate that fabric breaking load increased in both warp and weft directions after scouring with either lipase or with sodium carbonate. Although there was a slight decrease of the breaking load in warp direction (-0.16) after scouring with sodium carbonate but it was insignificant. This could be due to an error of the testing equipment. The overall results illustrate that polyester fabric gained strength after scouring and this could be explained as follows. In general polyester fiber has a smooth surface and a hydrophobic character. Scouring polyester fiber with an alkali or with a lipase enzyme could alter the fiber surface from a smooth surface into a rougher surface and thus increase the water absorbency and also the interfiber friction.

Table 4.19 % Weight loss and breaking load of unscoured and scoured polyester fabrics.

Scouring Procedure	Weight Loss(%)	Breaking Load(N)			
		Warp	% Change*	Weft	% Change*
No scouring	0.00	688.28	0.00	310.86	0.00
With Na_2CO_3	0.32	687.18	-0.16	333.50	6.79
With Lipase	0.26	815.20	15.57	362.60	14.27

* + = breaking load increased - = breaking load decreased

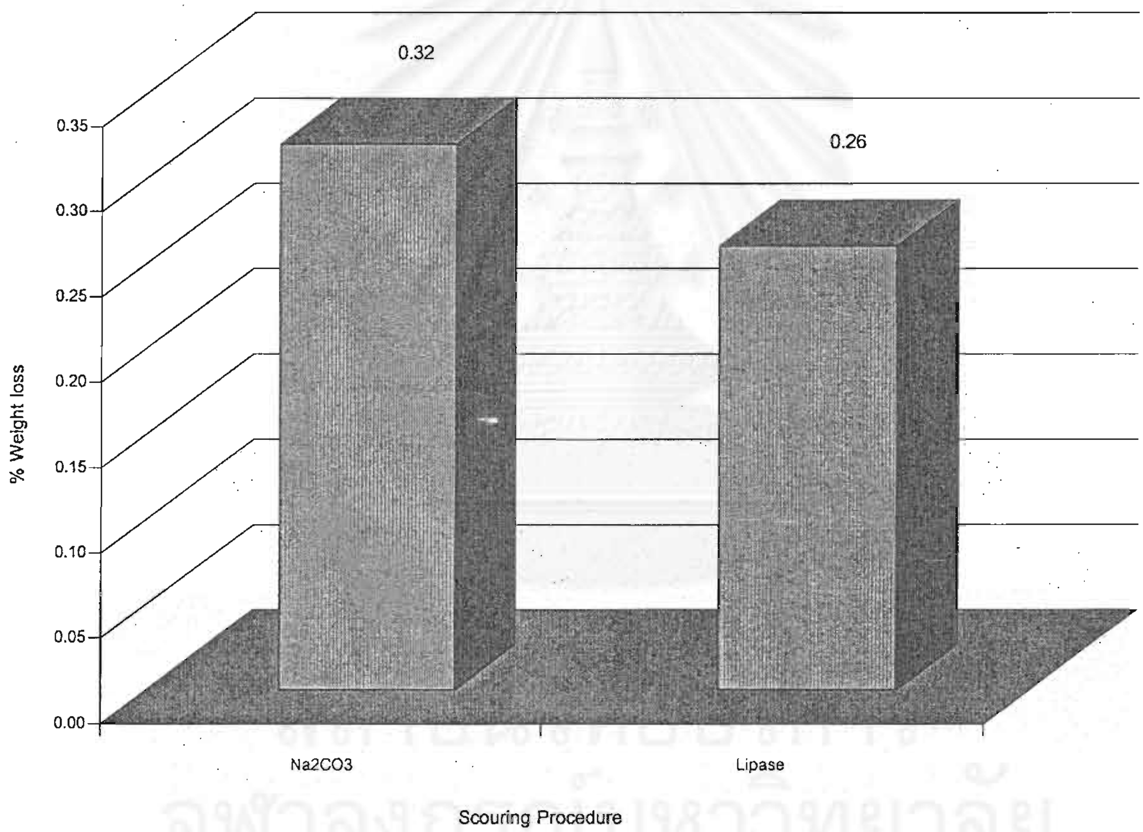


Figure 4.16 %Weight loss of scoured polyester fabrics, compared with unscoured.

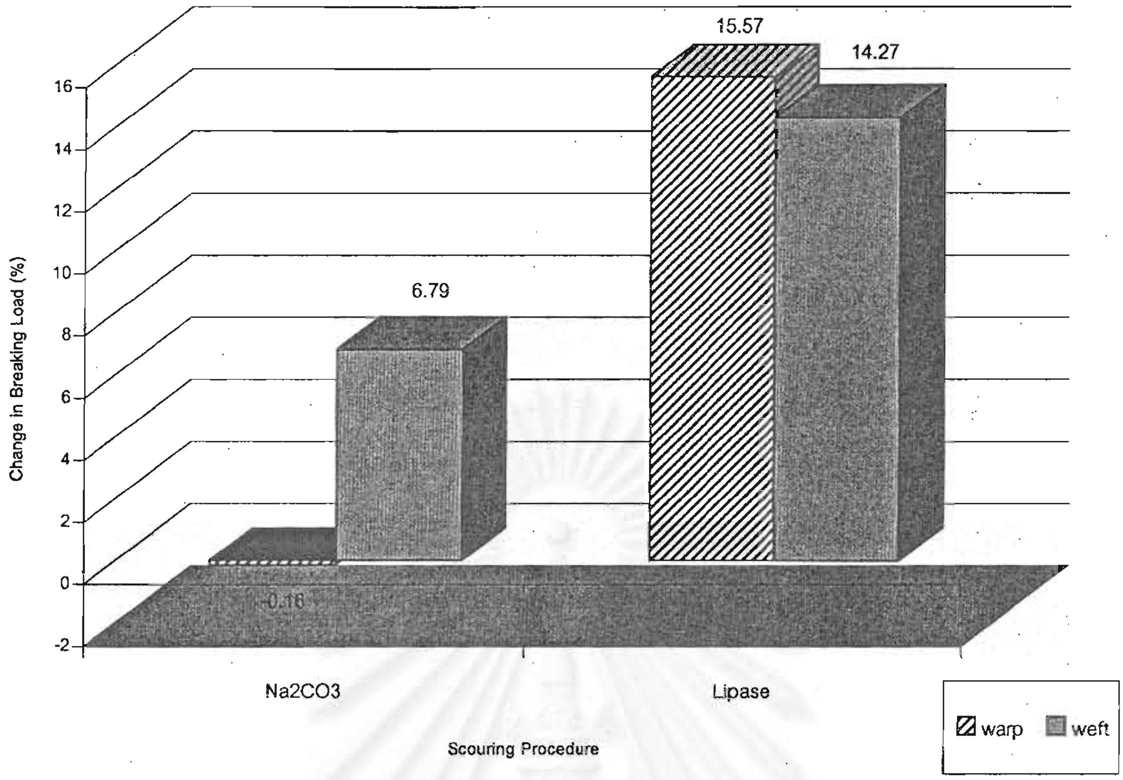


Figure 4.17 Change in breaking load of scoured polyester fabrics, compared with unscoured.

4.5.4 Stiffness of the Conventional and the Enzymatic Scoured Polyester Fabrics

Scoured polyester fabrics were tested for the bending length and the fabric stiffness was calculated as shown in Table 4.20.

After scouring, the polyester fabrics decreased in stiffness. In other words, the scoured fabrics were softer than the unscoured fabric. This could be because the unscoured fabric contain impurities on the fiber surface and these stiffened the fabric. Once they were removed, the fibers were relaxed and easily bended, and thus the stiffness of the scoured polyester fabric decreased. The sodium carbonate scouring process produced softer polyester fabric than the lipase scouring process and this could be because the alkali scouring process could remove a slightly higher impurities from the fabric than the enzymatic scouring process (see Table 4.19).

Table 4.20 Stiffness of unscoured and scoured polyester fabrics.

Scouring Procedure	Bending length(cm.)		Stiffness(mg.cm)		% Change*	
	Warp	Weft	Warp	Weft	Warp	Weft
No scouring	2.79	2.54	454.03	354.29	0.00	0.00
With Na_2CO_3	2.27	1.88	290.86	169.63	-35.94	-52.12
With Lipase	2.46	1.92	386.12	180.23	-14.96	-49.13

* + = stiffness increased - = stiffness decreased

4.5.5 Dye Absorption and Whiteness of the Conventional and the Enzymatic Scoured Polyester Fabrics

Table 4.21 and 4.22 illustrate the fabric whiteness and the color strength of the dyed fabric respectively. The whiteness of polyester fabric slightly increased after scouring. Table 4.21 also shows that both scouring processes produced fabrics with similar whiteness. After scouring, the fabric was dyed with a disperse dye. The color strength of the dyed fabric shown in Table 4.22 indicates that both scouring processes were equally effective in terms of removing the impurities from the fabric, and thus produced fabrics with similar dyeability.

Table 4.21 Whiteness of unscoured and scoured polyester fabrics.

Scouring Procedure	Whiteness
No scouring	66.450
With Na_2CO_3	69.206
With Lipase	68.283

Table 4.22 Color strength of scoured polyester fabrics.

Scouring Procedure	K/S
With Na_2CO_3	16.717
With Lipase	17.480

4.5.6 Fiber Surface Morphology of the Conventional and the Enzymatic Scoured Polyester Fabrics

Figure 4.18 shows pictures of polyester fibers before and after scouring. Unscoured polyester fibers contained a lot of impurities on the surface (picture a). After the fabric was scoured using sodium carbonate, less impurities were left on the fiber surface. But when the fabric was scoured with lipase, almost all impurities were removed, the fiber looked clean and some fibers were peeled at the surface. This could be because before the fabric was scoured with lipase, it was prewashed in a solution containing a wetting agent and it was then scoured. Therefore the fiber surface was nearly freed from the impurities due to these two cleaning steps.

4.5.7 Conclusions of the Conventional and the Enzymatic Scouring of Polyester Fabrics.

To effectively scour the polyester fabric using lipase enzyme, the fabric should be prewashed in a solution containing a wetting agent before conducting the enzymatic scouring. Both conventional and enzymatic scouring processes could produce good quality scoured polyester fabric with an adequate absorbency, a very low weight loss, increases of strength, whiteness, softness, and a good dyeability.

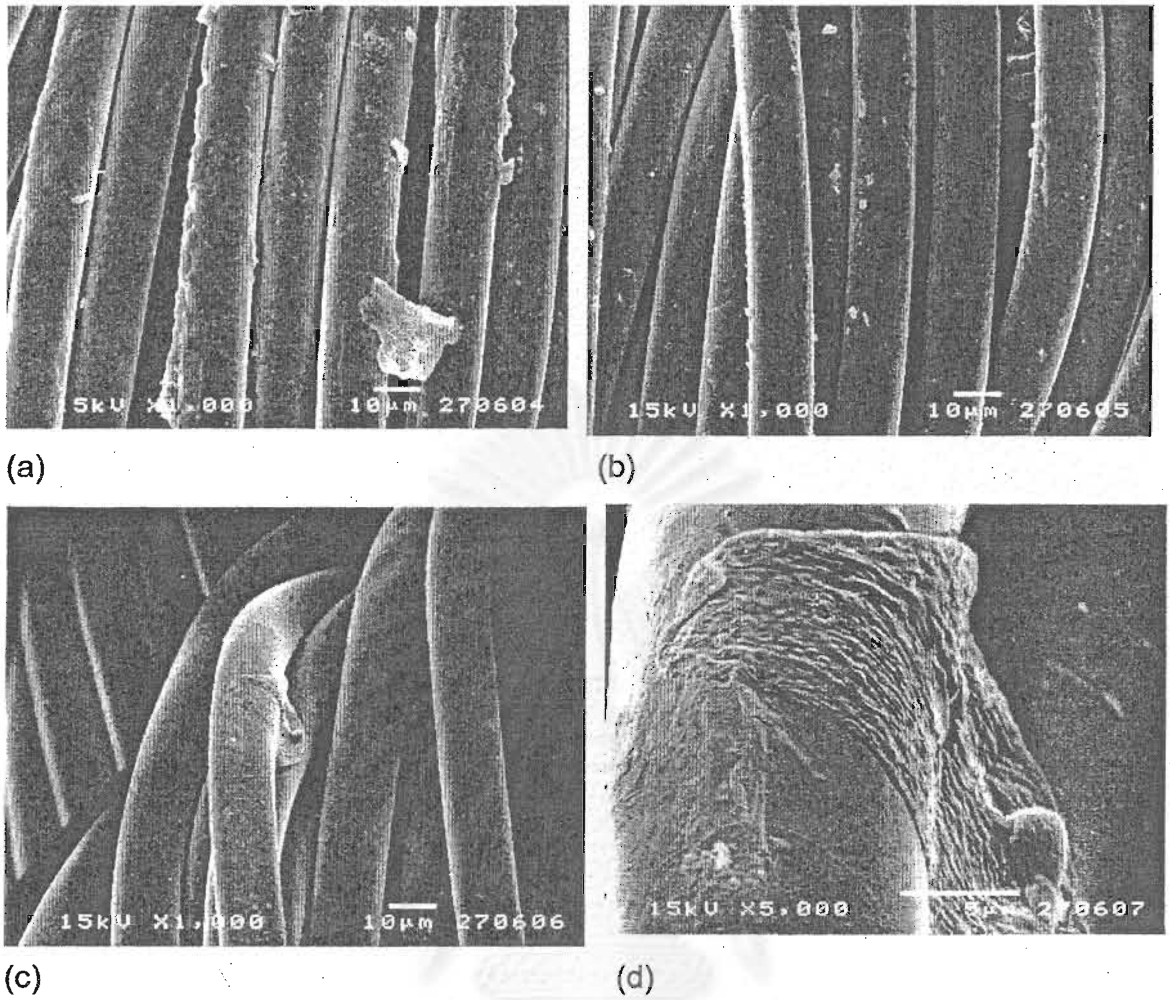


Figure 4.18 SEM micrograghs of greige (x 1,000) and scoured polyester fibers (x 1,000): (a) greige polyester, (b) sodium carbonate scoured polyester, (c) lipase scoured polyester, (d) lipase scoured polyester (x 5,000).

4.6 Scoured Nylon Fabric

Prewashed nylon fabric was scoured in a solution containing only a nonionic wetting agent 3 g/l and found that the fabric was able to absorb water adequately. Therefore it was not necessary to use other chemicals or enzymes in the scouring process except a wetting agent. Nylon fabric could contain very small amount of impurities and/or the impurities were easily removed using only a wetting agent.

4.6.1 Strength and Weight Loss of the Conventional Scoured Nylon Fabric

Scoured nylon fabric was tested for the fabric breaking load in order to determine for the fabric strength and it also was tested for the fabric weight loss, and the results are shown in Table 4.23.

Nylon fabric lost approximately 5% of the fabric weight after scouring. This means that nylon fabric could contain at least 5% impurities of the fabric weight but these impurities were easily removed in a solution of wetting agent.

Nylon fabric gained strength in warp direction and lost strength in weft direction after scouring.

Table 4.23 % Weight loss and breaking load of conventional scoured nylon fabric.

Scouring Procedure	Weight Loss (%)	Breaking Load(N)			
		Warp	% Change*	Weft	% Change*
No scouring	0.00	412.58	0.00	330.50	0.00
With wetting agent	5.09	444.08	7.09	316.83	-4.31

* + = breaking load increased - = breaking load decreased

4.6.2 Stiffness of the Conventional Scoured Nylon Fabric

Table 4.24 illustrates the fabric bending length and the fabric stiffness. It was found that after scouring, the fabric stiffness decreased both in warp and weft directions. The impurities could have been removed from the fiber surface and thus soften the fabric .

Table 4.24 Stiffness of conventional scoured nylon fabric.

Scouring Procedure	Bending length(cm.)		Stiffness (mg.cm)		% Change*	
	Warp	Weft	Warp	Weft	Warp	Weft
No scouring	3.43	2.91	258.72	156.94	0.00	0.00
With wetting agent	3.37	2.49	269.29	110.11	4.09	-29.84

* + = stiffness increased - = stiffness decreased

4.6.3 Dye Absorption and Whiteness of the Conventional Scoured Nylon Fabric

Scoured nylon fabric was tested for the fabric whiteness and the color strength and the results are shown in Tables 4.25 and 4.26.

Table 4.25 shows whiteness of the unscoured and the scoured nylon fabrics. It was found that nylon fabric slightly gained whiteness after scouring. Nylon fabric already contained high whiteness at 71.6 before scouring and once it was scoured, the fabric whiteness slightly increased to 74.7 due to the removal of the impurities from the fiber surface. Table 4.26 shows the color strength of the dyed nylon fabric at 15.6.

Table 4.25 Whiteness of conventional scoured nylon fabric.

Scouring Procedure	Whiteness
No scouring	71.576
With wetting agent	74.738

Table 4.26 Color strength of conventional scoured nylon fabric.

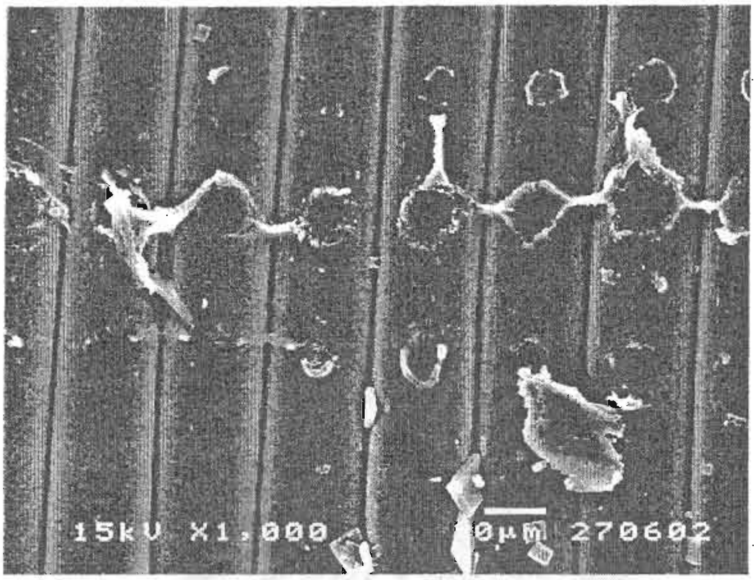
Scouring Procedure	K/S
With wetting agent	15.635

4.6.4 Fiber Surface Morphology of the Conventional Scoured Nylon Fabric

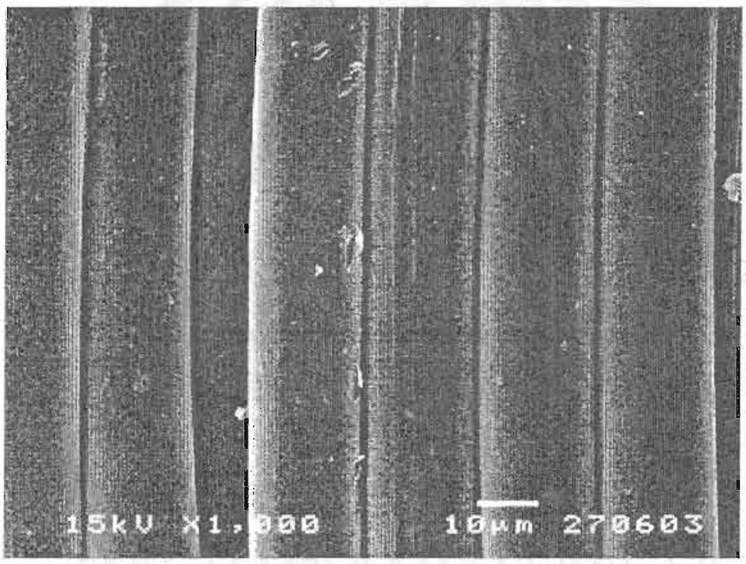
Pictures (a) and (b) in Figure 4.19 show the appearance of the nylon fiber surface before and after scouring, respectively. Unscoured nylon fibers contained a lot of impurities on the fiber surface while the scoured fibers looked clean on the surface. There was no fiber damage shown in both pictures.

4.6.5 Conclusions of the Conventional Scouring of Nylon Fabric

Nylon fabric was easily scoured in a solution of wetting agent. The scoured nylon fabric had an adequate absorbency, a loss of 5% fabric weight, an increase of whiteness, and a soft touch.



(a)



(b)

Figure 4.19 SEM micrographs of greige (x 1,000) and scoured nylon fibers (x 1,000): (a) greige nylon, (b) nonionic wetting agent scoured nylon.

CHAPTER 5

Conclusions

From the results and discussion shown in Chapter 4, some conclusions can be drawn as follows.

1. In this experiment, various enzymatic scouring processes using lipase, protease, and cellulase enzymes were conducted on various fabrics and found that the scouring results were very impressive and were comparable to the conventional scouring results. Scoured fabrics were clean and absorbed water instantaneously. Although they lost 0.2-1.0% of the fabric weight after scouring, they gained strength and whiteness. In addition, this scouring process made the fabric soften as well.
2. Lipase, protease and cellulase were less effective for scouring cotton or cotton/polyester blends when each enzyme was used alone. To successfully scour these fibers, two scouring steps were needed by scouring with lipase, protease, or lipase and protease in the first step and scouring with cellulase in the second step.
3. Results from the enzymatic scouring of T/C and polyester fabrics indicate that the synthetic oils coated on the fiber surface hindered the enzyme activity and thus needed to be removed before conducting an enzymatic scouring on these fabrics.
4. Lipase could successfully scour the polyester fabric and acquire desirable fabric properties.
5. Impurities on the nylon fabric could be easily removed by scouring in a solution of wetting agent.
6. In every enzymatic scouring step, it was necessary to add a nonionic wetting agent in the system.

CHAPTER 6

Recommendation

1. It would be interested to study for the effectiveness of an enzymatic scouring of other cellulosic fibers such as rayon and lyocell.

2. The effectiveness of the enzymatic scouring can also be evaluated from the amount of the soluble/insoluble materials removed from the fabric, in the remaining solution.

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Appendix

Appendix

Table A1 Weight of each various fabric(g/100 cm^2).

Fabric	Weight (g/100 cm^2)			Mean (g/100 cm^2)
Cotton	1.1613	1.1615	1.1613	**1.1614**
CVC	1.9405	1.941	1.9396	**1.9404**
TC	1.7831	1.782	1.7824	**1.7825**
Polyester	2.0804	2.0804	2.0815	**2.0808**
Nylon	0.5972	0.5965	0.5963	**0.5967**

Table A2 Extractable materials in cotton knit fabric: hot water, and solvent extractions.

	Trial1		Trial2		Trial3		Mean
	g	%	g	%	g	%	%
Greige fabric	2.540		2.561		2.603		
Hot water ext. fabric	2.474		2.498		2.543		
Sol.ext. fabric	2.466		2.490		2.535		
Water soluble		2.60		2.46		2.31	**2.45**
Solvent extractable		0.32		0.32		0.31	**0.32**
Total ext. materials		2.92		2.78		2.62	**2.77**

Table A3 Extractable materials in CVC knit fabric: hot water, and solvent extractions.

	Trial1		Trial2		Trial3		Mean
	g	%	g	%	g	%	%
Greige fabric	4.314		4.444		4.59		
Hot water ext. fabric	4.252		4.381		4.525		
Sol.ext. fabric	4.242		4.371		4.517		
Water soluble		1.44		1.42		1.42	**1.42**
Solvent extractable		0.24		0.23		0.18	**0.21**
Total ext. materials		1.67		1.65		1.59	**1.64**

Table A4 Extractable materials in T/C knit fabric: hot water, and solvent extractions.

	Trial1		Trial2		Trial3		Mean
	g	%	g	%	g	%	%
Greige fabric	3.960		4.092		4.048		
Hot water ext. fabric	3.923		4.055		4.003		
Sol.ext. fabric	3.914		4.049		3.999		
Water soluble		0.93		0.90		1.11	**0.98**
Solvent extractable		0.23		0.15		0.10	**0.16**
Total ext. materials		1.16		1.05		1.21	**1.14**

Table A5 Weight loss (%) of prewashing and conventional scoured knit cotton fabrics.

	Conventional Process			Mean
	1	2	3	
Greige	1.616	1.611	1.611	
Prewashing	1.573	1.573	1.575	
Scoured	1.555	1.556	1.56	
%Weight loss after prewashing	-2.73	-2.42	-2.29	-2.48
%Weight loss after scouring	-1.16	-1.09	-0.96	-1.07

Table A6 Weight loss (%) of prewashing and lipase/cellulase scoured knit cotton fabrics.

	Enzymatic Process			Mean
	1	2	3	
Greige	1.557	1.555	1.554	
Prewashing	1.522	1.520	1.522	
Scoured	1.507	1.509	1.508	
%Weight loss after prewashing	-2.30	-2.30	-2.10	-2.23
%Weight loss after scouring	-1.00	-0.73	-0.93	-0.88

Table A7 Weight loss (%) of prewashing and protease/cellulase scoured knit cotton fabrics.

	Enzymatic Process			Mean
	1	2	3	
Greige	1.598	1.594	1.593	
Prewashing	1.560	1.556	1.558	
Scoured	1.544	1.546	1.546	
%Weight loss after prewashing	-2.44	-2.44	-2.25	**-2.37**
%Weight loss after scouring	-1.04	-0.65	-0.78	**-0.82**

Table A8 Weight loss (%) of prewashing and lipase+protease/cellulase scoured knit cotton fabrics.

	Enzymatic Process			Mean
	1	2	3	
Greige	1.554	1.568	1.567	
Prewashing	1.538	1.534	1.535	
Scoured	1.526	1.525	1.521	
%Weight loss after prewashing	-1.04	-2.22	-2.08	**-1.78**
%Weight loss after scouring	-0.79	-0.59	-0.92	**-0.77**

Table A9 Weight loss (%) of prewashing and conventional scoured knit CVC fabrics.

	Conventional Process			Mean
	1	2	3	
Greige	3.019	3.011	3.011	
Prewashing	2.972	2.970	2.970	
Scoured	2.956	2.957	2.956	
%Weight loss after prewashing	-1.58	-1.38	-1.38	**-1.45**
%Weight loss after scouring	-0.54	-0.44	-0.47	**-0.48**

Table A10 Weight loss (%) of prewashing and lipase/cellulase scoured knit CVC fabrics.

	Enzymatic process			Mean
	1	2	3	
Greige	3.003	2.997	2.996	
Prewashing	2.959	2.955	2.953	
Scoured	2.943	2.940	2.940	
%Weight loss after prewashing	-1.49	-1.42	-1.46	**-1.45**
%Weight loss after scouring	-0.54	-0.51	-0.44	**-0.50**

Table A11 Weight loss (%) of prewashing and protease/cellulase scoured knit CVC fabrics.

	Enzymatic process			Mean
	1	2	3	
Greige	2.918	2.912	2.911	
Prewashing	2.873	2.873	2.871	
Scoured	2.860	2.861	2.861	
%Weight loss after prewashing	-1.57	-1.36	-1.39	**-1.44**
%Weight loss after scouring	-0.45	-0.42	-0.35	**-0.41**

Table A12 Weight loss (%) of prewashing and lipase+protease/cellulase scoured knit CVC fabrics.

	Enzymatic process			Mean
	1	2	3	
Greige	2.878	2.874	2.874	
Prewashing	2.830	2.833	2.831	
Scoured	2.821	2.820	2.821	
%Weight loss after prewashing	-1.70	-1.45	-1.52	**-1.55**
%Weight loss after scouring	-0.32	-0.46	-0.35	**-0.38**

Table A13 Weight loss (%) of prewashing and cellulase scoured knit CVC fabrics.

	Enzymatic process			Mean
	1	2	3	
Greige	2.956	2.952	2.952	
Prewashing	2.914	2.912	2.911	
Scoured	2.897	2.896	2.896	
%Weight loss after prewashing	-1.44	-1.37	-1.41	-1.41
%Weight loss after scouring	-0.59	-0.55	-0.52	-0.55

Table A14 Weight loss (%) of prewashing and conventional scoured knit T/C fabrics.

	Conventional process			Mean
	1	2	3	
Greige	2.531	2.529	2.529	
Prewashing	2.501	2.504	2.505	
Scoured	2.494	2.494	2.495	
%Weight loss after prewashing	-1.20	-1.00	-0.96	-1.05
%Weight loss after scouring	-0.28	-0.40	-0.40	-0.36

Table A15 Weight loss (%) of prewashing and lipase scoured knit T/C fabrics.

	Enzymatic process			Mean
	1	2	3	
Greige	2.622	2.620	2.620	
Prewashing	2.589	2.589	2.589	
Scoured	2.578	2.575	2.574	
%Weight loss after prewashing	-1.27	-1.20	-1.20	-1.22
%Weight loss after scouring	-0.43	-0.54	-0.58	-0.52

Table A16 Weight loss (%) of prewashing and protease scoured knit T/C fabrics.

	Enzymatic process			Mean
	1	2	3	
Greige	2.530	2.529	2.528	
Prewashing	2.498	2.500	2.496	
Scoured	2.484	2.482	2.481	
%Weight loss after prewashing	-1.28	-1.16	-1.28	-1.24
%Weight loss after scouring	-0.56	-0.73	-0.60	-0.63

Table A17 Weight loss(%) of prewashing and lipase+protease scoured knit T/C fabrics.

	Enzymatic process			**Mean**
	1	**2**	**3**	
Greige	2.552	2.551	2.550	
Prewashing	2.523	2.521	2.520	
Scoured	2.508	2.505	2.505	
%Weight loss after prewashing	-1.15	-1.19	-1.19	**-1.18**
%Weight loss after scouring	-0.60	-0.64	-0.60	**-0.61**

Table A18 Weight loss(%) of prewashing and conventional scoured woven polyester fabrics.

	Conventional process			**Mean**
	1	2	3	
Greige	3.146	3.143	3.141	
Prewashing	3.104	3.104	3.104	
Scoured	3.094	3.094	3.094	
%Weight loss after prewashing	-1.35	-1.26	-1.19	**-1.27**
%Weight loss after scouring	-0.32	-0.32	-0.32	**-0.32**

Table A19 Weight loss(%) of prewashing and lipase scoured woven polyester fabrics.

	Enzymatic process			Mean
	1	2	3	
Greige	3.101	3.085	3.098	
Prewashing	3.072	3.07	3.07	
Scoured	3.063	3.063	3.064	
%Weight loss after prewashing	-0.94	-0.49	-0.91	**-0.78**
%Weight loss after scouring	-0.29	-0.23	-0.20	**-0.24**

Table A20 Weight loss(%) of prewashing and conventional scoured woven nylon fabrics.

	Conventional process			Mean
	1	2	3	
Greige	1.375	1.375	1.375	
Scoured	1.302	1.305	1.309	
%Weight loss after scouring	-5.61	-5.36	-5.04	**-5.34**

Table A21 Bursting strength (kg/cm^2) of greige, scoured knitted cotton fabric.

Cotton	Bursting strength of each formular fabric (kg/cm^2)				
Trial No.	Greige	NaOH	Llipase/ cellulase	Protease/ cellulase	Lipase+Protease/ cellulase
1	6.80	5.50	6.40	7.10	6.60
2	5.20	6.30	6.30	6.30	6.30
3	6.30	6.00	6.70	6.60	6.30
4	6.20	6.40	5.80	6.10	5.90
5	5.80	6.50	6.00	5.70	6.70
6	6.20	6.20	6.20	6.60	6.10
7	5.70	6.40	6.40	6.10	6.90
8	6.10	6.50	5.50	6.60	5.70
9	5.80	7.10	5.80	6.30	6.60
10	6.20	5.70	6.40	6.20	6.30
Mean	6.03	6.26	6.15	6.36	6.34

Table A22 Bursting strength (kg/cm^2) of greige, scoured knitted CVC fabric.

CVC	Bursting strength of each formular fabric (kg/cm2)					
Trial No.	Grige	NaOH	Lipase/ cellulase	Protease/ cellulase	Lipase +protease/ cellulase	Cellulase
1	11.10	11.00	11.20	10.60	11.10	9.80
2	10.60	11.00	9.90	10.90	11.50	11.10
3	10.40	11.10	11.50	11.00	9.70	10.00
4	10.80	10.50	10.60	10.30	11.20	10.30
5	10.30	10.90	11.50	9.60	11.50	11.30
6	10.20	10.90	10.50	11.00	10.50	11.40
7	11.50	11.10	11.20	11.80	11.00	10.90
8	11.00	10.90	11.10	11.80	9.80	10.20
9	10.60	11.10	10.40	10.50	10.50	10.80
10	10.40	10.60	10.20	10.80	10.20	10.50
Mean	**10.69**	**10.91**	**10.81**	**10.83**	**10.70**	**10.63**

Table A23 Bursting strength (kg/cm2) of greige, scoured knitted T/C fabric.

T/C	Bursting strength of each formular fabric (kg/cm^2)				
Trial No.	Greige	NaOH	Lipase/ cellulase	Protease/ cellulase	Lipase+Protease/ cellulase
1	11.2	11.8	11.6	11.4	11.8
2	11.2	11.5	11.7	11.5	11.2
3	11.9	11.5	11.7	11.5	11.5
4	10.8	11.6	11.4	10.9	11.5
5	11.2	11.8	11.4	11.1	11.7
6	12	11.7	11.1	11.7	11.6
7	11.4	11.3	11.6	11	11
8	10.7	11.2	11.2	11.8	11.7
9	11.5	11.7	11.4	11.2	11.2
10	11.2	12.1	11.5	11.5	12.3
Mean	**11.31**	**11.62**	**11.46**	**11.36**	**11.55**

Table A24 Breaking loads (N) in warp direction of greige, scoured woven polyester fabrics.

Trial No.	Greige	Scoured fabric	
		Conventional	Enzymatic
1	681.9	624.2	853.9
2	680.2	658.5	885.9
3	662.7	712.9	760.5
4	716.7	722.3	803.7
5	699.9	718	772.2
Mean	**688.3**	**687.2**	**815.2**

Table A25 Breaking loads (N) in weft direction of greige, scoured woven polyester fabrics.

Trial No.	Greige	Scoured fabric	
		Conventional	Enzymatic
1	293.8	317.4	372.4
2	286.3	336.4	352.1
3	309.4	332.3	353.3
4	324.2	350.2	319.7
5	318.6	332.7	368.8
6	314.0	365.4	417.0
7	323.8	302.6	414.2
8	316.8	331.3	303.1
Mean	**310.9**	**333.5**	**362.6**

Table A26 Breaking loads (N) in warp direction of greige, scoured woven nylon fabrics.

Trial No.	Greige	Conventional scoured fabric
1	433.5	475.0
2	399.8	462.3
3	411.5	430.3
4	402.5	426.8
5	415.6	426.0
Mean	**412.6**	**444.1**

Table A27 Breaking loads (N) in weft direction of greige, scoured woven nylon fabrics.

Trial No.	Greige	Conventional scoured fabric
1	343.9	346.0
2	325.0	299.3
3	319.1	304.0
4	322.5	283.7
5	329.0	294.2
6	331.1	372.1
7	333.3	268.5
8	340.1	366.8
Mean	**330.5**	**316.8**

Table A28 Stiffness (mg.cm) in warp of greige polyester fabrics.

Trail No.	Weight (g)	Bending length (cm)				Stiffness
1	1.0523	3.00	2.75	2.70	2.95	487.20
2	1.0172	2.83	2.85	2.75	2.60	427.70
3	1.0664	2.80	2.70	2.70	2.80	443.56
Mean						452.82

Table A29 Stiffness(mg.cm) in weft of greige polyester fabrics.

Trail No.	Weight (g)	Bending length (cm)				Stiffness
1	1.0757	2.75	2.3	2.4	2.7	351.51
2	1.0862	2.4	2.4	2.27	2.35	283.73
3	1.0804	2.97	2.55	2.8	2.6	439.65
Mean						358.30

Table A30 Stiffness(mg.cm) in warp of conventional scoured polyester fabrics.

Trail No.	Weight (g)	Bending length (cm)				Stiffness
1	1.2613	2.25	2.15	2.43	2.50	320.12
2	1.2270	2.25	2.10	2.45	2.30	288.95
3	1.2416	2.25	2.15	2.25	2.20	268.94
Mean						292.67

Table A31 Stiffness(mg.cm) in weft of conventional scoured polyester fabrics.

Trail No.	Weight (g)	Bending length (cm)				Stiffness
1	1.2513	1.73	1.90	1.60	1.95	144.74
2	1.3001	1.85	1.85	2.15	1.87	186.93
3	1.2778	1.95	1.85	1.95	1.95	182.30
Mean						171.32

Table A32 Stiffness(mg.cm) in warp of enzymatic scoured polyester fabrics.

Trail No.	Weight (g)	Bending length (cm)				Stiffness
1	1.2716	2.1	2.6	2.6	2.43	366.05
2	1.3117	2.7	2.4	2.15	2.6	391.74
3	1.3073	2.73	2.3	2.45	2.47	402.43
Mean						386.74

Table A33 Stiffness(mg.cm) in weft of enzymatic scoured polyester fabrics.

Trail No.	Weight (g)	Bending length (cm)				Stiffness
1	1.2859	1.70	1.97	2.10	1.80	174.32
2	1.2729	1.85	1.95	1.80	2.15	185.16
3	1.2608	1.85	1.90	2.00	2.00	183.40
Mean						180.96

Table A34 Stiffness(mg.cm) in warp of greige nylon fabrics.

Trail No.	Weight (g)	Bending length (cm)				Stiffness
1	0.3172	3.20	3.20	3.70	3.60	254.89
2	0.3216	3.20	3.20	3.65	3.75	264.12
3	0.3229	3.15	3.23	3.65	3.60	255.51
Mean						258.17

Table A35 Stiffness(mg.cm) in weft of greige nylon fabrics.

Trail No.	Weight (g)	Bending length (cm)				Stiffness
1	0.2995	3.1	3.2	3.6	3.6	230.28
2	0.3312	2.65	2.6	2.73	2.73	127.15
3	0.3246	2.65	2.6	2.73	2.73	124.61
Mean						160.68

Table A36 Stiffness(mg.cm) in warp of conventional scoured nylon fabrics.

Trail No.	Weight (g)	Bending length (cm)				Stiffness
1	0.354	2.95	3.5	3.6	3.8	293.9
2	0.3509	3.45	3.37	3.3	3.1	253.35
3	0.3505	3.33	3.1	3.57	3.35	260.6
Mean						269.28

Table A37 Stiffness(mg.cm) in weft of conventional scoured nylon fabrics.

Trail No.	Weight (g)	Bending length (cm)				Stiffness
1	0.3557	2.75	2.33	2.43	2.83	122.88
2	0.3554	2.33	2.45	2.5	2.45	102.31
3	0.3587	2.6	2.45	2.43	2.37	107.12
Mean						110.77

Table A38 Adsorption of methylene blue (MB) on fabrics.

Weight (g)	L:R(30:1)	Absorbence	Y=177.87X	Dilute soln (40 times)	MB on Sub.	MB (g/l)	MB (g/kg)
greige cotton							
1.3141	39.4	0.65	0.00365	0.1462	0.3538	0.0139	10.61
1.2695	38.1	0.659	0.00370	0.1482	0.3518	0.0134	10.55
			Mean				10.58
NaOH							
1.6421	49.3	1.09	0.00613	0.2451	0.2549	0.0126	7.65
1.5952	47.9	1.058	0.00595	0.2379	0.2621	0.0125	7.86
			Mean				7.75
Lipase/cellulase							
1.5939	47.8	0.978	0.00550	0.2199	0.2801	0.0134	8.40
1.5507	46.5	0.976	0.00549	0.2195	0.2805	0.0130	8.42
			Mean				8.41
protease/cellulase							
1.6483	49.4	0.966	0.00543	0.2172	0.2828	0.0140	8.48
1.7621	52.9	0.976	0.00549	0.2195	0.2805	0.0148	8.42
			Mean				8.45
lipase+protease/cellulase							
1.6069	48.2	1.038	0.00584	0.2334	0.2666	0.0129	8.00
1.7011	51.0	1.03	0.00579	0.2316	0.2684	0.0137	8.05
			Mean				8.02
greige CVC							
2.2880	68.6	0.929	0.00522	0.2089	0.2911	0.0200	8.73
2.2968	68.9	0.92	0.00517	0.2069	0.2931	0.0202	8.79
			Mean				8.76

Table A38 (continued)

Weight (g)	L:R(30:1)	Absorbence	Y=177.87X	Dilute soln (40 times)	MB on Sub.	MB (g/l)	MB (g/kg)
NaOH							
2.5914	77.7	1.284	0.00722	0.2888	0.2112	0.0164	6.34
2.6572	79.7	1.295	0.00728	0.2912	0.2088	0.0166	6.26
Mean							**6.30**
Lipase/cellulase							
2.7023	81.1	1.298	0.00730	0.2919	0.2081	0.0169	6.24
2.5698	77.1	1.286	0.00723	0.2892	0.2108	0.0163	6.32
2.6418	79.3	1.315	0.00739	0.2957	0.2043	0.0162	6.13
Mean							**6.23**
Protease/cellulase							
2.4869	74.6	1.277	0.00718	0.2872	0.2128	0.0159	6.38
2.5991	78.0	1.283	0.00721	0.2885	0.2115	0.0165	6.34
Mean							**6.36**
Lipase+Protease/cellulase							
2.5447	76.3	1.227	0.00690	0.2759	0.2241	0.0171	6.72
2.5647	76.9	1.242	0.00698	0.2793	0.2207	0.0170	6.62
Mean							**6.67**
Cellulase							
2.5851	77.6	1.364	0.00767	0.3067	0.1933	0.0150	5.80
2.6094	78.3	1.365	0.00767	0.3070	0.1930	0.0151	5.79
Mean							**5.79**

Table A38 (continued)

Weight (g)	L:R(30:1)	Absorbence	Y=177.87X	Dilute soln (40 times)	MB on Sub.	MB (g/l)	MB (g/kg)
greige TC							
2.0458	61.4	1.25	0.00703	0.2811	0.2189	0.0134	6.57
2.0701	62.1	1.241	0.00698	0.2791	0.2209	0.0137	6.63
			Mean				6.60
NaOH							
2.7020	81.1	1.397	0.00785	0.3142	0.1858	0.0151	5.58
2.6435	79.3	1.382	0.00777	0.3108	0.1892	0.0150	5.68
						Mean	5.63
Lipase/cellulase							
2.6651	80.0	1.464	0.00823	0.3292	0.1708	0.0137	5.12
2.6213	78.6	1.468	0.00825	0.3301	0.1699	0.0134	5.10
			Mean				5.11
Protease/cellulase							
2.4560	73.7	1.534	0.00862	0.3450	0.1550	0.0114	4.65
2.5090	75.3	1.538	0.00865	0.3459	0.1541	0.0116	4.62
			Mean				4.64
Lipase+Protease/cellulase							
2.4635	73.9	1.451	0.00816	0.3263	0.1737	0.0128	5.21
2.4802	74.4	1.463	0.00823	0.3290	0.1710	0.0127	5.13
			Mean				5.17

Table A39 Whiteness index of greige, scoured cotton knit fabrics.

cotton	Whiteness index				
Trial No.	Greige	NaOH	Lipase/ cellulase	Protease/ cellulase	Li+Pro/ cellulase
1	-5.869	23.846	17.578	18.278	17.971
2	-5.352	21.814	16.866	16.965	17.174
3	-6.989	24.207	16.834	18.016	18.152
4	-6.321	22.875	16.765	18.831	17.703
5	-6.191	24.144	18.177	17.886	18.25
6	-8.391	23.247	18.138	18.353	16.985
7	-5.306	22.418	17.332	17.959	17.611
8	-5.289	23.667	17.173	18.369	17.735
9	-5.423	20.668	17.059	18.107	17.823
10	-4.844	20.231	17.431	18.362	17.258
Mean	-5.998	22.712	17.335	18.112	17.666

Table A40 Whiteness index of greige, scoured CVC knit fabrics.

CVC	Whiteness index					
Trial No.	Greige	NaOH	Lipase/ cellulase	Protease/ cellulase	Li+Pro/ cellulase	Celluase
1	2.497	36.111	25.777	28.714	27.193	26.059
2	1.889	38.048	28.554	29.192	27.247	27.178
3	1.926	37.935	26.67	27.898	28.55	26.605
4	0.316	35.222	26.908	28.944	26.652	26.601
5	1.200	35.918	27.486	28.071	26.555	26.635
6	-0.105	34.321	28.212	28.041	25.341	28.060
7	0.075	35.436	27.081	27.422	24.961	27.160
8	0.713	33.875	26.102	27.794	25.594	26.704
9	0.979	35.415	27.111	27.368	25.446	27.593
10	0.660	34.695	25.718	28.625	26.38	27.433
Mean	**1.015**	**35.698**	**26.962**	**28.207**	**26.392**	**27.003**

Table A41 Whiteness index of greige, scoured T/C knit fabrics.

T/C	Whiteness index				
Trial No.	Greige	NaOH	Lipase/ cellulase	Protease/ cellulase	Li+Pro/ cellulase
1	20.684	38.444	39.740	38.723	40.099
2	20.274	37.475	40.138	39.283	39.496
3	20.621	37.828	39.414	38.295	39.710
4	19.955	38.004	39.135	38.969	40.799
5	19.813	38.273	38.651	36.114	40.071
6	19.580	39.283	36.941	38.821	39.975
7	19.681	39.238	39.040	38.859	40.952
8	19.092	38.611	39.086	34.981	37.891
9	19.669	38.093	39.686	38.267	40.028
10	20.298	38.350	38.999	37.481	40.066
Mean	19.967	38.360	39.083	37.979	39.909

Table A42 Whiteness index of greige, scoured polyester fabrics.

Trial No.	Whiteness index		
	Greige	Na_2CO_3	Lipase
1	66.883	68.259	68.625
2	66.753	69.266	68.062
3	66.801	69.117	68.458
4	66.007	69.429	68.160
5	66.131	69.680	68.483
6	66.280	69.330	67.990
7	66.294	69.360	68.206
Mean	**66.450**	**69.206**	**68.283**

Table A43 Whiteness index of greige, scoured nylon fabrics.

Trial No.	Whiteness index	
	Greige	Wetting agent
1	71.353	74.380
2	71.488	74.144
3	71.427	74.264
4	71.785	74.502
5	71.544	75.610
6	71.861	75.528
Mean	**71.576**	**74.738**

BIOGRAPHY

Mr. Puwadol Kitchareonseree was born in Bangkok, Thailand, on January 26, 1977. He received a Bachelor of Science degree with a major in Petrochemicals and Polymeric Materials from Silpakorn University in 2000. He started as a graduate student in Department of Materials Science with a major in Applied Polymer Science and Textile Technology, Chulalongkorn University in June 2000, and completed the programme in October 2002.