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APPLICATION OF OZONE SOLUTION FOR TEXTILE WASHING

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โชติกา ชะโลธร: การประยุกต์ใช้สารละลายโอโซนสำหรับการซักล้างสิ่งทอ (APPLICATION OF OZONE SOLUTION FOR TEXTILE WASHING). อ. ที่ปรึกษาวิทยานิพนธ์หลัก : รศ. ดร.ธวัชชัย ชรินพาณิชกุล, 71 หน้า

อุตสาหกรรมสิ่งทอเป็นอุตสาหกรรมหนึ่งที่สำคัญ สิ่งสำคัญสิ่งหนึ่งของอุตสาหกรรมนี้ คือวิธีการทำความสะอาดสิ่งทอ โดยที่คุณภาพของสิ่งทอยังคงเดิม ในการวิจัยนี้เป็นการประยุกต์นำสารละลายโอโซนเพื่อกำจัดคราบน้ำมันออกจากผ้าฝ้าย 100 %โดยใช้กรดลิโนลิกเป็นตัวแทนของน้ำมัน โดยสารละลายโอโซนถูกเตรียมโดยวิธีอิเล็คโตไลซิส ที่อุณหภูมิห้อง โดยศึกษาผลของความเข้มข้นโอโซนในช่วง 0.5 ถึง 2.5 มิลลิกรัมต่อลิตร และเวลาในการทำปฏิกิริยา ตั้งแต่ 5 ถึง 20 นาที ที่มีต่อคุณสมบัติของสิ่งทอที่ถูกทำความสะอาดด้วยโอโซน

จากผลการทดลอง การกำจัดคราบกรดลิโนลิกขึ้นกับค่าความเป็นกรดด่างของสารละลาย และการเกิดออกซิเดชั่นที่เร็วนั้น(0.0023 วินาที$^{-1}$) เกิดขึ้นเมื่อสารละลายอยู่ในสถาวะเป็นเบส ($pH \geq 9$) การเพิ่มความเข้มข้นของสารละลายโอโซน และเวลาที่ใช้ในการซัก สามารถกำจัดคราบน้ำมันไปได้ดียิ่งขึ้น ความขาวของผ้าเมื่อซักด้วยโอโซนที่ความเข้มข้น 2.5 มิลลิกร้มต่อลิตร เพิ่มขึ้นร้อยละ 6.3 แต่ทำให้ความแข็งแรงของผ้าฝ้ายลดลงเมื่อเทียบกลับผ้าฝ้ายก่อนทำการซัก เมื่อทำการซักผ้าซ้ำ 30 ครั้ง หลังการซักพบว่า ความขาวของผ้ายังคงเท่าเดิม และความความแข็งแรงของผ้าลดลงเพียงร้อยละ 7.29 เมื่อเทียบกับการซักผ้าแบบดังเดิม ข้อดีของระบบการซักผ้าด้วยสารละลายโอโซนนั้น คือช่วยประหยัดสารเคมี ยืดอายุการใช้งานของผ้าฝ้ายได้ดีขึ้นเมื่อเทียบกับระบบแบบเดิม

ภาควิชา วิศวกรรมเคมี ลายมือชื่อนิสิต.....

สาขาวิชา วิศวกรรมเคมี ลายมือชื่ออ.ที่ปรึกษาวิทยานิพนธ์หลัก.....

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CHOTIKA CHALOTORN: APPLICATION OF OZONE SOLUTION FOR TEXTILE WASHING. THESIS ADVISOR: ASSOC. PROF. TAWATCHAI CHARINPANITKUL, D.Eng., 71 pp.

Textile manufacturing is one of the most important industries. One important issue in this industry is how to clean fabric by maintaining its quality. In this work, the application of ozone solution for removing linoleic acid as a simulated strain from 100% cotton fabric was experimentally investigated. The ozone solution was prepared by electrolysis technique at room temperature. The effects of ozone concentration in the range of 0.5 to 2.5 ppm and washing time of 5 to 20 min on the properties of ozone treated fabric samples was studied.

From the experimental results, the removal of linoleic acid depended on pH of the ozone solution. The fastest oxidation rate constant of 0.0023 sec^{-1} could be achieved under the basic condition ($pH \geq 9$). The increasing ozone concentration and washing time could provide appreciable removal of the simulated stain. Whiteness index of the fabric treated with 2.5 ppm ozone solution increased 6.3 % comparing with the untreated one but the strength of fabric was decreased comparing with original fabric. After 30 washing cycles, the strength of the ozone washed fabric decreased only 7.29% in comparison with its original value but its whiteness index was still at the same level as that of the original one. The advantages of the ozone washing system were saving in the chemicals and extending lift time of the cotton fabric, when compared with the conventional washing system.

Department: …..Chemical Engineering…..Student's Signature…….……...

Field of Study:...Chemical Engineering…..Advisor's Signature……

Academic Year:……2008……...

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NOMENCLATURES

A_0	Absorption time prior to staining
A_w	Absorption time after stained with oil
A_p	Absorption time after washing
FTIR	Fourier Transform Infrared Spectroscopy
D	Diffusivity of ozone
J	Rate of mass transfer of ozone solution
k’	Pseudo first-order rate constant of linoleic acid
SEM	Scanning Electron Microscope
TOC	Total organic carbon
$[O_3]$	Ozone concentration
[LA]	Linoleic acid concentration

CHAPTER I

INTRODUCTION

1.1 Background

Nowadays, Industry in Thailand has been expanded continuously. Textile industry is one of the most important industries in Thailand. One important issue in this industry is to clean textile by maintaining its quality. Additionally, in commercial laundry by various end-users, such as restaurants, hotels and hospital, a large quantity of fabric must be carefully cleaned. Although use of commercial laundry systems in larger washing is now available, it is necessary to use hot water in laundry process for killing various bacteria and virus. But current wet textile cleanings suffer from two major drawbacks. There is large process time and low energy efficiency. Therefore, there are requirements of developing new alternative technologies.

However, it is possible that hot water could damage the fabric [1]. Therefore, there are requirements of developing new alternative washing technologies. Fabric is cleaned by many alternative technologies such as liquid carbon dioxide cleaning, ultrasonic washing and cold water with ozone solution washing [2-4]. The methods of washing depend on the type of stain and characteristics of textile.

Ozone has been recognized as a powerful oxidizing agent. It could react with organic and inorganic molecules. In general, fabrics would be contaminated with many kinds of strains, such as vegetable oils, which are found in cooking activities and hard to clean up. These issues lead to requirement of detergent applications [5].

Meanwhile, it should be noted that vegetable oils, which are coconut oil, soybean oil and safflower oil, are normally composed of linoleic acid which is polyunsaturated fatty acid [6]. To remove such contamination would strongly rely on application of sufficient use of detergent and mechanical washing effort.

In general, fabrics would be contaminated with many kinds of stains, such as vegetable oils, which are found in cooking activities and hard to clean up. It could affect environment if its concentration is higher than 0.1 mg/L [1].

To the best of our knowledge, there are only few investigations reporting on use of ozone in laundry process for effective removal of organic strain. In this work, the effects of experimental parameters which are ozone concentration and washing time on the effectiveness of fabric cleaning would be examined. The optimum condition by ozone cleaning regarding to stain removal performance without providing damage on the fabric would be the focal issue of this work. The results were compared with the conventional hot-water cleaning methods.

1.2 Objective of the study

The main objective of this research is to investigate suitable means for application of ozone solution to clean textile surface. Determination of an optimal concentration of ozone and washing time to clean textile without providing damage will also be set as an issue of study in this work.

1.3 Scopes of the study

1.3.1 Study on ozone concentration in 0.5, 1.0, 1.5, 2.0 and 2.5 ppm

1.3.2 Study on washing time in 5, 10, 15 and 20 min.

1.3.3 Stain to be used in this work is linoleic acid.

1.3.4 Analysis of properties textile:

1.3.4.1Whiteness index of textile using CCM. (Computer color matching)

1.3.4.2 Appearance of textile using microscopic analysis and SEM (Scanning electron microscope)

1.3.4.3 Weight loss (%) of stains.

1.3.4.4 Tensile strength of textile.

1.3.4.5 FT-IR (Fourier transform infrared spectroscopy)

1.3.4.6 UV-visible spectrophotometer

1.3.4.7 TOC (Total organic carbon analyzer)

1.4 Procedures of the research

1.4.1 Conducting literature review.

1.4.2 Setting the instrument.

1.4.3 Preparing ozone generating system and investigating its performance.

1.4.4 Testing the ozone laundry system.

1.4.5 Investigating effects of ozone concentration, temperature and washing time on properties and appearance of fabric which will use a 100% cotton fabric.

1.4.6 Analyzing and concluding the experimental results.

1.4.7 Preparing a manuscript for journal publication and thesis.

CHAPTER II

FUNDAMENTALS AND LITERATURES REVIEW

2.1 Laundry processes [7,8]

Textile cleaning procedures have many factors to consider for removing soil and returning textile to nearly new fabric. And if we use wrong or inappropriate handling, there is a risk of damaging the textile.

2.1.1 Soil and stain

It is substance not intended to be on a textile by the contact or adsorption of any substance. Water-soluble soils such as coffee or milk are absorbed into hydrophilic fiber. Dust soils help on the fabric by electrostatic force. Organic soils such as oil, grease or gravy are absorbed by oleophilic fiber. They require suitable techniques and cleaning agents.

2.1.2 Method of cleaning

The techniques depend on the nature of the textile and the kind of soils. There are wet cleaning, dry cleaning, liquid supercritical, ultrasonic cleaning and ozone cleaning system etc. The cleaner’s goal is to remove the maximum amount of stains in the minimum amount of time with the least amount of damage possible to the fabric.

2.1.3 Hydrodynamic in household washing machines [9]

This model is based on a study of hydrodynamic of washing machines by Van den Brekel. In this study, four main movements of fabric are distinguished (Figure 2.1)

a) Pulling of the fabric through the liquid in the inner-drum

b) Lifting the fabric out of the liquid.

c) Falling of the fabric through the drum.

d) Impact of fabric on the wall (or on the liquid) after falling.

There four movements are all present in the drum partially filled with liquid and partially filled air. The movements create pressure drops across the fabric resulting in physical transport of the washing liquid and soil or stain through the fabric. Moreover, they also contribute to soil removal.

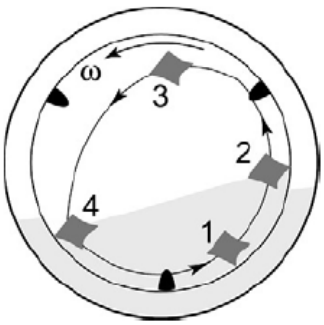


Figure 2.1. Path of a piece of a fabric in the rotating drum. [9]

2.1.4 Cleaning agent

To understand what is needed to achieve effective cleaning, it is helpful to have a basic knowledge of soap and detergent chemistry.

- Detergents or soap

They are a particular kind of surfactant. Surfactants (detergents) molecule includes two different parts. It have amphiphilic structure, meaning that one part of the molecule is a polar or ionic group(head) with a strong affinity for water, and the other part is a hydrocarbon group(tail) with an affinity for soil. Most surfactants and most fabrics carry negative electrostatic charges in water. These charges repel each other. Every time a soil particle surrounded by surfactant comes close to the fabric, the negative charges repel each other. This keeps the soil particles from reattaching themselves to the fabric. The soil particles stay suspended in water until they are flushed away in a rinse as could be schematically seen in Figer2.2.

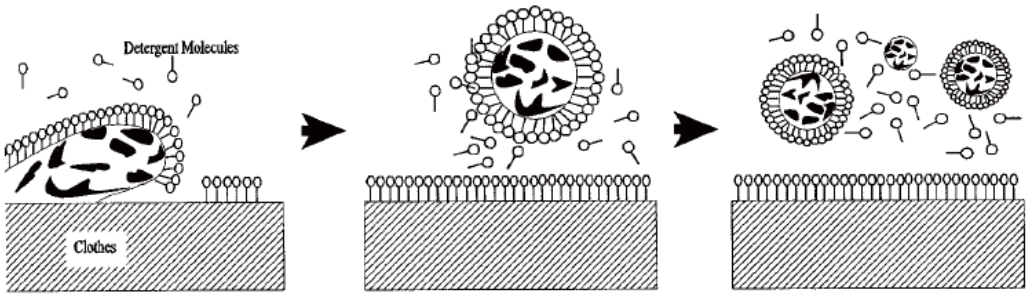


Figure 2.2 Surfactant process [7]

- Synthetic solvents

There are many synthetic surfactants and solvents on the market. Many of these solvents are made from petroleum. The most common synthetic solvent used for garment cleaning is perchloroethylene. Stoddard solvent, which is distilled from petroleum, is also commonly used in dry cleaning.

- Bleaches and oxidizers

Bleaching does not clean garments; it merely disinfects them. Whitening, odor removal and some organic stain removal is often accomplished by disinfection. Disinfection kills bacteria and mites that may accumulate on organic soils. Disinfection is accomplished by oxidation. Chlorinated bleaches oxidize and dissolve organisms that can then be flushed away in rinses. The speed of oxidation can be increased by exposing fabrics to certain forms of oxygen such as ozone or peroxide.

2.2 Textile fabrics

A textile is comprised of yarns, which in turn are made of fibers. So, fibers are the basic unit of most fabric. Textile fibers have been used to make cloth for several thousand years. There are two main types of textile fibers, namely natural fibers and man-made fibers. Natural fibers are obtained from various types plane, animal and mineral suck as cotton, flax and wool. Man-made fibers are made from chemical compounds produced in manufacturing facilities such as polyester and nylon.

2.2.1 Structure of fabric

The general structure and dual porosity of the textile can show in Figure 2.3 [3]. A woven textile fabric often has dual porosity: inter-yarn porosity and intra-yarn porosity.

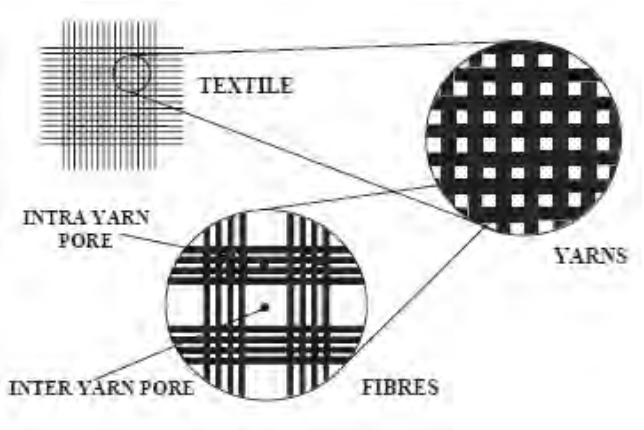


Figure 2.3. The general structure and dual porosity of the textiles [3]

2.2.2 Cotton [10 - 11]

- Physical structure of cotton

Naturally colored cotton is creamy white. Cotton cellulose is highly crystalline and oriented. The cotton fiber is made up of cuticle, primary wall, secondry wall, and luman. In the cell wall, cellulose occurs in small, crystalline microfibrils that are arranged in multilayer structures. (see Figure 2.4)

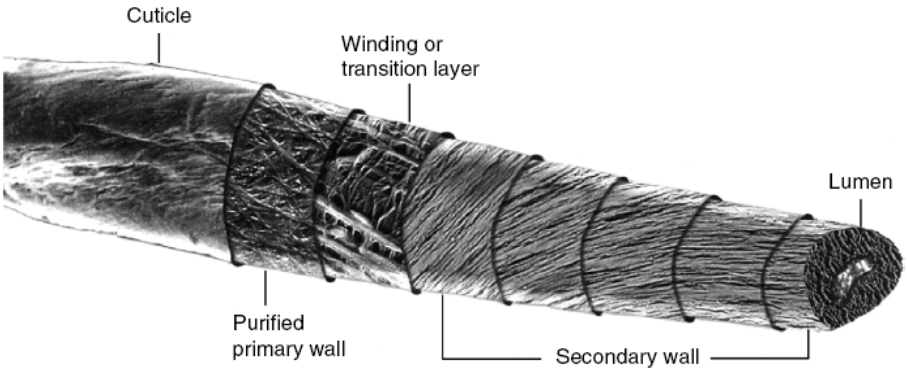


Figure 2.4 The structure of cotton [11]

- Chemical properties of cotton

The cotton fiber is composed of mostly ∝-cellulose (88.0–96.5%). The basic monomer of cellulose is glucose (Figure 2.5). Its chemical reactivity is the same as that of the cellulose polymer, a β-(1→4)-linked glucan. The chemical structure shows that the 2-OH, 3-OH, and 6-OH sites are potentially available for the same chemical reactions. the chains of cellulose molecules associate with each other by forming intermolecular hydrogen bonds and hydrophobic bonds.

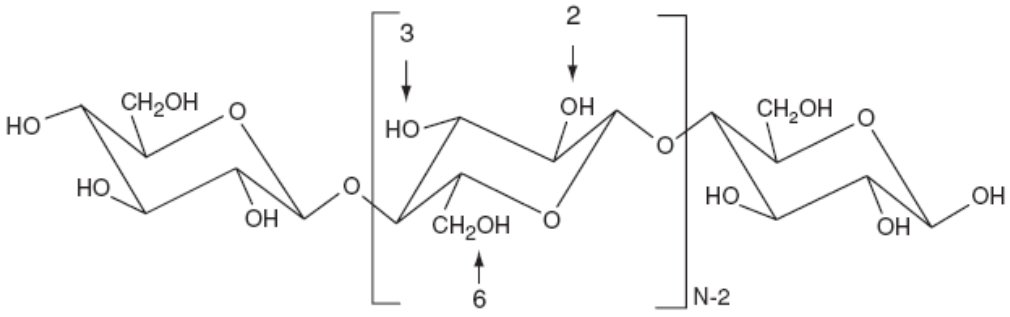


Figure 2.5 The chemical structure of cellulose

2.3 Transfer phenomena of chemicals in the fabric [12]

It has three steps to be considered.

a.) Transfer within the fiber

b.) Transfer within the fabric and between fibers

c.) Transfer in the water out of the fabric

Steps a and b are thought mainly due to diffusion. Step c is also due to diffusion near the fabric surface, but primarily depends on water movement.

From the results, the washing process may be shown in Figure 2.6. From state a to state b, there is a significant effect by initial condition, and the washing rate is very fast. From state b to c, the distribution of chemical concentration does not change, in the pattern and gradually becomes low. This period is the longest in the complete washing. From state c to d, the residual chemicals decrease to about two-thirds by squeezing. State d represents the beginning of the second washing, the distribution curve of which is different from the state a. So, there goes no fast washing like in the first washing. Similar processes are repeated in the third and further washing cycles. And squeezing always decreases residual chemical to about two-thirds independent of the fabric thickness. Hence, squeezing is much effective to washing.

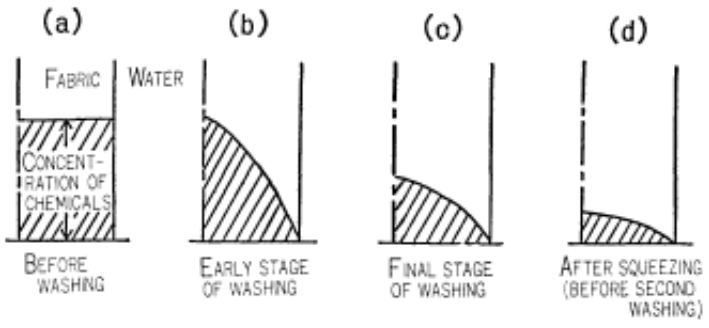


Figure 2.6 Distribution of chemicals in various stages of washing [12].

2.4 Oxygen [13]

Oxygen atom in nature has a lot of forms include with free atomic particle (O), oxygen (O_2), Ozone (O_3), O_4 is a very unstable and rare and oxygen anion (O^-).

Active oxygen anion (O^-), has strong oxidation power, is high reactive radical in anion chemistry, and at low temperature was oxidized hydrocarbons.

2.5 Ozone

2.5.1 Physical structure of ozone

Ozone is a molecule includes oxygen three atoms(Figure 2.7). It has molecular weight of 48 and has bond angle of 116.78°. The two bond lengths 1.278 °A. The bonding is single bond on one side and double bond on the other side. At room temperature ozone is a blue gas. In below -112 °C, ozone became dark blue liquid, and below -193 °C it became dark blue solid. [14]

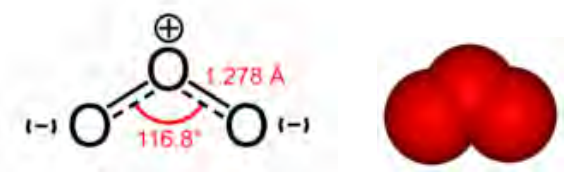


Figure 2.7 Structure of ozone [15]

2.5.2 Chemistry of ozone

One of powerful oxidizing agent is ozone. It don't produce by-product. It can react with organic compound, microorganism such as bacteria virus. It used to reduce color, odor and total organic carbon. It has oxidation potential 2.07 Volt. Ozone is

unstable gas, so it can decay to oxygen (O_2). The bonds are very weak, which is react with substance readily as it do. [13]

Ozone is a gaseous compound that contains three oxygen atoms. It is widely used in various applications. One of the most common use is to treat air or water for disinfection treatment because it is a powerful oxidizing agent. In oxidation, ozone molecule reacts with another substance. In general, double or triple bonds of unsaturated organic molecules could be easily destroyed.[16] Ozone is recognize as a very effective in removing a wide range of organics and inorganics compounds, killing bacteria and virus, removing colorating substance, reducing odor. Therefore, it is reasonable to consider that ozone is a superior cleaning reagent for fabric cleaning. There are several methods of generating ozone. Conventional ozone generating system is used corona discharge to produce ozone in gas phase. It suffers with limitation of dissolving ozone in water. Therefore, this work proposes to use electrolytic generation of ozone because electrolysis is considered to be more advantageous than the classical corona discharge [16,17]. With this technique, water is direct employed to ozone solution.

2.5.3 Electrochemical oxidation of ozone [18]

Ozone is sometimes used as a disinfectant in place of chlorine. Ozone oxidizes water contaminants directly through the reaction of O_3 and indirectly by generating hydroxyl radical ($HO\cdot$), a reactive strong oxidant.

The Electrochemical Oxidation Potential (EOP) of ozone is considerably higher than other disinfecting agents:

Table 2.1 The Electrochemical Oxidation Potential [18]

Disinfecting agent	Eop (V)	Eop v. Chlorine
Fluorine	3.06	2.25
Hydroxyl-redical	2.08	2.05
Oxygen (atomic)	2.42	1.78
Ozone	2.08	1.52
Hydrogen dioxide	1.78	1.30
Hyperchlorite	1.49	1.10
Chlorine	1.36	1.00
Chlorine dioxide	1.27	0.93
Oxygen (molecular)	1.23	0.90

2.5.4 Applications of ozone

Ozone is a strong oxidizing agent. It can kill bacteria and decreases color and odor. There are some evidences reported that it is suitable for industry or home use. Ozone is used in wide, air treatment and water treatment. Applications of ozone have variety types such as [15]

- Laundry in hospitals and hotel
- Disinfect water before used in home
- Ozone swimming pool and spa sanitation
- Wash fresh fruits and vegetables
- Removal of dyes from textile industry waste water
- Recycle water in cooling towers
- Disinfection of water for pharmaceuticals

2.6 Ozone generations [13,14]

Ozone is generated in various methods. Three processes are currently known for the production of ozone including ultra-violet light (UV-light), corona discharge and electrolysis. Choice of used ozone generations depend sources and field of application.

2.6.1 Ultraviolet light (UV-light)

Ozone is produced by air or oxygen exposed to ultra-violet (UV) light at a wavelength of 185 nm ultraviolet radiation from a mercury vapor lamp. If used higher wavelength than 200 nm can became toxic gas. O_2-molecule can dissociate in two oxygen atoms. The oxygen atom can then combine with a neighbor O_2 molecule producing a ozone molecule.

$$O_2 + hv \rightarrow 2O^o$$

$$O^o + O_2 \rightarrow O_3$$

2.6.2 Corona discharge

Ozone is generated by passing air or oxygen through a high voltage electrical field. A high voltage alternating current is applied to the plates and the ozone is formed in the air gap when O_2 molecules disassociate and recombine into O_3.(Figure 2.8)

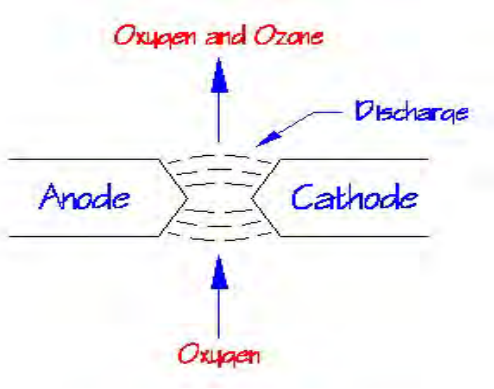


Figure 2.8 Diagram of the corona discharge ozone generator [14]

A voltage is placed across an electrical discharge (anode, cathode) and either air through the gap at high temperature. The quantity of ozone produced is dependent on several factors, such as the voltage and frequency of the alternating current applied to the cell

2.6.3 Electrolysis

The electrolysis generator consists electrolytic cells, anode and cathode, electrolyte membrane. Ozone is generated from high pure water. O_3 and O_2 leave on the anode side, while H_2 leaves cathode side. Therefore, the anode and cathode would be made of porous, water can flow. Anode used current-conductive carrier material is coated with an active layer such as catalyst. Materials of the cell have to stable and high conductivity, consist of metals or metal-oxides (Figure 2.9).

. The membrane, which functions as both electrolyte and separator between anode and cathode, is contacted on both sides by the activated porous electrodes. The water fed to the anode side of the cell is dissociated at the interface between the anode and membrane as a result of the DC current being applied. To ensure that as much

ozone as possible is produced, the anode must have an over potential above the decomposition and the ozone reaction potential and the catalytic layer must inhibit the formation of diatomic oxygen and encourage the formation of ozone.

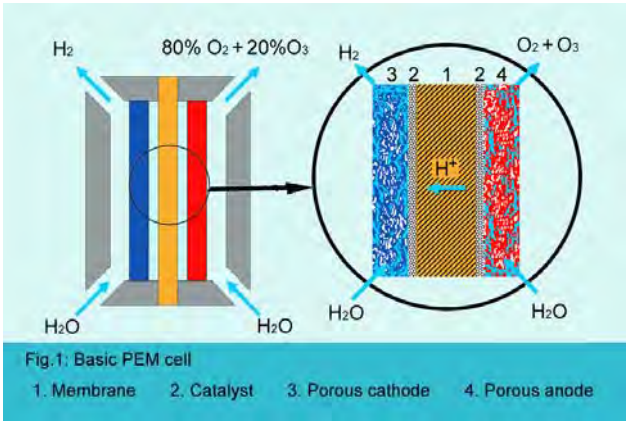


Figure 2.9 Diagram of the electrolytic ozone generator [19]

2.7 Mass transfer of ozone into aqueous systems

When gas and liquid phases are in contact, components can be transferred from one phase to the other until equilibrium is recahed. The transfer of a component from one phase to another across a separating interface is due to a concentration gradient, which is caused by a resistance to the mass transfer developed in each phase. The resistance in one phase is the contribution of the diffusional resistance in the laminar film and the resistance in the bulk fluid. The latter resistance usually is considered negligible compared to the diffusional resistance. The film mass transfer coefficient is proportional to the molecular diffusion coefficient at a certain power n, (D^n)(see in Figure 2.10) .

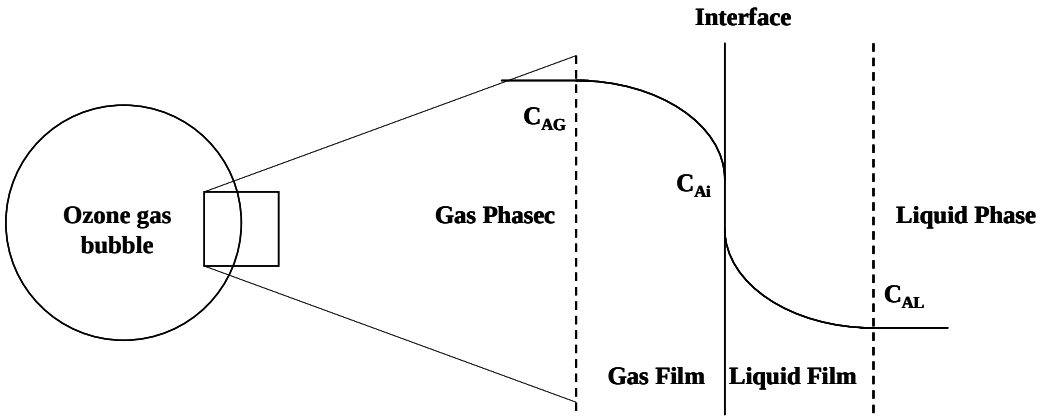


Figure 2.10 Mass transfers between two phases.

2.8 Ozone reaction mechanisms

In general, ozone reacts substance with two different ways, direct and indirect reactions. These different reactions are controlled by different types of kinetics(Figure 2.11).

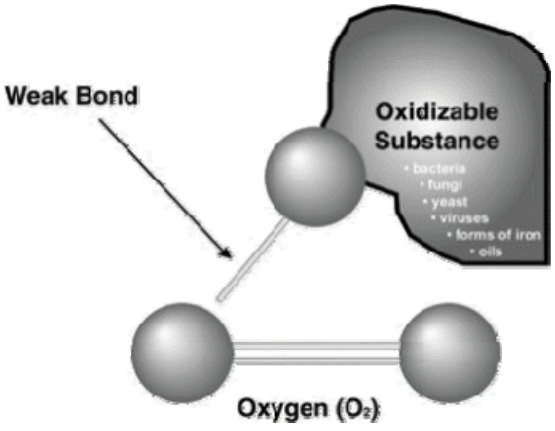


Figure 2.11 Reaction of substance [20]

2.8.1 Direct reactions [13]

Ozone molecule reacts with organic compounds by unsaturated bond or double bond of organic compounds which is broken with ozone and lead to different oxidation products.

$O_3 + M \text{ -----> } M_{oxide}$ M: Micropollutant

2.8.2 Indirect reactions

Ozone is unstable in water and decomposes into secondary oxidants, hydroxyl radicals ($OH^{\bullet}$).The first step is decay of ozone, accelerated by initiators such as OH^-. The radical pathway is influenced by the type of dissolved substances in water. Three different steps are the widely used mechanism (Figure 2.12)

- Initiation step - Formation of superoxide anion radical ($O_2^{\bullet -}$)

- Propagation step - Formation of hydroxyl radicals and re-initiation of the chain reaction

- Termination step - Inhibitors (scavengers) stop the re-formation of the superoxide anion radical.

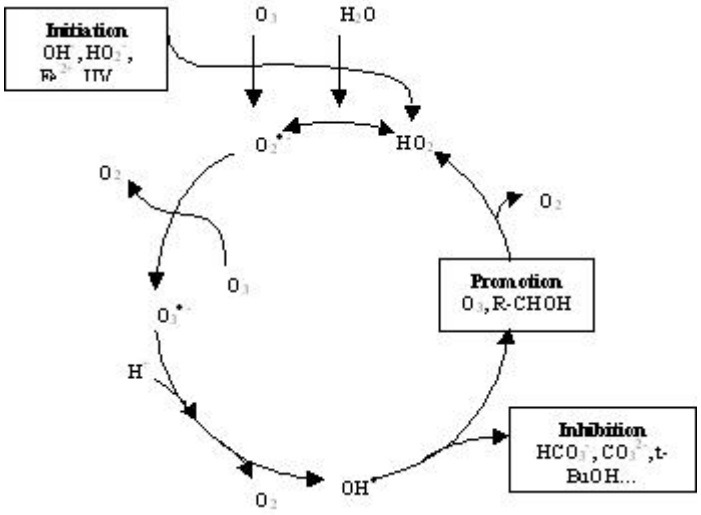


Figure 2.12 Mechanism of the indirect ozonation. [14]

2.9 Linoleic acid ($C_{18}H_{32}O_2$)

Linoleic acid is an unsaturated omega-6 fatty acid. It is a colorless liquid. In physiological literature, it is called 18:2(n-6). Chemically, linoleic acid is a carboxylic acid with an 18-carbon chain and two *cis* double bonds; the first double bond is located at the sixth carbon from the omega end (Figure 2.13).

Linoleic acid is a polyunsaturated fatty acid used in the biosynthesis of prostaglandins. It is found in the lipids of cell membranes. It is abundant in many vegetable oils, especially safflower and sunflower oils.

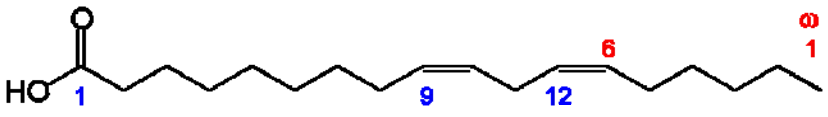


Figure 2.13 Chemical structure of linoleic acid [15]

2.9 Literature reviews

S. Stucki et al., (1985) [21] investigated the solid polymer electrolyte for ozone generator. The solid polymer electrolyte had current efficiency of 15% using low cell voltage at room temperature. The main advantage was the fact that the membrel cell directly produces an aqueous solution of ozone ready for various oxidizing and disinfectant applications.

B. Carroll (1995) [22] investigated the removal of oil droplets from solid substrates by hydrodynamic forces for removing oily soil. The drag forces were controllable. This research was studied about as the contact angle, the interfacial tension, the droplet size and the substrate type. This result showed the contact angles did not affect to the removal oily. The droplet size and the interfacial tension was inversely the ease of the removal of the oily.

M. Prabaharan et al., (2000) [23] studied on bleached cotton fabric by ozone/oxygen gas mixture. The ozone flow rate was 0.5 l/min. Fix ozone concentration and treatment time, which were 25-100 g/m^3 and 5- 60 min, respectively. The effects of ozone concentration and treatment time on property of bleached fabric were studied. Result, bleaching fabric at ozone concentration 100 g/m^3 in very short time (5 min) at room temperature can treatment fabric has whiteness index 80%.

L. M. D. Silva et al., (2002) [24] reported about ozone production using corona discharge technology. The output from a modern corona-discharge system had been report to vary from 2 wt% for air as the input gas to 7 wt% for oxygen as the input gas. This study revealed an increase in O_3 concentration with decreasing temperature.

B. T. Stanley (2004) [18] reported about the disinfection with ozone solution. At low ozone concentration in 0.008 ppm could stop the microbial growth (Figure 2.14). This result showed that very low ozone concentrations in the magnitude of 0. 1 to 0.2 mg/l were sufficient to keep germ counts below 1 c.f.u. per 100 ml.

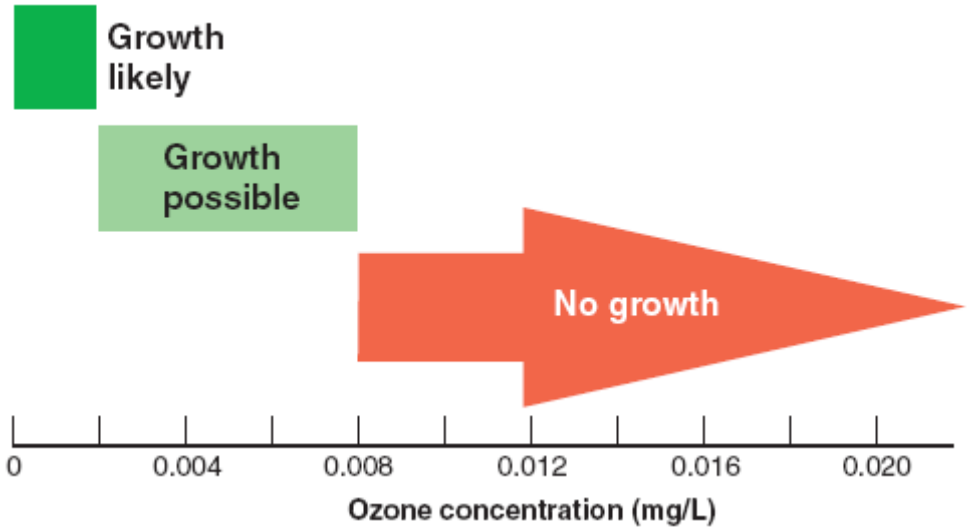


Figure 2.14 Microbial growth as a function of ozone concentration [23]

C. Kulchanrat et al., (2005) [25] investigated the performance for laundry system was used ozone technology. This research would be studied about comparative evaluation of energy and cost saving. In the experimental, the ozone generator was corona discharge. Ozone level of 0.01- 0.02 ppm by flow water passes ozone gas. They were mixed and then flow ozone solution into laundry system. The results showed the ozone system could save the total 50.02 % with comparing to the older system. Bacteria on fabric were decreased 0.02 $\times 10^3$ CFU/g.

S.N. Patel et al., (2005) studied demonstrate that domestic washing mashing machines was laundered hospital staff uniforms at home. The clothing was including bacterial count of *Staphylococus aureus.* This system was used temperature for washing at 40 °C and 60 °C and remaining fabric were tumble dried separately 30 min. The dried fabrics were ironed. The results showed it can reduce *S. aureus* to below detectable levels from an inoculum of $10^8 – 10^{12}$ colony-forming units ($\geq 10^6$-fold reduction), even using low temperature (40 °C) programmes. And *S. aureus* were destroyed by tumble dried and ironed.

A. Kraft et al., (2006) [22] developed electrolysis ozone production by using diamond anodes and solid polymer electrolyte. The ozone production rate depends on current density, water flow rate and water conductivity. The electrode sandwich was positioned inside a pipe flow through reactor. Water with different conductivities was pumped only through the reactor. The temperature was about 20°C for all experiments. The current supply was 20V, 25 A and were carried out in two-

electrode-technique with galvanostatic current mode. They found ozone production is good at high flow rate and low conductivity of the electrolyed water.

D. Sargunamani et al., (2006) [26] investigated the effect of ozone treatment on the properties of raw silk fabrics compared with hydrogen peroxide and soad degrumming. The treatment resulted in increase in yellowness index and decrease in breaking strength and elongation, weight and flexural rigidity. Soap degumming of raw silk fabric resulted in lower yellowness index and flexural rigidity and lesser loss in breaking strength and elongation compared to that of ozone treated material. There was not much of difference between ozone and hydrogen peroxide treatments of degummed silk fabric except for the lower yellowness index.

S. D. Perincek et al., (2007) [27] investigated about ozone gas in bleaching of cotton fabrics. They found ozone gas bleaching of cellulose much faster than hydrogen peroxide and hypochlorite. But this process induced some damage to cellulose. Moreover, ozone water had disinfecting and cleaning power without the need for hot water, it could clean commercial laundry with little or no need for hot water, enhances chemical utilization, and greatly reduces water usage. In this study, they were found that room temperature and 10–12°C of medium provided nearly the same whiteness degrees. They decided to make treatments at room temperature.

CHAPTER III

EXPERIMENTAL

The chapter explains the experiment of ozone washing system about the fabric, the chemicals, the equipment, the experimental procedure and the analytical instruments.

3.1 Materials

3.1.1 100% cotton fabric with 162 g/m^2

3.1.2 Linoleic acid ($C_{18}H_{32}O_2$) from Sigma Chemical, 99.99%

3.1.3 Potassium iodide (KI) from Sigma Chemical, 98%

3.1.4 Sodium thiosulfate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$) from Sigma Chemical, 98%

3.1.5 Sulfuric acid (H_2SO_4) from Sigma Chemical

3.1.6 Sodium hydroxide (NaOH) from Sigma Chemical, 97%

3.1.7 Ethyl alcohol (C_2H_6O) from Sigma Chemical, 99 %

3.1.8 ECE non-phosphate reference detergent from SDC Enterprises Ltd.

3.2 Experimental set-up

The system consists of deionised water generator, electrolysis ozone generator and washing machine. The equipment was set up as shown in Figure 3.1

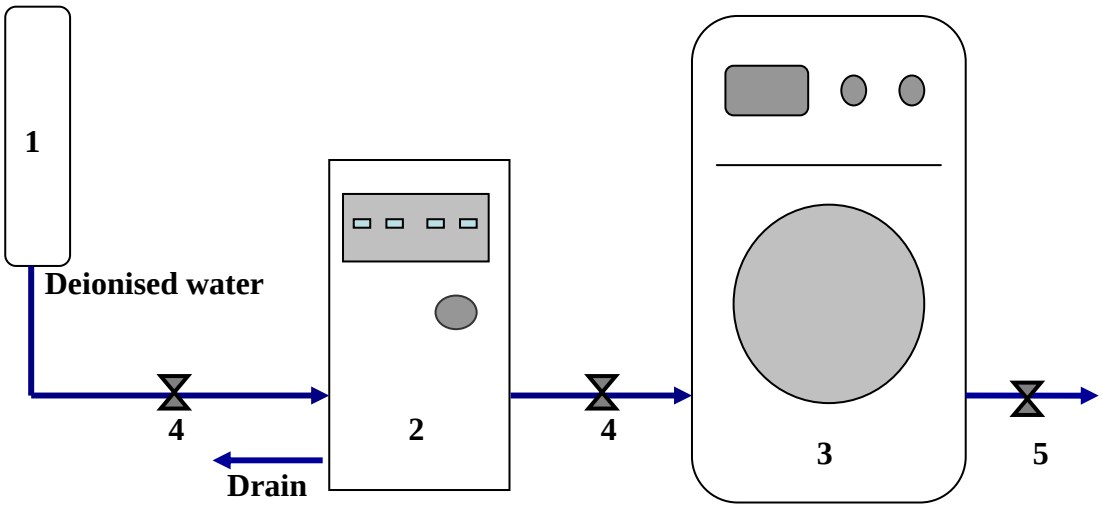


Figure 3.1 Schematic diagram of experimental for electrolysis ozone washing system 1. deionised water generator, 2. electrolysis ozone generator, 3. washing machine 4. 3-way valves and 5. drain valve

Deionised water was produced by bed ion exchange connected into electrolysis ozone generator. The conductivity of the deionised water was about 1 μS/cm. The water temperature was about room temperature (25°C) in all experiment.

The electrolysis ozone generator was produce by Ozzon model I-8200 ozonated water generator. The ozone concentration in the deionised water was determined by iodometric method. And it was connected to the washing machine (Electrolux model EW 660F).

3.3 Experimental procedures

3.3.1 Ozone measurement (Iodometric method)

In my experiment had used iodometric method for measuring ozone concentration. An ozone solution was mixed with potassium iodide (KI). The iodide (I^-) is oxidized by ozone. The reaction product iodine I_2 was titrated immediately with $Na_2S_2O_3$ to a pale yellow color with a starch solution indicator. The endpoint of titration could be intensified (deep blue). The ozone concentration could be calculated by the consumption of $Na_2S_2O_3$.

3.3.2 Fabric preparation

All fabrics used in experiment were 100% cotton. The fabric sized is 15 × 15 cm squares.

- Conventional alkaline scouring

Alkaline scouring is removed the non-cellulose compounds (hemicellulose, waxes, pectin and proteins) by boiling in an aqueous solution containing 5 g/l NaOH at 80°C for 20 min using a material to liquor ratio 1:20. After the alkaline treatment, the fabrics were washed twice times with the distilled water. Then, the specimens were squeezed by the mangle machine for wringing out water. And it was dried at 60 ^oC in oven until it dried.

- Preparing stain on the fabric

The fabrics were soaked in linoleic acid which is considered as the simulated stain, for 5 min at room temperature. Then the fabric samples were squeezed by the mangle machine for remove more stain from fabric in prior to testing.

3.3.3 Washing experiments

In the conventional cleaning process, the specimens were put through to stirred hot water with a constant temperature of 60 ºC for 15 min and then rise 3 cycles for 3 min in each cycle. Next squeezed by mangle machine and then dried in an oven for 45 min at 60 ºC.

In the ozone cleaning system, the soaked the specimens were washed in the solution of ozone with varied concentrations of 0.5, 1.0, 1.5, 2.0 and 2.5 ppm. Standard ECE detergent was added to wash the specimens for 5 -20 min (decrease of chemical agent to 50% of normal used). The rising step would be conducted twice for 3 min in each cycle. Finally, the specimens were dried by the same condition. The washing processes are shown in Table 3.1.

Table 3.1 Washing processes

Step	**Conventional process**	**Ozone solution process (0.5 -2.5 ppm)**
Washing	Time : 15 min Temp : 60 °C Detergent : -66 g/1.8kg sample load - ECE Reference detergent	Time : 5 -20 min Temp : room temperature Detergent : -33 g/1.8kg sample load - ECE Reference detergent
Rinsing	Time : 3 min Cycles : 3 Cycles	Time : 3 min Cycles : 2 Cycles
Dryer	Time : 45 min or until dry Temp : 60 °C	Time : 45 min or until dry Temp : 60 °C

3.3.4 The water absorbency test [28]

This method based on the AATCC 130-1995. The water absorbency was determined by measuring the time it took for 250 micro-liters of water to be absorbed. The water absorbency was recorded three times, these is prior to staining, after staining and prior to washing and after washing. Resulted from these tests were used to calculate the Recovery Factor(R). This factor measures the ability of the washing process to restore the absorbency of the specimens to its initial state prior to staing. A 100% recovery is ideal. The formula used was a follows below;

$$R = \left[\frac{\left(A_0 - \left(A_w - A_p \right) \right)}{A_0} \right] \qquad (3.1)$$

Where A_0 = Absorption time prior to staining

A_w = Absorption time after stained with oil

A_p = Absorption time after washing

3.4 Analytical instruments

3.4.1 Scanning electron microscopy (SEM)

A microscopic evaluation of physical morphological appearances of cotton fiber surface occurring during of washing and unwashing cotton specimens were carried out using scanning electron microscope (Jeol model JSM-6400), where samples were coated with a thin layer of gold before observation. This instrument is shown in Figure 3.2

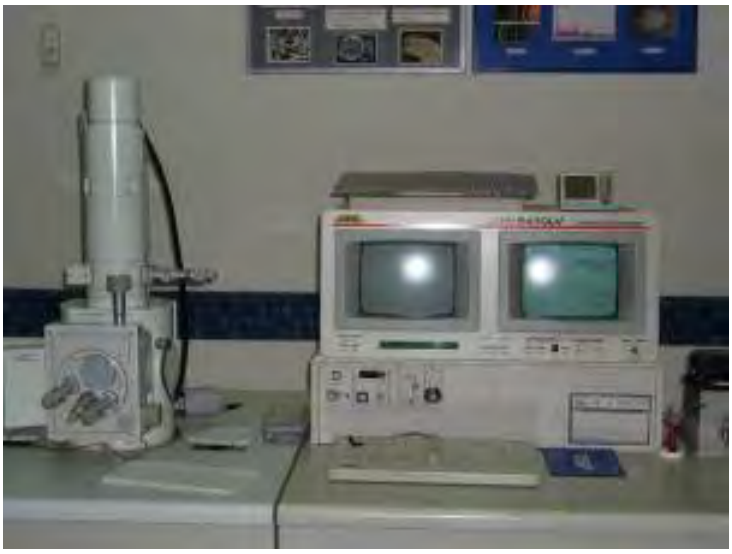


Figure 3.2 Scanning electron microscope (SEM) (Jeol model JSM-6400)

3.4.2 Fourier transform infrared spectroscopy (FT-IR)

A FTIR was adopted to study the physical structure of the surface fiber cotton. That was obtained with the FT-IR model1760x. FT-IR spectrometer, equipped with an attenuated total reflection (ATR) cell. It was used to separate the function organic, inorganic compounds and chemical bonding of material in liquid phase and solid phase. This instrument was shown in Figure 3.3.

Figure 3.3 Fourier transform infrared spectrometer (FT-IR) (model1760x)

3.4.3 Tensile testing

Tensile property of the warp yarns of the fabric sample was measured using universal materials testing machines (INSTRON model 5567) in accordance with ASTM D 5034 -95. As least ten specimens were tested for each set of the sample and the mean and standard deviation values were reported. This instrument was shown in Figure 3.4**.**

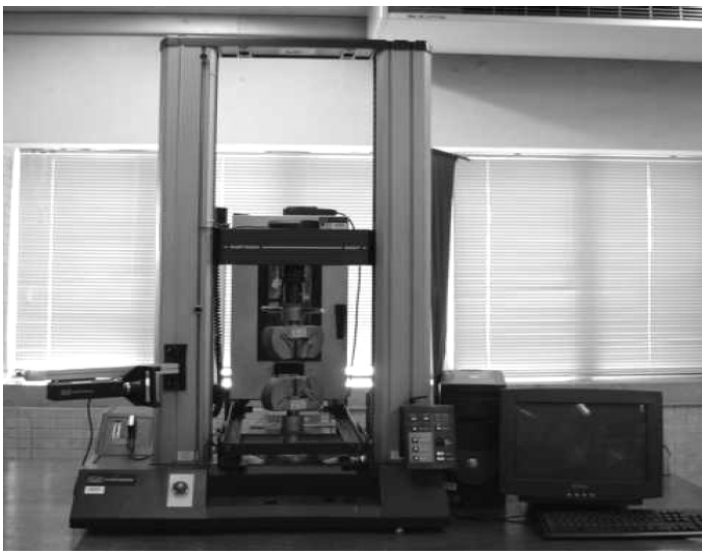


Figure 3.4 Universal materials testing machine (INSTRON model 5567)

3.4.4 Computer color matching (CCM)

The color of fabric was measured using Macbeth Color Eye 3100 spectrophotometer. It could be used for indicating the change of the color before and after washing. This instrument was shown in Figure 3.5.

Figure 3.5 Computer color matching machine (CCM) (Macbeth color eye 3100)

3.4.5 UV-visible spectroscopy

The instrument of UV-visible spectrophotometer (Phamaspec UV-1700, Shimadzu) was used to measure the transparent solution at wavelength 300-700 nm. Furthermore, this apparatus could be measured the transparent of the draining water. This instrument was shown in Figure 3.6.

Figure 3.6 UV-visible spectrophotometer

3.4.6 Total organic carbon analysis (TOC)

The TOC disappearance was obtained from Shimadzu TOC-VCPH. It could be used for indicating the total mineralization of organic carbon. The picture of TOC analyzer is shown in Figure 3.7.

Figure 3.7 Total organic carbon analyzer (TOC) (Shimadzu TOC-VCPH)

CHAPTER IV

RESULTS AND DISCUSSION

In this chapter experimental results on preparation of ozone solution using an electrolysis ozone generator, reaction of ozone and linoleic acid, reaction between ozone and clean cotton fabric and treatment of linoleic acid-doped cotton fabric by ozone solutions would be described and discussed.

4.1 Effect of water flow rate on preparation of ozone solution

In this part, the water flow was continuously fed into the ozone generator which was operated at 25 °C. Ozone concentration was measured using the iodometric method by sampling ozone solution at the outlet of the generator.

Based on the schematic diagram shown in Figure 4.1, it could be seen that ozone, which is generated via electrolysis reaction at the cathode, would be dissolved into the water flow by mass transfer at the surface of the electrode. With the higher water flow rate it could reasonably be implied that contact time of water with the electrode surface would become shorter. As a result, Figure 4.2 shows that the concentration of ozone in the solution was strongly dependent on the water flow rate. The ozone concentration was inversely proportional to the flow rate of water. The ozone concentration was higher with a decrease in the water flow rate. This result suggested that the ozone gas emerging from the electrode surface would require a certain time period to contact with the water flow before being dissolved into the

water. At a sufficiently low water flow rate, ozone would possibly be dissolved until achieving its saturated concentration. It should be noted that because of the effective volume of the ozone generator is constant, the residence time of water in the generator would be inversely proportionally to the water flow rate. As could be seen in Figure 4.2(b), ozone could not be dissolved into the water if the residence time was shorter than 120 sec. On the other hand, the ozone concentration would achieve a certain level of 2.62 ppm if the residence time was longer than 480 sec (or water flow rate of 22 ml/sec). As a result, it could be implied that under the experimental conditions employed in this work, the saturated ozone concentration was c.a. 2.62 ppm.

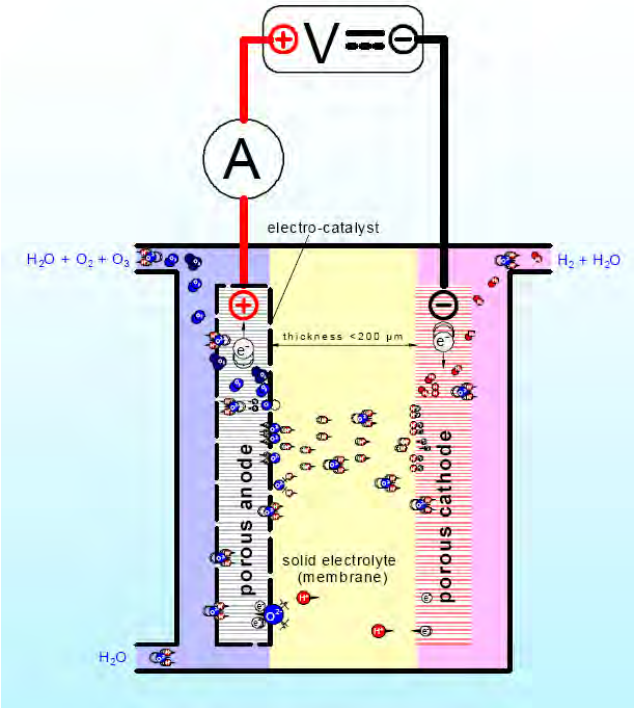
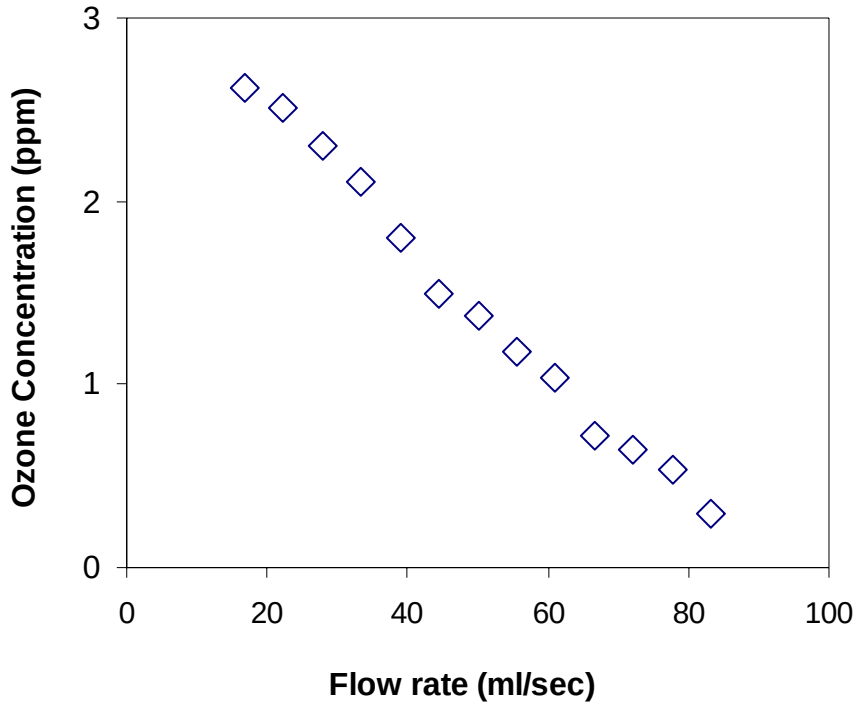
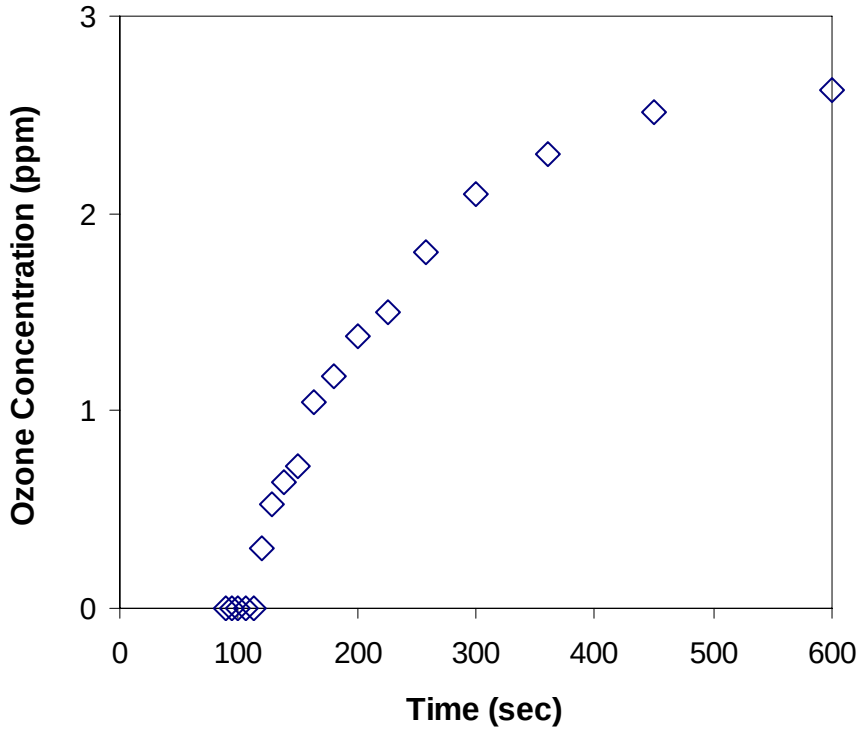


Figure 4.1 Ozone transfer within the ozone generator employed in this work [14]



(a)



(b)

Figure 4.2 Dependence of ozone concentration on (a) water flow rate and (b) residence time of water flow

With the steady state and isothermal assumptions, the ozone concentration could be determined using Henry's law which takes into account the solubility of ozone into the water. It is generally known that the Henry's law constant is dependent on the partial pressure of dissolved gas and temperature [29]. At constant temperature, the ozone gas dissolved into water is directly proportional to the partial pressure of ozone gas generated from the electrolysis. Based on the gas analysis, the saturated concentration of ozone was 5 ppm. This calculation result is in good agreement with the experimental result.

4.2 Removal of linoleic acid by ozone

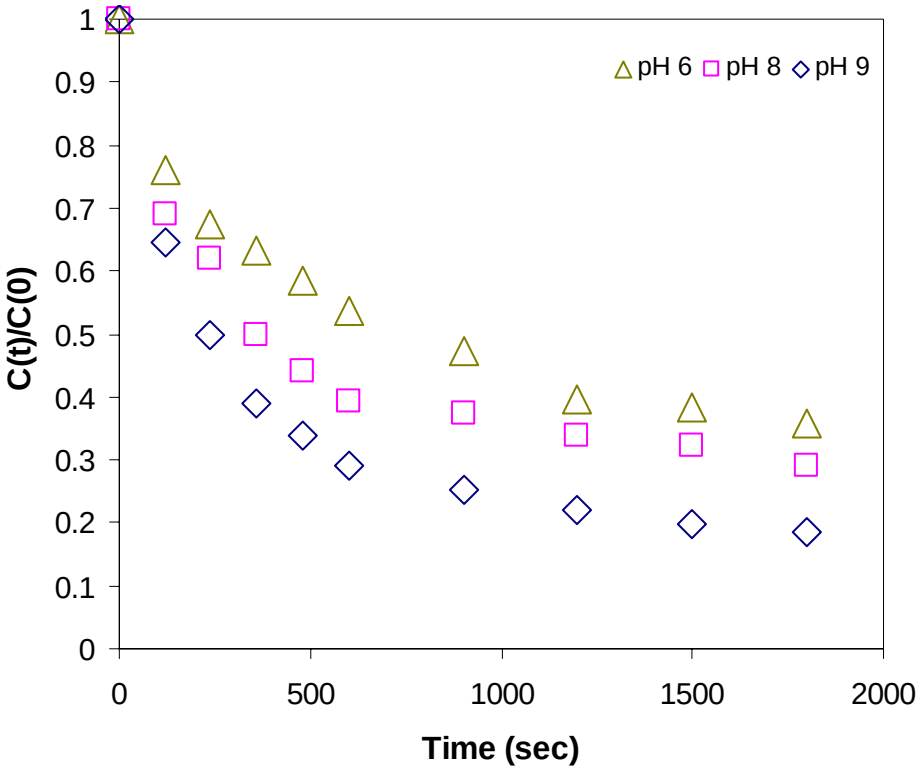
Before employing the prepare ozone solution to treat cotton fabric contaminated with the representative contaminant, the reaction between ozone and linoleic acid was intentionally examined. The experiments were conducted with 100 ml of 10 ppm of linoleic acid concentration into 1000 ml. of 0.5, 1.0, 1.5, 2.0 and 2.5 ppm ozone solution. The concentration of linoleic acid was analysed by UV-vis spectrometer which provides a strong absorption band at 233 nm [30]. Then some important parameters, pH and ozone concentration were examined and discussed as follows,

4.2.1 Effect of pH on removal of linoleic acid

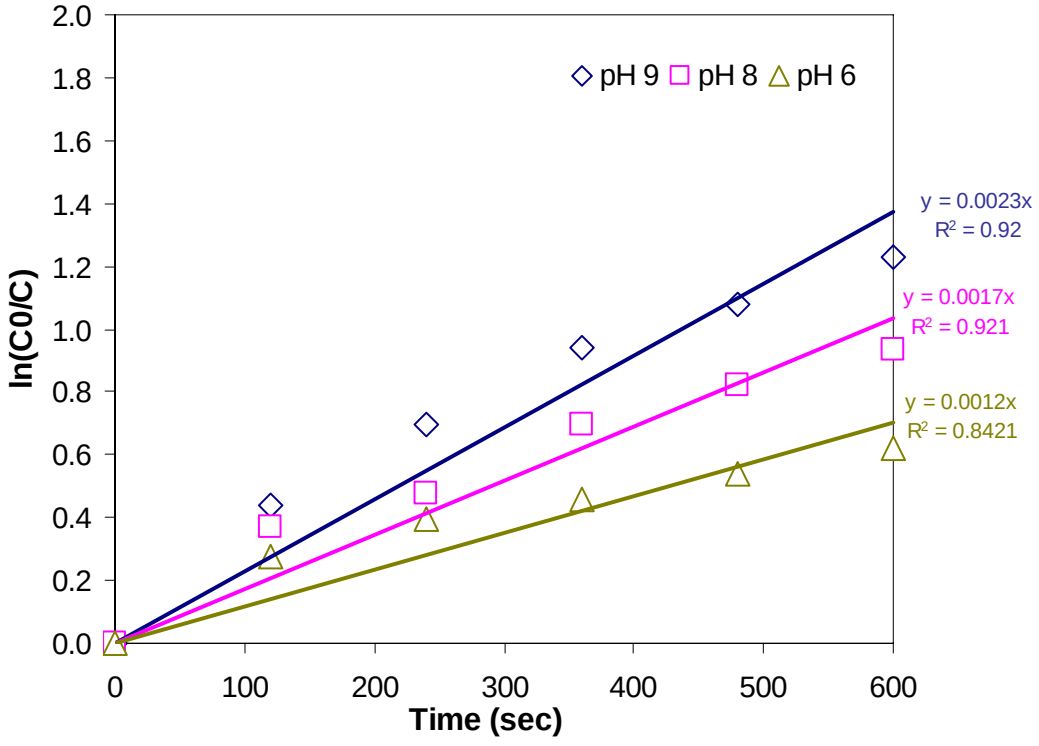
The pH of the solution was adjusted by hydrochloric acid (HCl) or sodium hydroxide solution (NaOH). The removal of linoleic acid by ozone has been investigated at different pH (6, 8 and 9) in 25°C. At pH 9, the reaction of linoleic acid with ozone solution was significantly fast. 50 % of linoleic acid was removed within

300 sec 5 min (Figure 4.3(a)). The enhanced oxidation reaction of linoleic acid in the ozone solution with the increase in pH would be attributed to the formation of some radicals, such as hydroxyl radical (·OH). It was reported that ozone would react with organic compounds in water through two different pathways [13]. With the direct oxidation, ozone attacked on the linoleic acid molecules directly. On the other hand, with the indirect oxidation mechanism, ozone would self-decompose in water to produce ·OH, which can act as stronger oxidant. The existence of OH anion would play an important role in formation of the ·OH radical [13]. As a result, with the oxidation of linoelic acid in the ozone solution with the pH 9 would preferably undergo the indirect oxidation route.

The experimental results could be employed to confirm that when the ozone solution with pH 8 and 6, the degradation of linoelic acid became much slower. These results would reasonably be ascribed to the direct oxidation of linoelic acid by ozone. Rate of reaction at each time point was also analyzed and shown in Figure 4.3(b). These results confirmed that the degradation rate of linoelic acid in the ozone solution with higher pH would be more enhanced by additional formation of radical reaction.



(a)



(b)

Figure 4.3 (a) Degradation of linoleic acid within ozone solution with different pH

(b) Rate of linoelic acid degradation with different pH

4.2.2 Effect of ozone concentration on degradation of linoleic acid

Based on previous investigation the condition at pH 9.0 was selected for further examination because of the fastest reaction rate. Linoleic acid removal is defined as:

$$\text{Linoleic acid removal (\%)} = \frac{[LA]_0 - [LA]_t}{[LA]_0} \times 100 \qquad 4.1$$

It could be found in figure 4.4 that the faster degradation of linoleic acid could be achieved with the increased ozone concentration from 0.5 mg/l to 2.5 mg/l. It should also be noted that the 1.0 ppm ozone solution could oxidize 50% of linoleic acid. With the maximal value of 2.5 ppm the prepared ozone solution could oxidize linoleic acid up to 80 %.

The ozone oxidation is subject to the direct reaction with double bonds of the linoleic acid. The mechanism of ozone reaction was to break the unsaturated bond (double bonds) of linoleic acid, resulting in many products which are nonenal, hexanal, nonanoic acid and azelaic acid as reported by Tamar and et al. [31].

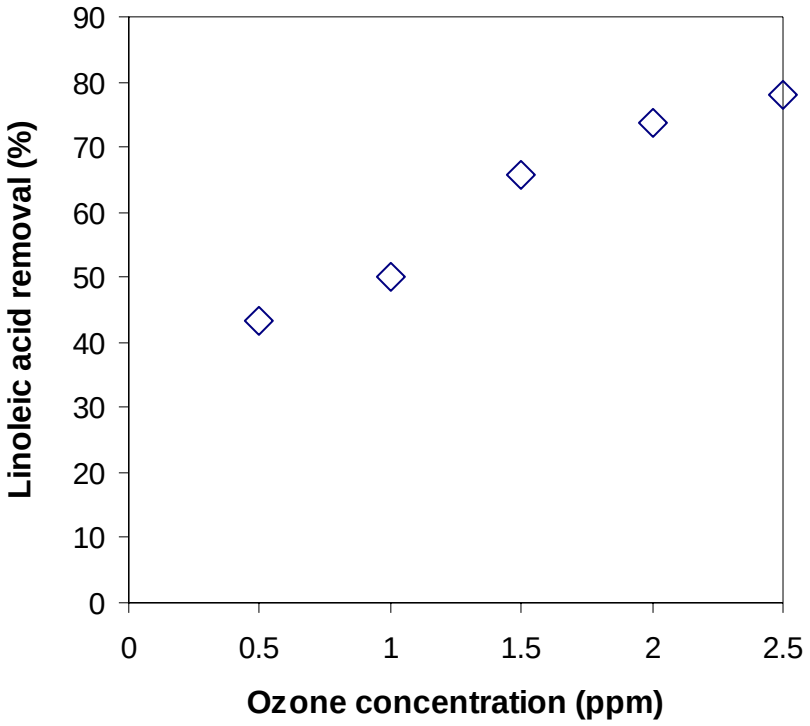


Figure 4.4 Linoleic acid removal with different ozone concentration.

(pH = 9, reaction time = 20 min)

4.2.3 Ozonation kinetics

The general rate equation of linoleic acid oxidized by ozone could be formulated by Eq. 4.2. However, with an assumption of excessive amount of ozone, the reaction is pseudo-first-order with respect to the linoleic acid concentration as derived in Eq. 4.3.

$$-r_{LA} = \frac{-d[LA]}{dt} = k[O_3][LA] \qquad 4.2$$

In this study, the pseudo-first-order trend was observed in all experiments. It could also be confirmed that the ozone concentration was almost constant, leading to

the estimation of the rate constant of the degradation of linoleic acid by employing Eq. 4.3.

$$-r_{LA} = \frac{-d[LA]}{dt} = k'[LA] \qquad 4.3$$

where k'is the pseudo first-order rate constant of linoleic acid.

Figure 4.5 shows the change of ln (C_0/C) against the elapsed time. It is a straight-line relationship for a typical condition of ozone concentration of 2.5 ppm. The rate constant, k' determined from the slope was 2.3×10^{-3} (sec^{-1}).

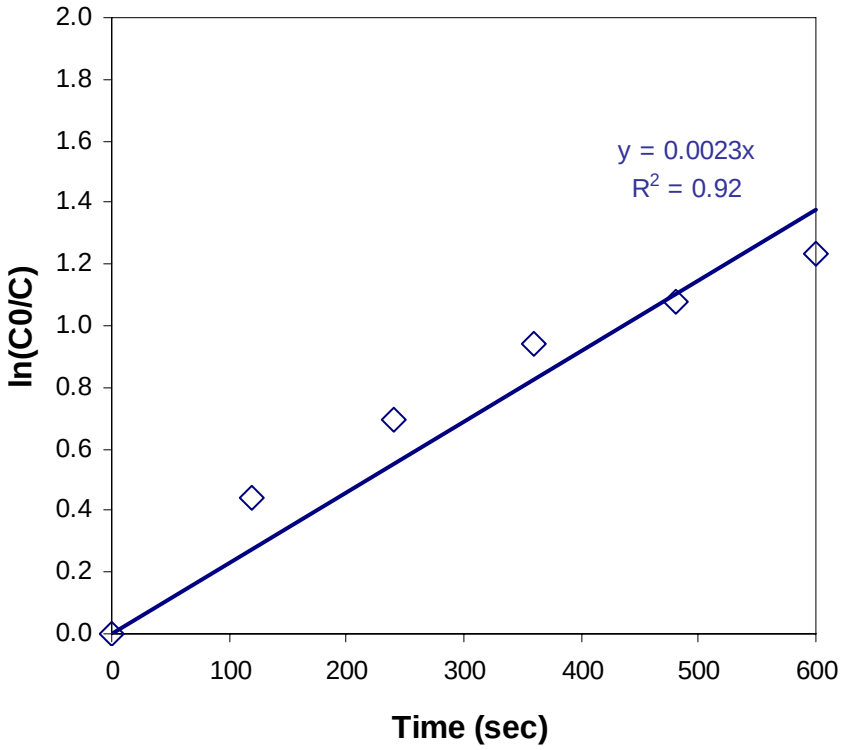


Figure 4.5 Linoleic acid removed rate by ozonation. (pH = 9, T = 25°C)

For further analysis of dependence of the rate constant, k' on the initial concentration of ozone solution was also conducted. The natural logarithm of the pseudo first-order rate constant (ln k') was plotted against the natural logarithm of the initial ozone concentration ($\ln[O_{30}]$). As could be observed in Figure 4.6, it was also a straight-line relationship. The slope of the relationship was 1.12. As a result, an empirical equation for predicting the pseudo-first order rate constant from the initial ozone concentration could be expressed as follows,

$$\ln k' = a \ln\left[O_3\right] + b \qquad 4.4$$

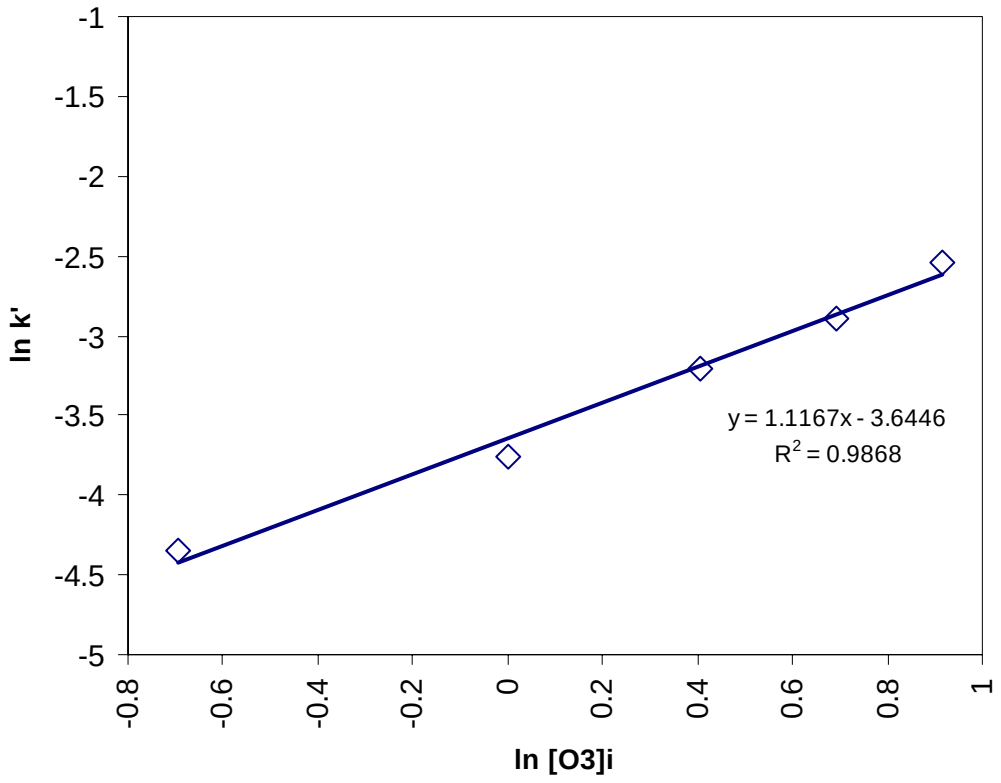


Figure 4.6 Relationship between the pseudo first-order rate constant and initial ozone concentration

4.3 Oxidation of cotton fabric by ozone solution

Cotton fiber is mainly composed of cellulose. We could see from the chemical structures of the cellulose in Figure 4.7. Cellulose is carbohydrate polymers. It is an organic compound, which has carboxaldehyde group, hydroxyl group and carbonyl group.

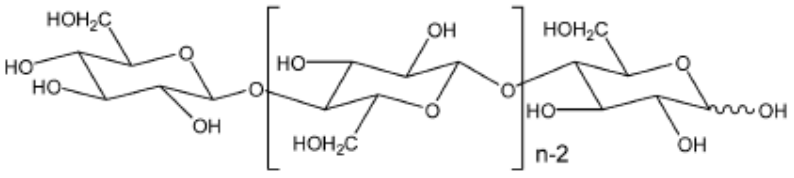


Figure 4.7 The chemical structure of cellulose.

FT-IR measurements indicated the changes in the main characteristic groups of non-cellulose impurities on the cotton fabric. A. sample with 2 mm diameter was measured. The FT-IR spectra were recorded over the range 4000-500 cm^{-1}. Important information about the differences in washing of cotton fabric sample can be provided by the infrared spectral analysis. Characteristic peaks related to the chemical structure of cellulose were shown in Table 4.1. FTIR spectra of original cotton fiber and washing cotton fiber were shown in Figure 4.8.

Table 4.1. Infrared absorption bands of cellulose and other impurities.

Wavenumber (cm^{-1})	Peak characteristics
3400	H-bonded OH stretch
2800	C-H stretching
1740	C=O stretching

Figure 4.8(a) was the spectrum of fabric which showed typical characteristic peaks for pure cellulose. Broad C-H stretching band appears from 2800 cm^{-1} region. Spectrum of the greige fabric in figure These peaks prove the presence of waxes. The other impurities were such as pectins and proteins. The intensities of peaks at 2800 cm^{-1} indicate the amount of waxes remained on the fabric.

Figures 4.8 (b) and (c) were found a new peak appeared at around 1740 cm^{-1}. These are from the protonation of carboxylate groups. If the carboxylate exist in ionized form (COO^-), it would show one peaks at 1740 cm^{-1} for the COO^- ion.

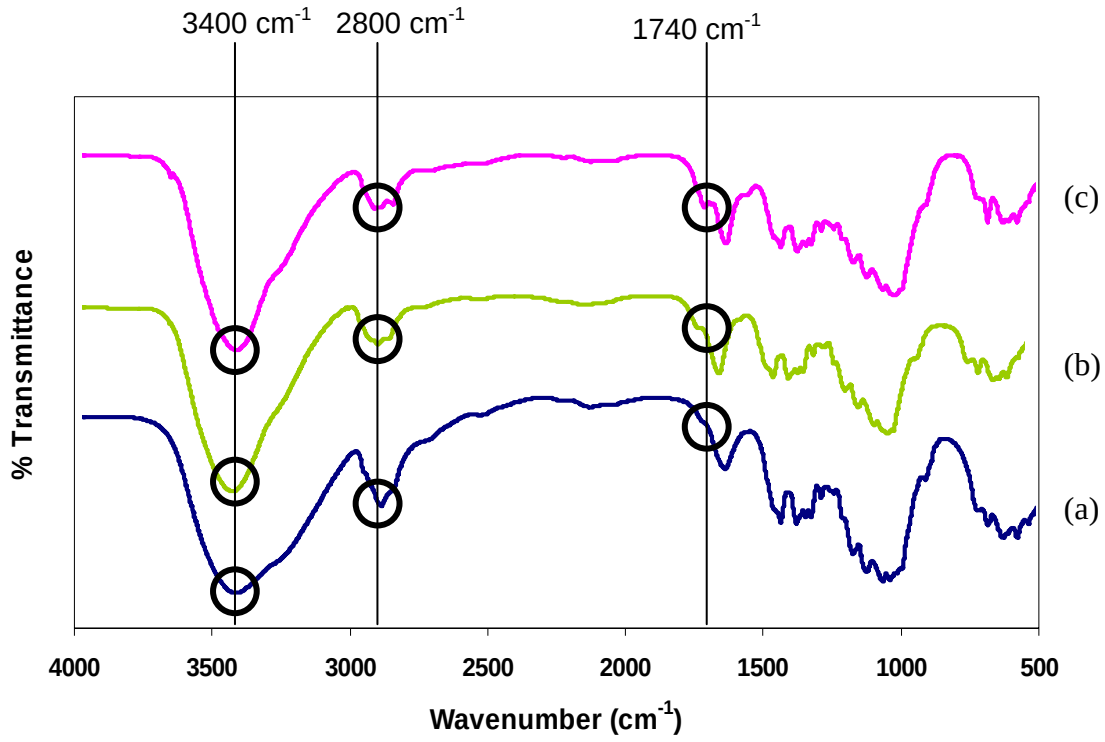


Figure 4.8 FTIR spectra of cotton fabric

(a) Original cotton fabric (b) 2.0 ppm at 20 min and (c) 2.5 ppm at 20 min

4.4 Treatment of fabric in ozone solution system

From figure 4.9, it could be clearly observed that linoleic stain removal could be much improved with the increased ozone concentration and washing time. With the conventional cleaning process using hot water, the percentage of linoleic acid removed by washing with hot water was 51.7 %. Better removal could be achieved by the cleaning processes using 1.5 ppm O_3 for 20 min, 2.0 ppm O_3 for 15, 20 min and 2.5 ppm O_3 for 10,15,20 min. This would be attributed to the fact that aqueous ozone oxidize the linoleic molecule to form lower molecular-weight substance. However, it should be noted that there was some remaining stain in specimens subject to both cleaning methods.

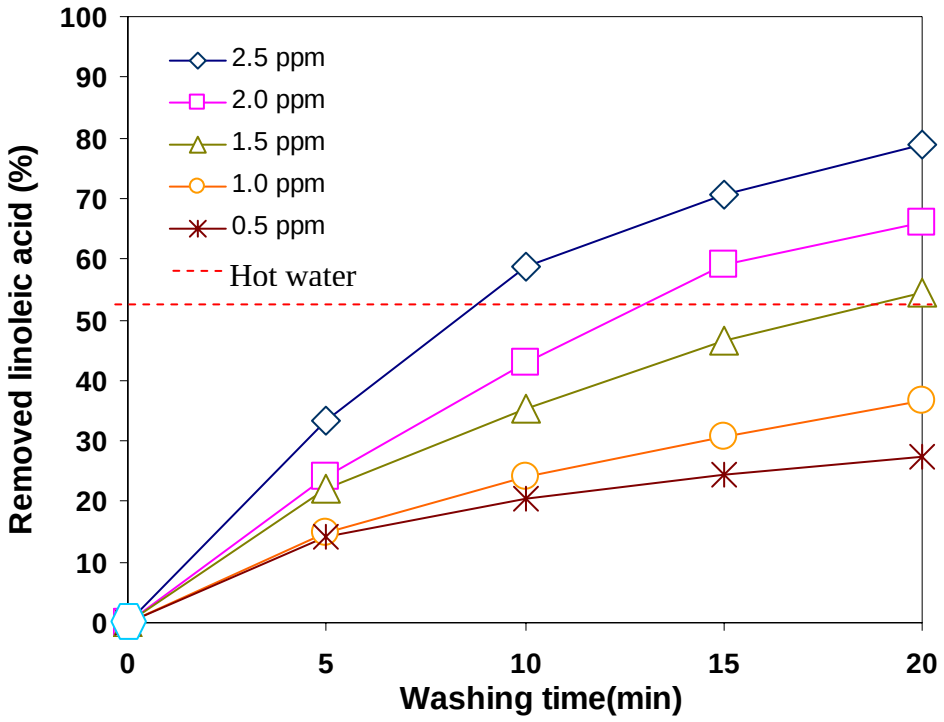


Figure 4.9 Effect of ozone concentration on the percentage of linoleic acid removed.

The phenomena of ozone and stain transfer on the fabric surface would be divided into 3 steps; (a) diffusion of ozone solution to the fabric surface (b) reaction of ozone with stain on the fabric surface and finally (c) diffusion of products from the fabric surface (Figure 4.10).

The main influence of mechanical action would then be restricted to mass transfer. Mass transfer in the washing process is supposed to be the process by which ozone solution is diffused from the solution to the fabric surface.

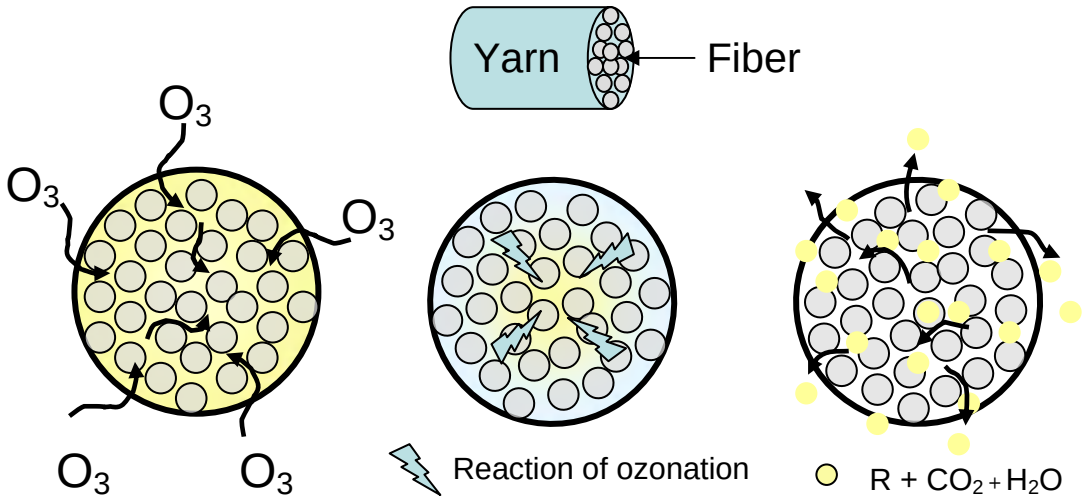


Figure 4.10 The phenomena on the textile surface fabric

The relationship between fabric surface and ozone concentration could be explained by diffusion behavior of solution into the fabric. Diffusion of ozone solution into the fabric was assumed to be steady state. Then the rate of mass transfer of ozone solution was defined by:

$$J = -D\frac{dc}{dz} \qquad 4.5$$

For my work, the concentration gradient was a significant parameter which affected the flux of ozone diffusion into the interface of liquid and fabric. An increase in ozone concentration led to the increased diffusion flux. With larger number of molecule diffusing into the fabric, a higher rate of absorption would occur. It means that, higher ozone concentration gave higher absorption rate of fabric; then the removal of stain would be higher at high ozone concentration than at low vapor concentration.

Products of ozone – linoleic acid reaction are smaller and more polar than linoleic acid molecule [31]. So, it could be dissolved into the water. The ozone solution improved the mechanical removal from the stain from the surface of the fiber. It could be observed that washing by ozone solution could improve the dissolution of the linoleic acid by reducing the ability of the linoleic acid to redeposit onto the cotton surface.

4.5 Physical Properties of cotton fabric

4.5.1 Whiteness Index

The color of cotton fabric could be related to mechanical and chemical damages to cotton fabric and cellulose fiber of cotton fabric. In general, the whiteness index was measured using Macbeth color eye 3100 spectrophotometer.

From Figure 4.11, the whiteness index of the original cotton fabric sample was 84.32 %. In Meanwhile, the whiteness index of cleaned specimens was slightly increased with the increased ozone concentration, resulted by the removal of contamination by ozone solution. The whiteness index of the cotton fabric washed by hot water was 84.48 %. The whiteness index of cotton fabric washed by ozone solution is higher than that hot water. Cleaning with 2.5 ppm O_3 for 20 min could result in the maximal increase in the whiteness index of 6.3%.

Attribution of the increased whiteness index is due to the increase in the carboxyl group content because of the reaction between ozone and cellulose. So the oxidation would eliminate impurity on the fiber surface and form new carboxyl group in the cellulose chains [32,33].

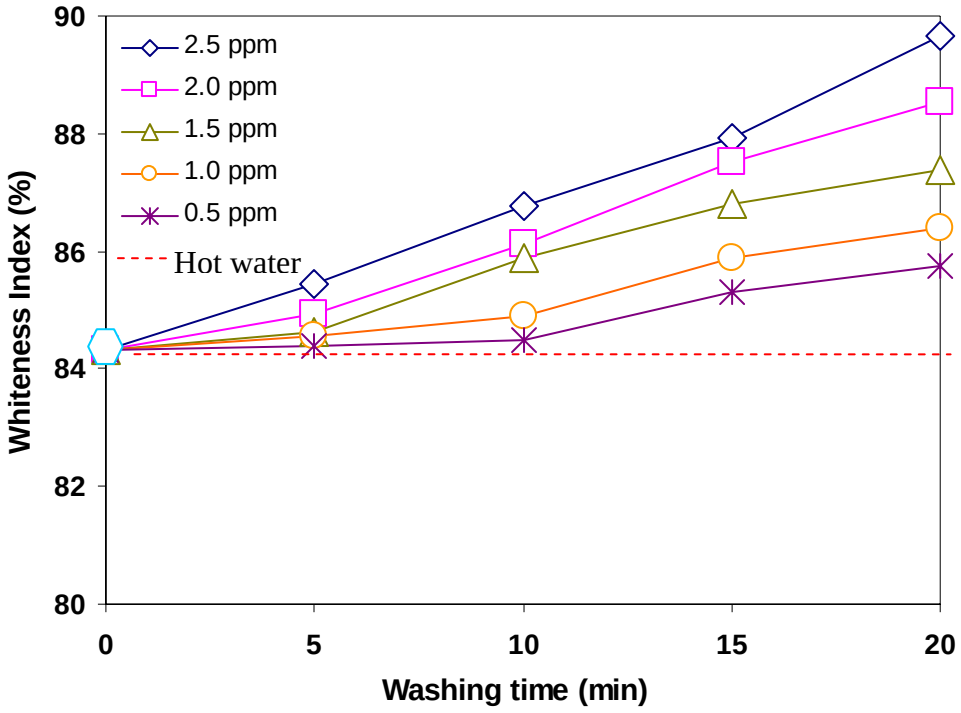
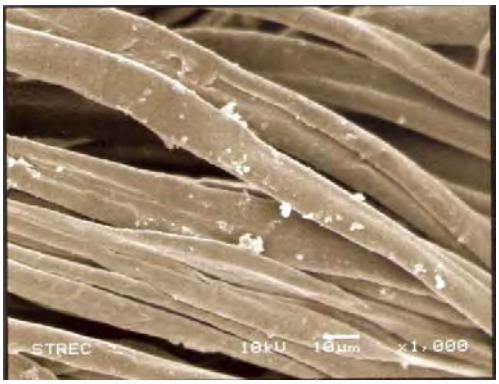


Figure 4.11 Whiteness index changing of cotton fabric versus washing time

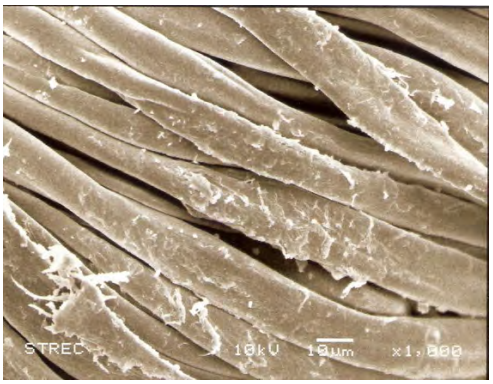
4.5.2 SEM

SEM observation shows the surface of unwashed and washed cotton samples. Figure4.12 (a) shows the surface of unwashed cotton sample, the surface appearance of unwashed cotton fiber could be observed that the washed cotton fiber had a smoother surface. Figure4.12 (b) shows the surface of washed cotton sample with conventional cleaning process (hot water), the surface appearance of cotton fiber was destroyed due to high temperature could affect to swell cotton surface. Figures 4.12 (c) and (d) show the surface of washed cotton sample with 0.5 and 1.0 ppm of ozone concentration respectively, the cotton surface appearance had remained some stain on the fiber. Figures 4.12 (e) (f) and (g) show the surface of washed cotton sample with 1.5 2.0 and 2.0 ppm of ozone concentration respectively, the surface appearance

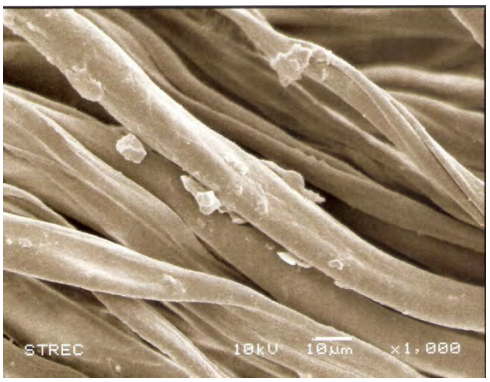
cotton fiber could be observed that the washed cotton fiber had a smoother surface. At Figure 4.12 (g) shows the washed cotton surface with high ozone concentration (2.5 ppm), the cotton fiber had rougher surface. The crack and rougher surface occurred by oxidizing the cellulose fiber with ozone solution.



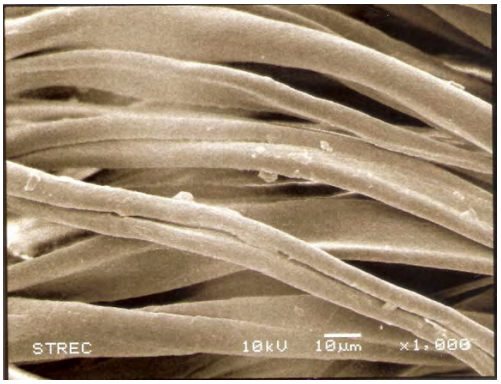
(a)



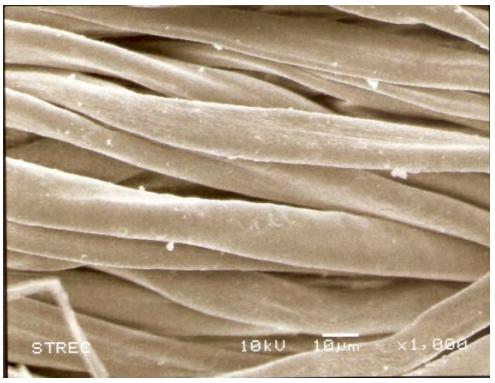
(b)



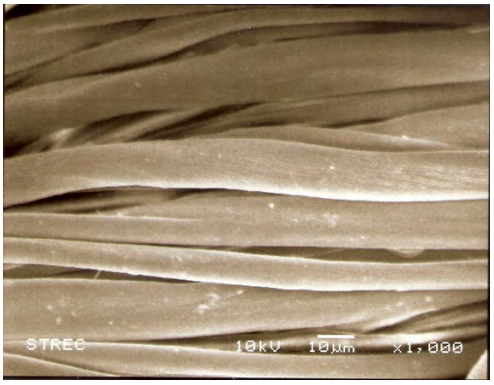
(c)



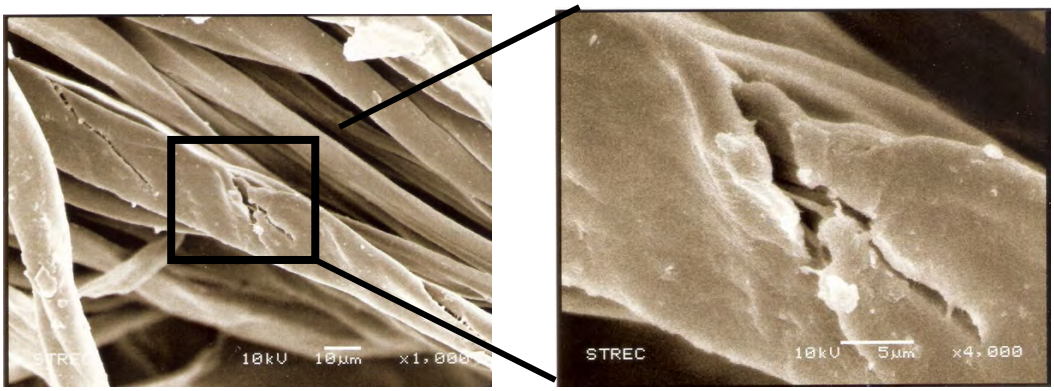
(d)



(e)



(f)



(g)

Figure 4.12 SEM of cotton fiber (a) original Fiber (b) hot water (c) 0.5 ppm (d) 1.0 ppm (e) 1.5 ppm (f) 2.0 ppm (g) 2.5 ppm of ozone concentration

4.5.3 Tensile Testing

The tensile strength, which is an important mechanical property of a fabric, is directly influenced by the cotton fabric degradation. The high tensile strength is the high cotton fabric strength. Meanwhile, the tensile strength of washed specimens was examined using ASTM D 5034 – 95 method, reporting on a maximal force applied on the fabric specimen until its rupture. The high maximal rupturing force had affect to high tensile strength.

From Figure 4.13, the original cotton fabric tensile strength was 1.14 MPa. The increased ozone concentration could lead to a decrease in the tensile strength. When decreased tensile strength could be observed the fabric had decreased cotton fabric strength. This result reveals that the fabrics subject to ozone solution would possibly be damaged by oxidation. However, it is worth noted that hot water washing also increased of damage the fabric due to the irreversible expansion and contraction

of fabric structure before and after subject to hot water under mechanical stain [1]. The cotton fabric tensile strength with hot water process was 1.03 MPa.

The surface of cotton fiber was crack evident in the Figure 4.12. It had affected to the strength of textile. It could be show that the washing ystems by high ozone concentration had affect to cotton surface. Then, the cotton surface had weak the strength of fabric. i.e., the strength of textile decreased when increasing ozone concentration.

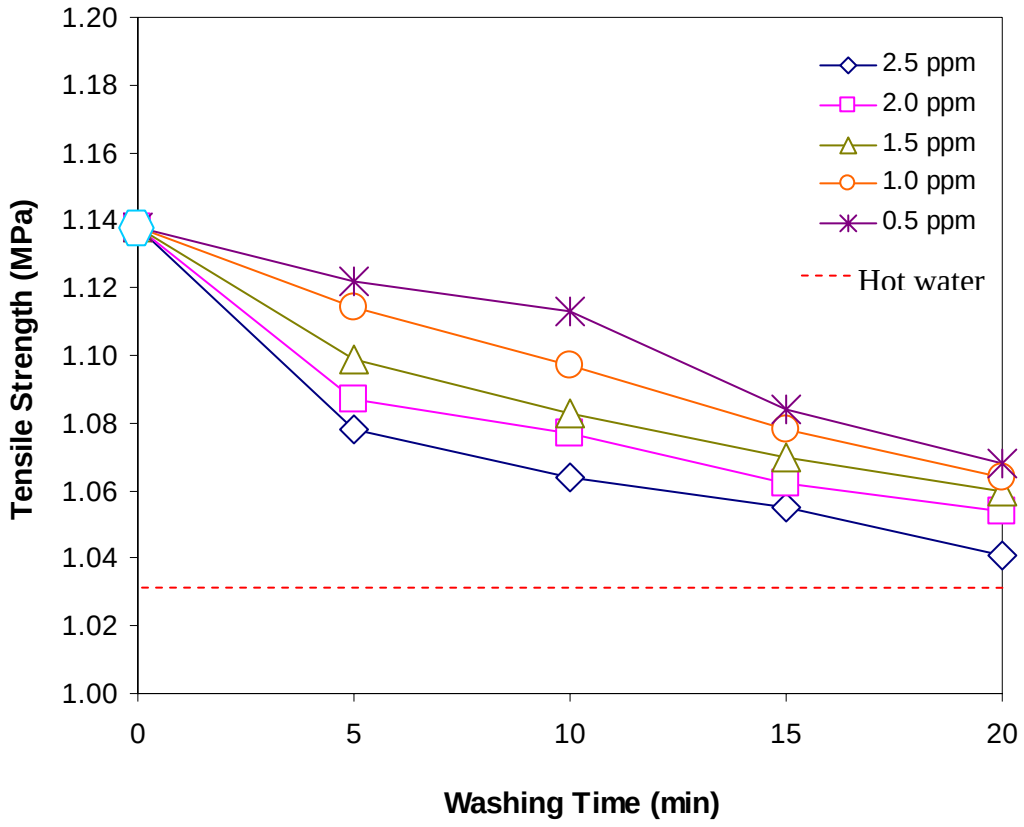


Figure 4.13 Effect of ozone concentration on tensile strength of cotton fabric

4.5.4 Water absorbency

Oily on the fabric was shown hydrophobic property. The effect of hydrophobic of oil, the water has not been absorbed within the fabric. In order to confirm the content of remaining stain on the fabrics, the water absorbency was determined from time spent for water to be absorbed onto the fabric. The higher the water absorbency was the shorter the absorption time. When a fabric is contained with an oily stain, its water absorbency would be worse, resulting in a longer absorption time. Thereby, after the fabric is subject to detergency, its absorption time would become shorter.

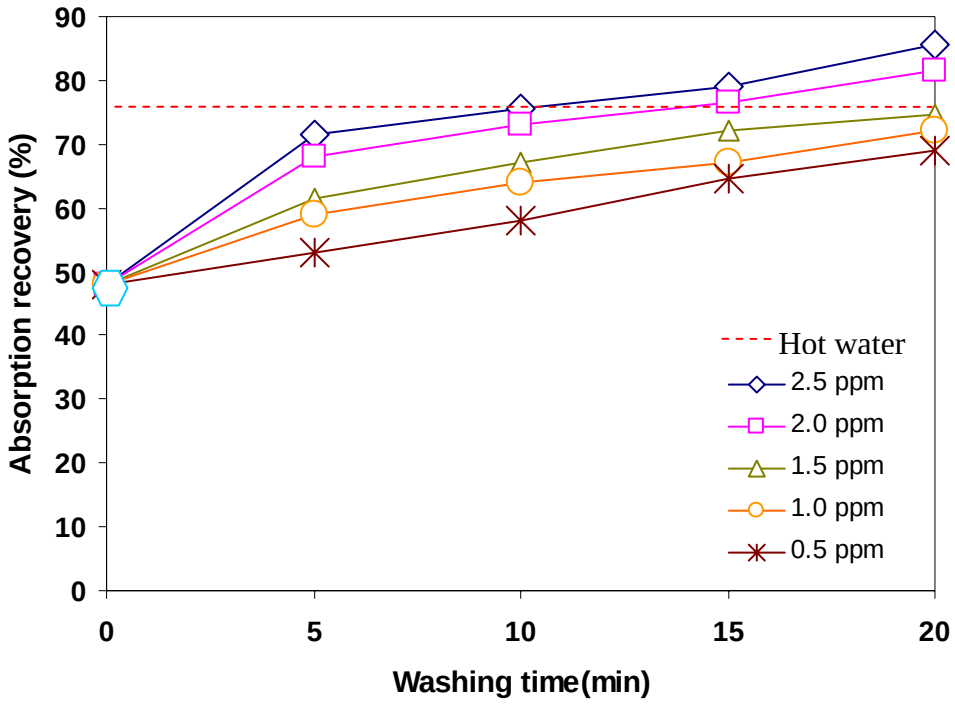


Figure 4.14 Effect of ozone concentration on the recovery factor of water absorbency

In general, the relative value of absorption time difference due to applying of strain onto the fabric is employed for assessing whether the washing process is effective or not. Under the ideal condition, the relative absorption recovery would be 100%. Figure 4.14, the absorption recovery of cotton fabric contain with linoleic acid was 47.89 %. An increase in the ozone concentration and washing time, the absorption recovery factor increased steadily. As a reference, the absorption recovery factor of fabrics subject to hot water washing was 74.80 %. Therefore, the cleaning processes with 1.5 ppm O_3 for 20 min, 2.0 ppm O_3 for 15, 20 min and 2.5 ppm O_3 for 10,15,20 min could provide higher absorption recovery, attributed to higher removal of linoleic strain from the cleaned specimens.

4.5.5 Repeated washing of the textile

Repeated cycles of washing would possibly result in change of fabric properties and they are actually performed in the real life condition. Repeated washing of the fabric, after 30 wash cycles tensile strength and whiteness index shown in Figure 4.15 and 4.16, respectively. It was found when used textile in everyday life, hot water system was short life time than ozone washing system.

The properties of textile after washing with ozone were greater, i.e. more linoleic acid was removed in 2.0 ppm of ozone concentration at 20 min was choose to comparing with hot water system. And the other reason for choosing only this condition of washing is due to the strength of cotton fabric has been strong morn than hot water and other condition.

From Figure 4.15, the increasing number of washing cycles resulted in a significant decrease in the tensile strength. However, it should also be noted that

regardless of number of washing cycles, with hot-water washed, the washed fabrics significantly lost their strength compared with the ozone-washed fabrics. The 30 washed cycles with 2.0 ppm O_3 for 20 min and hot water could result in the decrease tensile strength of 7.27% and 11.93%, respectively.

From Figure 4.16, the increasing number of washing cycles had affect to whiteness index. In cotton fabric washed by hot water, which was decrease the whiteness index when increased cycles of washing. Meanwhile, ozone solution had little affected to the whiteness index of cotton fabric. However, it should also be noted that regardless of number of washing cycles, with hot-water washed, the washed fabrics significantly lost their whiteness index compared with the ozone-washed fabrics. The 30 wash cycles with 2.0 ppm O_3 for 20 min could result in the increase whiteness index of 0.95 % and the 30 wash cycles with hot water could result in the decrease whiteness index of 14.31%.

This result could observe that the washed cotton fabric with ozone washing system could extend the lifetime for using fabric in the real dayflies.

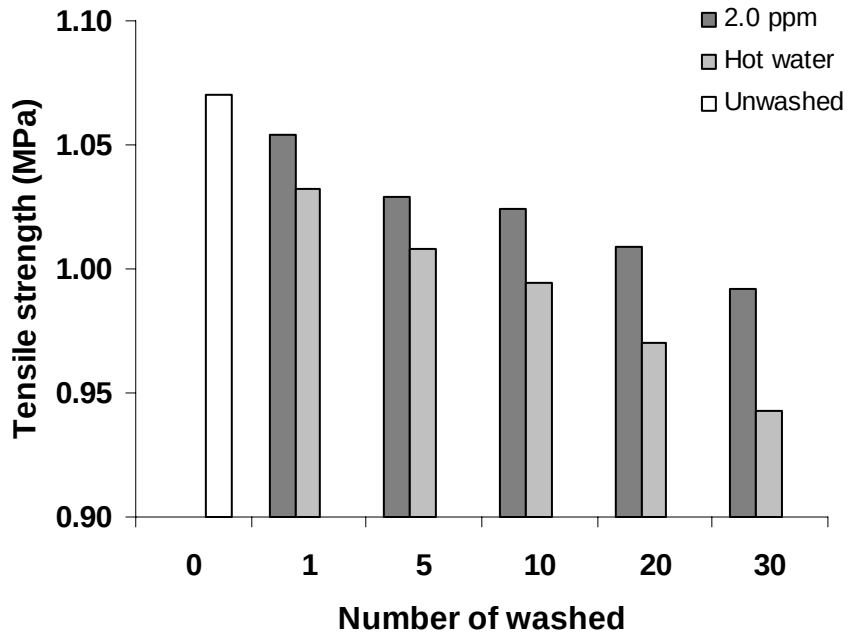


Figure 4.15 Tensile strength with repeated washing cycles

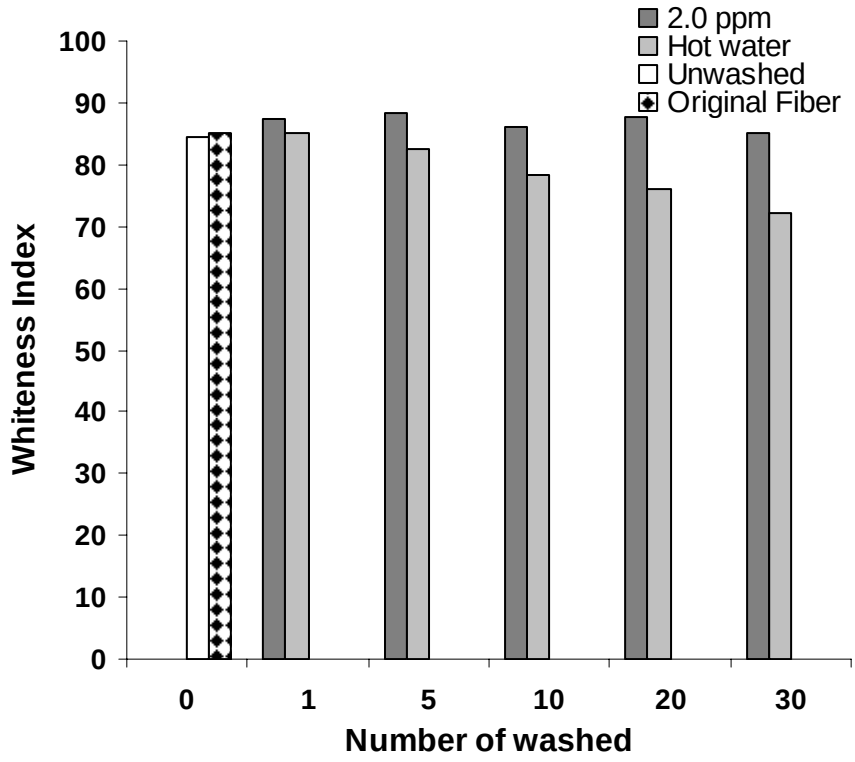


Figure 4.16 Whiteness index of cotton fabric with repeated washing cycles

CHAPTER V

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The conclusions of this research are as follows,

1. The linoleic acid can be removed effectively by ozone solution at high concentration (2.0-2.5 ppm).
2. The oxidation of linoleic acid depended on pH. The fastest oxidation reaction occurred at pH 9 due to the formation of hydroxyl radical.
3. The oxidation of cellulose fiber by ozone could produce carboxyl groups.
4. Ozone washing system could remove stain better the conventional washing system.
5. Ozone solution could increase whiteness index of fabric after washing.
6. The advantages of the ozone washing system were saving of chemicals and extension of service time of cotton fabric when compared with results of the conventional washing system.

5.2 Recommendations

1. To investigate about waste water from washing systems
2. To investigate about effect of ozone on past of washing machine

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APPENDICES

APPENDIX A

A.1 Technical specifications of washing machine (Electrolux EW 660F)

Dimensions	Height	850 mm
	Width	600 mm
	Depth	590 mm
Washing capacity		5.5 kg
Maximun spin speed		550 rpm
Thermostat		Adjustable
Power supply voltage/Frequency		220 V/ 50 Hz
Total power absorbed		2200 W
Minimun fuse protection		10 A

A.2 Technical specifications of ozone generator (OZZON I-8200)

Dimensions	Height	819 mm
	Width	280 mm
	Depth	706 mm
Net Weight		32 kg
Input Water Temperature		5-35°C
Power supply voltage/Frequency		200-240 V / 50- 60 Hz
Power Consumption		400 W
Minimun fuse protection		10 A

APPENDIX B

Conference

From This Research Work

CONFERENCE

International Proceedings

Chotika chalotorn, Apinan Soottitantawat, Yongyuth WorachartsirinoN and Tawatchai Charinpanitkul. “Removal of fabric stain using ozone solution” *Proceedings of international symposium on 12th APCChE Congress*, Aug 4-6,2008, Dalian,China.

Removal of fabric stain using ozone solution

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Textile manufacturing is one of the most important industries. One important issue in this industry is how to clean fabric by maintaining its quality. In this work, the application of ozone solution for removing linoleic stain as a simulated strain from 100% cotton fabric was experimentally investigated. The ozone solution was prepared by electrolysis technique at room temperature. The effects of ozone concentration in the range of 0.5 to 2.5 ppm and contacting time of 5 to 20 min on the properties of ozone treated fabric samples, which are whiteness index, physical appearance and tensile strength, were analyzed by the computerized color matching method (CCM), optical microscopy, scanning electron microscopy (SEM) and tensile strength analyser, respectively. Meanwhile, ozone treating performance was determined by weight difference and water absorbency test. From the experimental results, the increasing ozone concentration and contacting time could provide appreciable removal of the simulated strain with insignificant effect on the fabric strength.

Keyword: Ozone, laundry, electrolysis and fabric quality

INTRODUCTION

One important issue in textile industry is to clean fabric by maintaining its quality. Additionally, in commercial laundry applications by various end-users, such as restaurants, hotels and hospitals, a large quantity of fabric must be carefully cleaned. Although use of commercial laundry systems in larger washing is now available, it is necessary to use hot water in laundry process for antibacterial purpose. However, it is possible that hot water could damage the fabric [1]. Therefore, there are requirements of developing new alternative washing technologies. Fabric is cleaned by many alternative technologies such as liquid carbon dioxide cleaning, ultrasonic washing and cold water with ozone solution washing [2-4]. The methods of washing depend on the type of stain and characteristics of textile.

Ozone has been recognized as a powerful oxidizing agent. It could react with organic and inorganic molecules. In general, fabrics would be contaminated with many kinds of strains, such as vegetable oils, which are found in cooking activities and hard to clean up. Theses issues lead to requirement of detergent applications [5]. Meanwhile, it should be noted that vegetable oils, which are coconut oil, soybean oil and safflower oil, are normally composed of linoleic acid which is polyunsaturated fatty acid [6]. To remove such contamination would strongly rely on application of sufficient use of detergent and mechanical washing effort.

To the best of our knowledge, there is only few investigations reporting on use of ozone in laundry process for effective removal of organic strain. In this work, the effects of experimental parameters which are ozone concentration and washing time on the effectiveness of fabric cleaning would be examined. The optimum condition by ozone cleaning regarding to strain removal performance without providing damage on the fabric would be the focal issue of this work. There results were compared with the conventional hot-water cleaning methods.

2. EXPERIMENTAL

2.1 Fabric preparation

All fabrics used in experiment were 100% cotton. They were scoured by sodium hydroxide (Fluka) at 80°C for 20 min. The fabrics were soaked in linoleic acid (Fluka), which is considered as the simulated stain, for 5 minutes at room temperature. Then the fabric samples would be squeezed by the mangle machine for remove more stain from fabric in prior to testing.

2.2 Washing experiments

The ozone cleaning system consists of a commercial washing machine and an electrolysis ozone generator. First, the soaked fabrics would be washed in the solution of ozone with varied concentrations of 0.5, 1.0, 1.5, 2.0 and 2.5 ppm. Standard ECE detergent is added to wash the fabrics for 5 -20 min. The rising step would be conducted twice for 3 min in each cycle. Finally, the fabrics were dried in an oven for 45 min at 60 ºC. In the conventional cleaning process, specimen would be subject to stirred hot water with a constant temperature of 60 ºC for 12 min and then dried by the same condition.

2.3 Analysis and measurement

The ozone concentration was measured by iodometric method with potassium iodide solution [7]. Washing performance was determined by weight difference and water absorbency test based on Test Method 130 -1995 of AATCC [8]. The change of the color was measured using Macbeth Color Eye 3100 spectrophotometer. The physical microscopic appearances of fabrics were analyzed scanning electron microscopy (SEM). Finally the mechanical properties of fabrics in warp direction were analyzed by tensile testing according to ASTM D 5034 -95 standards by using instron universal testing machine.

3. RESULTS & DISCUSSION

3.1 Effect of ozone concentration on the stain decomposition from fabric

From figure 1, it could be clearly observed that linoleic stain removal could be increased with the increasing ozone concentration and contact time. With the conventional cleaning process using hot water, the percentage of linoleic acid removed by washed with hot water was 51.7 %. Better removal could be achieved by the cleaning processes using 1.5 ppm O_3 for 20 min, 2.0 ppm O_3 for 15, 20 min and 2.5 ppm O_3 for 10,15,20 min. This would be attributed to the fact that aqueous ozone oxidize the linoleic molecule to form lower molecular-weight substance [9]. However, it should be noted that there was some remaining strain in specimens subject to both cleaning methods.

In order to confirm the content of remaining strain on the fabrics, the water absorbency was determined from time spent for water to be absorbed onto the fabric

based on the standard method of AATCC [10]. The higher the water absorbency, the shorter the absorption time. When a fabric is contained with an oily strain, its water absorbency would be worse, resulting in a longer absorption time. Thereby, after the fabric is subject to detergency, its absorption time would become shorter. In general, the relative value of absorption time difference due to applying of strain onto the fabric is employed for assessing whether the cleaning process is effective or not. Under the ideal condition, the relative absorption recovery would be 100%. Figure 2 shows that with an increase in the ozone concentration and washing time, the absorption recovery factor increased steadily. As a reference, the absorption recovery factor of fabrics subject to hot water washing was c.a. 75 %. Therefore, the cleaning processes with 1.5 ppm O_3 for 20 min, 2.0 ppm O_3 for 15, 20 min and 2.5 ppm O_3 for 10,15,20 min could provide higher absorption recovery, attributed to higher removal of linoleic strain from the cleaned specimens.

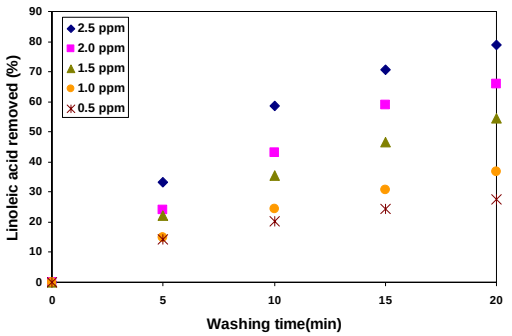

Fig.1 Effect of ozone concentration on the percentage of linoleic acid removed.

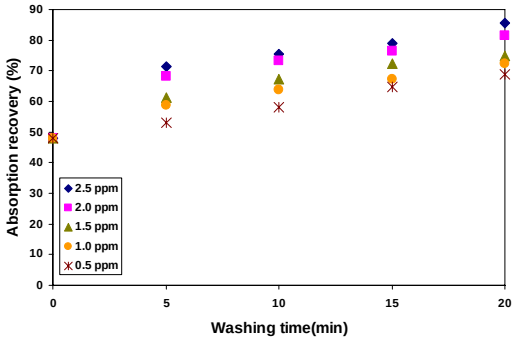

Fig.2 Effect of ozone concentration on the recovery factor of water absorbency

3.2 Effect of ozone concentration on the appearance and strength of fabrics

In the treatment process using ozone, the specimens cleaned by ozone solutions exhibited the whiteness index in the same level compared with that of specimens cleaned by the conventional method. Figure 3, the whiteness index of cleaned specimens was slightly increased with the increasing ozone concentration, resulted by the removal of contamination by oxidation by ozone [11]. Cleaning with 2.5 ppm O_3 for 20 min could result in the maximal increase in the whiteness of 6.3%.

Meanwhile, the tensile strength of washed specimens was examined using ASTM method, reporting on a maximal force applied on the fabric specimen until its rupture. From figure 4, the increasing ozone concentration could lead to a significant decrease in the maximal rupturing force. This result reveals that the fabrics subject to ozone solution would possibly be damaged by oxidation. However, it is worth noted that hot water cleaning also damaged the fabric due to the irreversible expansion and contraction of fabric structure before and after subject to hot water [1]. The average maximal force for rupturing specimens cleaned by hot water washing was 574 N.

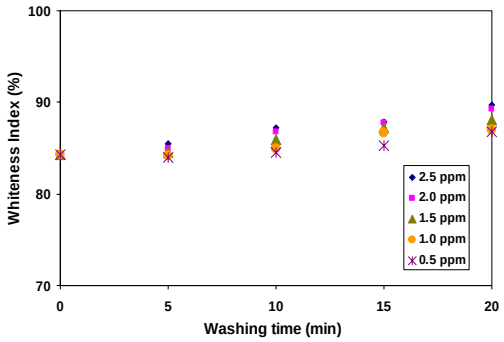

Fig.3 Effect of ozone concentration on the whiteness index of cotton fabric

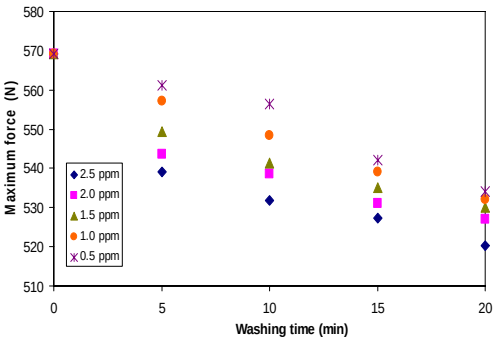

Fig.4 Effect of ozone concentration on tensile testing of cotton fabric

3.3 Effect of multiple cleaning cycle

Multiple cycles of cleaing would possibly result in change of fabric properties and they are actually performed in the real life condition. Based on previous results, conditions of cleaning by 1.5 ppm O_3 for 20 min, 2.0 ppm O_3 for 15, 20 min and 2.5 ppm O_3 for 10,15,20 min were focused for examining the effect of number of cleaning cycle on the strength of cleaned fabrics. From figure 5, the increasing number of cleaning cycles resulted in a significant decrease in the maximal rupturing force. However, it should also be noted that regardless of number of cleaning cycles, with hot-water cleaning, the cleaned fabrics significantly lost their strength compared with the ozone-cleaned fabrics.

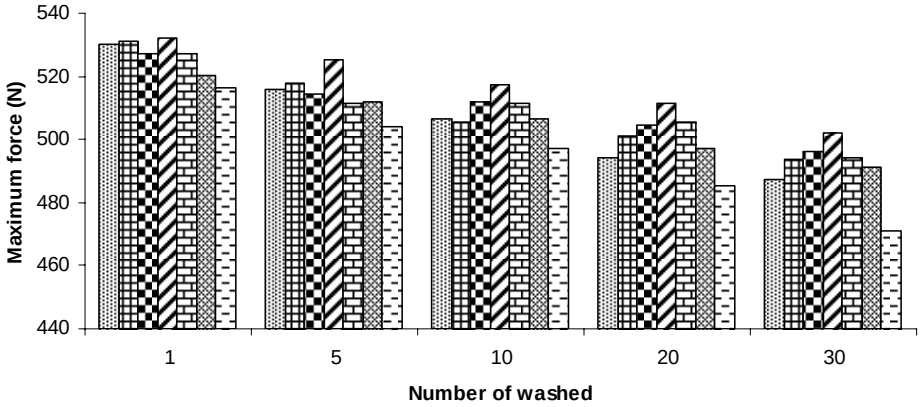

Fig.5 Effect of multiple cycle of washing on the properties of fabrics: 1.5 of O_3 for 20 min (▨), 2.0 ppm of O_3 for 15 min (▦), 2.0 ppm of O_3 for 20 min (▩), 2.5 ppm of O_3 for 10 min (▨), 2.5 ppm of O_3 for 20 min (▤), 2.5 ppm of O_3 for 20 min (▧) and hot water (▤)

CONCLUSION

Based on experimental results, the percentage of stain removes (Wt. %) increased with increasing ozone concentration and contact time. The linoleic acid can be effectively removed by ozone solution of high concentration. While, ozone solution could increase whiteness index of fabric after washing, it provided insignificant damage on the treated fabrics.

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