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FIXATION OF ACTIVATED CARBON POWDER ON KNITTED COTTON FABRICS

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งานวิจัยนี้ได้ทำการศึกษาเกี่ยวกับการนำผงถ่านกัมมันต์ที่ได้จากกะลามะพร้าวมาผนึกติดลงบนผ้าฝ้ายถักด้วยสารช่วยติดชนิดบิวทาไดอีน-อะคริโลไนทริล โคพอลิเมอร์ ด้วยวิธีอัดรีด-อบแห้ง-อบผนึก โดยอัดรีดผงถ่านกัมมันต์ลงบนผ้าก่อน แล้วอัดรีดซ้ำอีกครั้งด้วยสารละลายของสารช่วยยึด การอบผนึกจะทำในช่วงอุณหภูมิ 140 - 170 องศาเซลเซียส ทำการประเมินความสามารถในการดูดซับของผงถ่านด้วยการหาค่าเมทิลีนบลูและเปอร์เซ็นต์ในการดูดซับฟอร์มาลดีไฮด์ และศึกษาสมบัติของผ้าที่ผ่านการตกแต่งในการใช้งานทางด้านสิ่งทอ ได้แก่ ความคงทนต่อการซักล้าง และความคงทนต่อการขัดถู

จากผลการทดลองแสดงให้เห็นว่าผงถ่านกัมมันต์ผนึกติดบนผ้าฝ้ายถักได้ด้วยฟิล์มของสารช่วยยึด การเพิ่มปริมาณของสารช่วยยึด อุณหภูมิที่ใช้ในการอบผนึก และเวลาที่ใช้ในการอบผนึกสามารถช่วยปรับปรุงสมบัติในด้านความคงทนต่อการใช้งาน แต่ประสิทธิภาพในการดูดซับลดลง ซึ่งเห็นได้จากค่าไอโอดีนและเปอร์เซ็นต์ในการดูดซับฟอร์มาลดีไฮด์ที่ลดลง และความสามารถในการดูดซับของผ้าที่ผ่านการตกแต่งนั้นเพิ่มขึ้นเมื่อผ่านกระบวนการซักล้าง ซึ่งเป็นไปได้ว่าฟิล์มของสารช่วยยึดเกิดการแตกและหลุดลอกออกไปบางส่วนขณะที่ทำการซักล้าง จึงทำให้ประสิทธิภาพในการดูดซับของผ้าที่ผ่านการตกแต่งนั้นเพิ่มขึ้น โดยภาวะที่เหมาะสมสำหรับการผนึกผงถ่านกัมมันต์ คือ ปริมาณของสารช่วยยึด 50 กรัมต่อลิตร อุณหภูมิในการอบผนึก 150 องศาเซลเซียส และ เวลาในการอบผนึก 4 นาที และซักล้างหลังจากทำการตกแต่ง

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ลายมือชื่ออาจารย์ที่ปรึกษา
ลายมือชื่ออาจารย์ที่ปรึกษาร่วม

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This research was concerned with the fixation of activated carbon powder from coconut shell on knitted cotton fabrics using butadiene-acrylonitrile copolymer binder. The application was carried out by pad dry cure technique; first padding with activated carbon dispersion and then binder solution. The fixation was performed at the temperature ranging from 140 - 170 degree celsius. The adsorption efficiency was evaluated by methylene blue number and percent formaldehyde absorption. Wash fastness and rubbing fastness as well as properties evaluation of treated fabrics were performed.

The results showed that activated carbon was fixed onto knitted fabric through binder film network. An increase in the amount of binder, fixation temperature and curing time improved the fastness properties of activated carbon but reduced the absorption efficiency of activated carbon, judged by the reductions of iodine number and percent formaldehyde absorption. After washing, the absorption efficiency of fixed activated carbon was recovered. This phenomenon was believed that during washing crack and peel-off of binder film might occur, allowing an increase in the adsorption capacity observed. The optimum conditions for activated carbon fixation were recommended as follows : the amount of binder of 50 gram per liter, fixation temperature of 150 degree celsius and curing time of 4 minute and wash-off process after treatment.

Field of study Petrochemistry and Polymer Science Student's signature Nongnut Sasithorn
Academic year 2005 Advisor's signature T. Vitidsant
Co-advisor's signature Kawee Srikulkit

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CHAPTER I

INTRODUCTION

1.1 Introduction

Activated carbon is an effective absorbent primarily due to its extensive porosity and very large available surface area. The specific properties of an activated carbon are the result of both the raw material used to produce it and the activation process, which boosts its adsorbent qualities.

A variety of raw materials, including wood and coal, are used in activated carbon manufacture, making it plentiful, relatively inexpensive, and versatile. It is generally manufactured as either granular activated carbon (GAC) or powdered activated carbon (PAC). In the most popular granular form, activated carbon is used as a filter medium through which contaminated water or air is passed.

Most forms of activated carbon are non-polar in nature, so they have the greatest affinity for other non-polar substances. As a result, they are most effective in the removal of a variety of organic contaminants, including trihalomethanes, pesticides and herbicides, and polyaromatic hydrocarbons. However, activated carbon may also be used for the removal of trace metals such as cadmium and lead,

Activated carbon applications is used for many applications in industry and household such as the water and wastewater treatment, solvent recovery process, filtration of contaminated air, corn and cane sugar refining, dry cleaning recovery processes, gas adsorption, pharmaceuticals, fat and oil removal, electroplating, alcoholic beverage production, odor absorber in refrigerator and etc.

Thus, the main purpose of this work is to study about fixation of activated carbon powder on fabric to improve odor and moisture absorption property of fabric.

1.2 Objectives

1.2.1 To study feasibility in fixation of activated carbon powder on knitted cotton fabric.

1.2.2 Search for the appropriate condition for fixation of activated carbon and effective of binder to absorption efficiency of activated carbon powder.

1.3 Scope of the Research

The scope of this research work involves the fixation of activated carbon powder on knitted cotton fabric with butadiene-acrylonitrile copolymer binder (Acramin BA-N), employing a pad-dry-cure technique (two baths two steps), then observes the effect of time and temperature in fixation process and effective of binder to absorption efficiency of activated carbon powder. The stepwise investigation was carried out as follows

1.3.1 To do literature survey for related research work.

1.3.2 Find equipment and necessary raw material for using in experiment

1.3.3 Search suitable parameter condition for finishing *e.g.* amount of binder and temperature and time used in fixation process

1.3.4 To determine quality of treated fabric *e.g.* Scanning Electron Microscopy, adsorptive capacity, deodorization behavior, washing fastness, rubbing fastness, water absorbency, stiffness and air permeability of treated fabrics

1.3.5 To summarize the result.

CHAPTER II

THEORY AND LITERATURE REVIEWS

2.1 Activated Carbon

Activated carbon is an amorphous carbon-based material with a honeycomb like porous internal structure. Activated carbon is an effective absorbent primarily due to its extensive porosity and very large available surface area. The chemical nature of the carbon's adsorptive surface is also important but is usually considered much less significant. The specific properties of an activated carbon are the result of both the raw material used to produce it and the activation process, which boosts its adsorbent qualities. [1]

2.1.1 Characteristics of Activated Carbon

The effectiveness of activated carbon as an adsorbent is attributed to its unique properties, including large surface area, a high degree of surface reactivity, universal adsorption effect, and favorable pore size.

Activated carbon is often characterized by its extremely large surface area. In fact, the surface area per gram of material can range from 500 to 1,400 square meters, and values as high as 2,500 m^2/g have been reported. The complex internal surface area is usually divided into three components. Channels and pores with diameters less than 2 nm are commonly known as micropores; these micropores generally contain the largest portion of the carbon's surface area. On the other hand, pores with diameters between 2 and 50 nm are known as mesopores, and pores with diameters greater than 50 nm are defined as macropores. [1]

For comparison, a given type or sample of activated carbon is usually quantified based on four primary criteria: total surface area, carbon density, particle size distribution, and adsorptive capacity. Of course, all of these factors influence adsorption rate and capacity.

2.1.1.1 Total Surface Area

Total Surface Area is measured by the adsorption of nitrogen gas onto the carbon and is expressed in square meters of surface area per gram of carbon. Because the gas molecules used to measure adsorption are very small, it should be noted that this measurement of surface area may be misleading when considering the adsorptive capacity of a carbon for large organic macromolecules. Those types of compounds may have adsorption limited by pore size considerations.

2.1.1.2 Carbon Density

Carbon density is the weight of one milliliter of carbon in air. Bulk density is also sometimes used for carbon as it is for soil, and is expressed in pounds per cubic foot or in kilograms per liter. Particle sizes in carbons are measured using standard U.S. sieve sizes, as for soil constituents.

2.1.1.3 Particle Size Distributions

Particle size distributions are important in carbon systems because they influence handling of the activated carbon material. For example, in granular carbon, the particle size affects hydraulic loading and backwash rates for a filter. On the other hand, particle size is often important because of its effect on adsorption rates as well.

2.1.1.4 Adsorptive Capacity

Adsorptive capacity is characterized by the effectiveness of activated carbon in removing a given contaminant. For comparison, several standard compounds are used for these measurements. For example, the commonly-used iodine number describes the carbon’s capacity to adsorb low-molecular-weight substances, while the molasses number characterizes a carbon’s capacity for more complex compounds. [2]

2.1.2 Sorption Theory

Some initial definitions are necessary for the presentation of the various processes involved in the removal of contaminants from water by sorption onto the solid phase. The general term sorption includes adsorption, the process by which a solute clings to a solid surface, and absorption, the process by which the solute diffuses into a porous solid and clings to interior surfaces, according to Fetter. When the attachment of the solute is accomplished by means of a chemical reaction with the solid, the process is called chemisorption. Some authors also include cation exchange as an adsorption process, but this is usually considered to be a minor mechanism in activated carbon sorption processes, so it won't be considered here. These distinctions between types of sorption are subtle, but they are useful in understanding the unusual nature of activated carbon. Many materials can be used as sorbents, but activated carbon is so effective primarily because of its sorbent properties. The total process of a solute diffusing through a carbon particle to attach to an inner surface is most accurately referred to as absorption, though the actual attachment of the solute at the site is actually adsorption. For carbon the sorption process is considered primarily physical rather than chemical. It should be noted, however, that the term adsorption seems to be used rather freely to refer to sorption processes in general.

While the physical and chemical mechanisms by which compounds become attached to activated carbon are still not fully understood, the process is generally thought to be physical rather than chemical. It is commonly described by Adsorption Potential Theory. This theory was developed by Polanyi to describe vapor-phase adsorption, and was later modified by Manes and others to describe adsorption from solution; Wagner and Jula expanded on its applications to activated carbon. This theory of adsorption explains the attachment of solute molecules to the adsorbate surface by weak electrical forces known as Van der Waals bonds. Because the attachment bonds are physical rather than chemical, attachment can occur over multiple layers in a field near the adsorbent surface. Of course, adsorption is concentrated at the higher-energy sites on the surface. Wagner relates the attachment of molecules at these sites to the gradient of atmospheric pressure in a gravitational field, explaining that the highest concentration of solute is found right at the surface, and the concentration decreases toward the bulk of the solution. With this theory in mind, the process of adsorption becomes much more clear. Activation of carbon essentially increases the energy at the carbon surface, giving the surface much higher attraction for molecules in solution. [4]

The rate of at which sorption occurs can be very important. Perrich defines the rate of adsorption as the rate at which substances are removed from the liquid phase (adsorbate) to the solid phase (adsorbent). He describes three main processes which control the adsorption rate: diffusion of the solute through the layer of fluid surrounding the adsorbent particle, diffusion of the solute across the surface of the adsorbent, and adsorption of the solute onto the internal surfaces of the adsorbent pores.

For most purposes, the adsorption of the solute molecule at the site on the internal surface occurs almost instantaneously, so it has little effect on the rate of the overall reaction. The transfer of solute from the bulk liquid to the surface layer of fluid around a particle can occur rather slowly, but in most treatment systems, this is encouraged by the constant movement of fluid past the surface. On the other hand, the diffusion of the solute through or across the surface of the adsorbent may occur rather slowly, and this diffusion usually limits the rate of the sorption.

Because the rate of adsorption is usually limited by diffusion, it is influenced by the same variables which affect diffusion rate. The concentration gradient of the solute across the surface of the adsorbent has a large effect on the rate, as does the temperature of the system.

Adsorption is usually modeled by isotherms which relate the relative concentrations of solute adsorbed to the solid and in solution. The most commonly used isotherm is the most familiar Freundlich isotherm, which characterized most adsorption processes rather well under conditions present in most water treatment systems. One problem with this isotherm, however, is its failure to consider that there may be a limited amount of adsorption sites on the surface of the solid; the lack of further sites may prevent adsorption under conditions in which equilibrium is reached. The Langmuir isotherm was developed to represent this site limitation, and it does a better job at describing adsorption under some conditions, particularly as the solid becomes saturated and solute molecules have trouble finding adsorption sites. Finally, the Redlich-Peterson isotherm, a more recent development, incorporates some refinements which make it a better model than either of the other two. Unfortunately, this isotherm is rarely used because it requires the use of several constants, and one of the other isotherms usually approximates a particular adsorption system with a good enough degree of accuracy.

2.1.3 Sorption with Activated Carbon

The difficulty in accurately predicting adsorption in a given system is difficult, even assuming that only one solute is present. Predicting adsorption onto a complex activated carbon surface from an aqueous solution with multiple solutes becomes exceedingly more difficult. Nonetheless, some general knowledge about adsorption theory, along with experience, has allowed us to develop some general rules about carbon adsorption.

When considering these factors, two properties of the adsorption system are of interest: the adsorptive capacity of a given amount of carbon for a particular solute and the adsorption rate at which that solute is taken out of solution. Obviously, these system properties are affected by characteristics of both the carbon and the solute.

2.1.3.1 Factors Affecting Adsorption Capacity

Factors affecting adsorption capacity is surface area of activated carbon, pore size of carbon, solubility of solute in aqueous solution, pH and Temperature. Generally, straight-chain organic compounds are less adsorbable than branched-chain organics, while the type of attached functional group has been shown to affect adsorption. These phenomena are attributed to the influence of the molecular structure on the polarity and/or solubility of the compound of interest. Several authors have documented the fact that large molecules (with more adsorption sites) tend to be more sorbable than small molecules, but most also document cases in which large molecular size inhibits adsorption. This occurs when the molecular size approaches the size of the smaller carbon pores, and the pores screen out large numbers of molecules which might otherwise be strongly adsorbed. This occurrence is often referred to as the molecular sieve effect.

It is generally agreed that a solute’s aqueous solubility is inversely related to its adsorption onto carbon, and this conclusion is borne out by a comparison of data on individual solutes. Because adsorption reactions are usually exothermic, high temperatures would seem to inhibit or slow adsorption, but this is not usually found to be a factor in most systems. An explanation for this may be revealed by again considering the rate-limiting factor for adsorption. In carbon, adsorption is limited primarily by the diffusion of solute into the carbon particle. Higher temperatures may impede adsorption at the adsorption site, but they significantly speed up the pace of diffusion, offsetting any negative temperature effect.

2.1.3.2 Factors Affecting Adsorption Rate

Factors affecting adsorption rate are size of activated carbon particle, concentration of solute in solution and temperature of solution. Because adsorption rate is limited by diffusion, variables that influence diffusion have a significant effect on adsorption rate. For example, a higher concentration gradient across the surface of the carbon particle will increase the rate of adsorption, though a high concentration of solute can eventually have a negative effect on adsorptive capacity. In practice, the primary carbon variable influencing adsorption rate is the size of the carbon particle. Smaller carbon particles have a greater ratio of surface area to volume, making them more accessible to diffusion from solution.

Notably, the size of the particle has little effect on adsorptive capacity as expressed by total surface area. While it is well known that smaller particles of a given mass of carbon would have a greater surface area than larger particles, the huge internal surface area of carbon dominates the calculation so much that the particle size essentially has no effect on capacity.

2.1.4 Removal of Organic Contaminants by Activated Carbon Adsorption

Though activated carbon is effective at adsorbing many types of chemicals, it is especially known for its power in adsorbing organic compounds. As discussed previously, carbon’s particular affinity for organics is due to its non-polar nature. In fact, the effectiveness of activated carbon in adsorbing an organic compound is inversely related to the compound's solubility in water, which is itself a function of the compounds’ polarity.

It is also apparent that larger molecules tend to adsorb more strongly on activated carbon than smaller ones. For example, Methyl phenol is adsorbed more strongly than Phenol, and Dimethyl phenol is adsorbed even more strongly. The additions of these functional groups to the molecule decrease the molecules polarity, thereby also decreasing its solubility. Interestingly, the location of the group on the molecule does not affect the adsorption, but the number of groups seems to have a large effect. The lower adsorption of larger molecules has also been attributed simply to molecular size, because larger molecules present a larger surface for the intermolecular attractions known as Van der Waals forces. [1]

Of course, activated carbon does not effectively adsorb all organic chemicals. Predictably, the less adsorbed classes of organics include the more soluble ones: alcohols and organic acids.

Quantitative characterization of adsorption capacity is difficult because of the number of variables which seem to affect adsorption. For example, even one particular organic compound, such as benzene, is likely to adsorb somewhat differently onto coconut carbon than onto wood carbon. As a result, carrying out lab trials and pilot tests using the specific waste to be treated, and perhaps several different types of activated carbon, is considered the only effective way to design treatment systems for organic chemicals.

2.1.5 Removal of Inorganic Contaminants by Activated Carbon Adsorption

While activated carbon is especially known for its effectiveness in removing organic chemicals from water and waste water, it is also surprisingly effective in removing a variety of inorganic pollutants. Though the effective adsorption of inorganic constituents would not necessarily be predicted by the chemistry of activated carbon, low levels of these chemicals can be removed effectively, primarily due to physical adsorption mechanisms. Applications for this process can be found in water and wastewater engineering, metallurgy, and analytical chemistry.

Both anions and cations have been removed from waters with activated carbon. Researchers have found that carbon exhibits preferential adsorption for ionic species, and an order of preference has been defined: $H^+ > AL^{+3} > Ca^{+2} > Li^+ > Na^+ > K^+$. For anions, the NO^{3-} ion is preferred over the Cl^- ion. It is not surprising that pH plays an important role in the sorption characteristics of these ions, because the low pH provides large quantities of the preferred H^+ ion, which may take the space of other ions on potential adsorption sites.

Even without pH variations, inorganic compounds exhibit a wide range of adsorbability on carbon. For example, dissolved salts like potassium chloride and sodium sulfate are almost nonsorbable, while iodine is one of the most absorbable substances known. While some authors claim that significant reductions in inorganic compounds are not expected in carbon adsorption systems, others point out cases in which effective inorganic adsorption is accomplished. It should be noted that such adsorption is accomplished only at very low concentrations of these ions and compounds. For example, carbon drinking water filters are often used to bring lead concentrations down from the range of 100 ppb to below the action level of 15 ppb.

In general, it is true that carbon adsorption is not nearly as effective at removing metals and inorganics as it is at removing organic compounds. This is primarily because metals often exist in solution either as ions or as hydrous ionic complexes. Based on previous discussions of adsorption chemistry, neither of these forms are effectively adsorbed by carbon.

The following metals are often removed from industrial wastewaters via activated carbon adsorption. It is notable that most of these metals can be toxic at fairly low concentrations, and carbon can accomplish the type of finishing treatment often necessary with waste effluents containing them.

2.1.6 Application of Activated Carbon

Activated carbon has been historically used for removal of odor, removal of color pigments and various catalytic functions. However, applications of activated carbon have increased significantly in the recent years with the advancement of activated carbon process capability. The applications can be classified into two categories: gas phase and liquid phase.

Table 2.1 Application of Activated Carbon

States	**Purpose**	**Applications**
Gas Phase	Recovery	Solvent Recovery
		Gasoline Vapor Recovery
	Odor Removal	Room Order Removal
		Refrigerator
		Automobile
		Hospital
		Ozone Removal
		Tobacco
	Harmful Gas	Closed Environment
	Gas Separation	Nitrogen PSA
Liquid Phase	Water Treatment	Factory Waste Water
		Drinking Water Treatment
	Decolorization of Indusrial Chemicals	Industrial Use
	Medical Applications	Medical and Nursing
	Electronics	Eletrorodes
	Mineral Recovery	Gold Recovery

2.2 Binders [7]

Binders is an organic polymer products used for the fixation of pigment printing and dyeing and as adhesive bonding agents for web consolidation in the manufacture of stable composites and nonwovens, They are mainly solid plastic products (*e.g.* thermoplast powders), plastic solutions and dispersions. Binder additives based on N-methylol compounds, fatty acid derivatives and polyethers, etc. and have binder regular and/or modifying functions, as for the achievement of elastic resilience, flexibility, absorbency, wet fastness, etc.

An important requirement for binders is the balance between adhesion and cohesion. The adhesion of binder film/substrate should be as high as possible with good binder cohesion at the same time. If adhesion is too weak, unacceptable marking of the prints occurs on rubbing. If cohesion is too weak, the pigment particles are detached from the binder film relatively easily on rubbing.

2.2.1 Classification of Binders [8]

Due to their diversity, binders may be classified into several categories based on polymer chemical structure, functionality and the type of curing reactions.

2.2.1.1 Classification Based on Chemical Structure

There are three main kinds of binders : butadiene copolymers, acrylates, and vinyl copolymers. The chemical compositions influence T_g, hardness and softness, hydrophobicity and hydrophilicity, elasticity, aging, and dry tensile strength of binders. The higher the T_g, the higher will be the dry tensile strength of binders.

a) Butadiene Copolymers : The butadiene polymers are cross-linked by polysulphides, and their properties are modified by different copolymers. The butadiene monomers provide elasticity while styrene and acrylonitrile monomers give tensile strength, and oil and solvent resistance, respectively. Their disadvantages are oxidation and discoloration due to residual double bonds in their polymer chains.

b) Acrylic acid Derivatives : Acrylic binders are the most widely used and versatile binders available with various modifications. The properties of acrylic binders differ according to their derivatives and copolymers. They are frequently copolymerized with styrene, acrylonitrile, vinyl chloride or vinyl acetate, depending on the desirable properties. Some of these properties are hardness from styrene, solvent resistance from acrylonitrile, flame retardancy from vinyl chloride, and cost benefits from vinyl acetate.

c) Vinyl Copolymers : There are two main binders for vinyl copolymers as vinyl chloride and vinyl acetate. Since the vinyl binders are stiff, they are plasticized externally or internally. As internal plasticizers, ethylene and acrylate monomers are used, and external plasticizers consist of vinyl chloride. Due to its low T_g, vinyl acetate is not that stiff, and its advantage is low cost. The chlorides cause yellowing problems. The chemical structures are closely related T_g and stiffness of binders.

2.2.1.2 Classification Based on Functionality

The functionality of binders is in the functional groups attached to polymer chains, which influences wet and solvent properties. To modify binder properties, copolymerization with a small amount of monomers with special functionality is performed. The main functionalities in binders are carboxyl and amide side chains.

a) Carboxyl Functionality : This functionality is related to binders containing acrylic acid or methacrylic acid by copolymerization. The binders are crosslinkable since the functional group, carboxylic acid, provides sites for crosslinking reactions.

b) Amide Functionality : This functionality is related to binders containing acrylamide by copolymerization. The amide functionality provides crosslinking sites, and even the binders are self-crosslinkable.

c) N-metylol amide (NMA) Functionality : This functionality is obtained after acrylamide is reacted with formaldehyde. The binders containing the substituted acrylamide groups have self-crosslinking properties.

2.2.1.1 Classification Based on Type of Curing Reactions

The classification of reactivity refers to crosslinkability of binders, which is related to reaction with curing resins, crosslinking agents. The most common curing resin is melamine formaldehyde condensate resin involving reaction of n-methylol groups.

a) Non-crosslinkable Polymers : The polymers do not contain any of the functional groups. They cannot crosslink, even with external curing resins.

b) Crosslinkable Polymers : The polymers contain acid or amide functional groups. They can react with added curing resins, but the degree of crosslinking is limited.

c) Self-crosslinking Polymers : The polymers contain n-methylol functional groups. They can react with themselves, and a high crosslink density can be obtained by adding curing resins.

2.2.2 Types of Binders

The binder properties can be modified considerably by the presence of co-monomers.

2.2.2.1 Acrylic

These binders offer the greatest durability, color stability, and dry/wet performance. Acrylic binders have the widest range of fabric hand properties. They can be formulated to vary from very soft (T_g = - 40 ^{O}C) to extremely hard (T_g = 105 ^{O}C). These binders can be used in virtually textile applications, although they tend to be more costly. These polymers can be made to cross-link, with substantial improvement in durability.

2.2.2.2 Styrenated Acrylics

These are tough, hydrophobic binders. The resulting textile hand ranges from soft-to-firm (T_g varies from - 20 ^{O}C to +105 ^{O}C). These binders can be used in applications where there is a need for some wet strength without crosslinking. The use of this type of binder does involve some sacrifice in UV and solvent resistance.

2.2.2.3 Vinyl Acetate

The vinyl acetate binders are firm (T_g = +30 ^{O}C to +40 ^{O}C); however, they are relatively low cost and find extensive use. They offer good dry strength and toughness, but are somewhat hydrophilic and have a tendency to yellow when subjected to heat.

2.2.2.4 Vinyl Acrylics

These binders are more hydrophobic than the straight Vinyl Acetate binders. They provide excellent toughness, flexibility, and better color stability. They are the compromise between Vinyl Acetate and acrylic, and can compete on a cost/performance basis. The hand range is limited to intermediate softness (T_g = -10 ^{O}C) to a firm hand (T_g = +30 ^{O}C).

2.2.2.5 Ethylene Vinyl Acetate

These binders have a (T$_g$ range of - 20 ^{O}C to +115 ^{O}C, which is equivalent to soft ranging to an intermediate textile hand. They exhibit high wet strength, coupled with excellent absorbency. In general, they are less costly than acrylics. They do have a tendency to have more of an odor compared to other binders. They are used primarily in wipes, air-laid pulp fabrics and similar applications.

2.2.2.6 Styrene-Butadiene (Styrene Butadiene Rubber)

These binders have an excellent combination of flexibility and toughness. They range in hardness from very soft (T$_g$ = -30 ^{O}C) to very firm (T$_g$ = +80 ^{O}C). However, the T$_g$ of an Styrene-Butadiene binder is not strictly comparable to other classes of binders. The styrene-to-butadiene ratio (S/B ratio) is the most common method for describing the relative hand resulting from the use of these binders. When cross-linked, this class of binder is very hydrophobic and durable. They are affected somewhat by heat and light because of their tendency to oxidize.

2.2.2.7 Polyvinyl Chloride

The homopolymer of polyvinyl chloride is a very hard, rigid polymer (T$_g$ = +80 ^{O}C). This polymer must be plasticized to provide flexibility and film-forming properties. Normally, the Polyvinyl Chloride binders used in nonwovens are softened internally by co-polymerizing the vinyl chloride or with softer acrylic monomers. The hand range of most of these polymers is still relatively firm (T$_g$ is greater than the +30 ^{O}C). Because this type of polymer is a thermoplastic, it performs well in heat and dielectric sealing applications. This can be an advantage in some uses. The chlorine content of the polymer promotes flame retardency. This feature is one of the primary benefits of utilizing this type of binder. However, the chlorine also conveys the tendency to yellow upon heat aging, due to elimination of hydrogen chloride from the polymer.

2.2.2.8 Ethylene/Vinyl Chloride

Binders in this class have a slightly broader hand range (T$_g$ = 0 ^{O}C to +30 ^{O}C) without the external plasticization required of Polyvinyl Chloride binders. The presence of the chlorine again conveys some flame retardancy. These binders exhibit good acid resistance, fair water resistance, and excellent adhesion to synthetic fibers. There is some tendency to yellow upon aging. In essence, this is an internally plasticized Polyvinyl Chloride binder, considering the ethylene monomer to be the softener.

2.2.3 Fixation Process for Binders

Binders for textile pigment printing/dyeing contain self-crosslinking free methylol groups which are protected against premature crosslinking by esterification. During drying, film formation of the binder occurs in 2 stages. In the first stage, water and emulsifier are removed from the binder in drying process. the pigment print by evaporation and capillary action of the fiber whereby the dispersed materials (binder, pigment) coagulate to form an unstable gel layer. In the second stage, the gel layer merges to form a film which, from this point on, bonds (even though loosely) with the textile material. Only after subsequent crosslinking (fixation) does the binder film develop its full adhesion to the substrate and its elastic extensibility. The crosslinking reaction proceeds under acidic conditions (pH <4) by elimination of water and alcohol to form a three-dimensional structure. The best fixation medium is hot air treatment at e.g. 150 ^{O}C for 5 min. (or at 175 ^{O}C for 45- 60 sec). Wet steam is not suitable as a fixation medium. Fixation in superheated steam is also less suitable than hot air since only a 60 - 80% fixation yield is achieved in this case due to partial hydrolysis (High temperature steam consists exclusively of water vapor).

An additional crosslinking of the binder film is achieved with the aid of so called external crosslinkers of the esterified melamine type in accordance with the same (condensation) principle. Wet fastness properties, especially on synthetic fibers and their blends with cellulosic fibers, are improved by this further crosslinking although it is accompanied by an increased stiffening of the prints. Due to this undesirable effect on handle, the quantity of crosslinker applied should be limited only to that amount which is absolutely necessary. The binder film, in which the pigment particles are embedded, has a layer thickness of only 8 - 10 µm., a dimension which is not visible to the human eye.

2.3 Textile Finishing [11]

Textile finishing usually includes treatments such as scouring, bleaching, dyeing and/or printing, the final mechanical or chemical finishing operations, that during this stage are carried out on textile products (staple, sliver or top, yarns or filaments, woven or knitted fabrics) to enhance their basic characteristics like dye penetration, printability, wet ability, color, hand, and appearance.

By textile finishing, also mean the last manufacturing step in the production of textile fabrics that, though included in the so called finishing stage, are generally applied to the fabrics to improve the final properties of fabric. Finishing is an integral part of wet processing, but not restricted to wet processing alone. Finish can be either chemicals that change the fabric's aesthetic and/or physical properties or changes in texture or surface characteristics brought about by physically manipulating the fabric with mechanical devices. It can also be a combination of the two.

Finishing is commonly divided into two categories, Chemical and Mechanical**.** In chemical finishing, water is used as the medium for applying the chemicals. Heat is used to drive off the water and to activate the chemicals. Mechanical finishing is considered a dry operation even though moisture and chemicals are often needed to successfully process the fabric.

2.3.1 Classification of Finishing Operations

Finishing operations can be carried out by means of discontinuous, continuous and semi-continuous systems.

2.3.1.1 Discontinuous or Batch-type Systems

All the operations are carried out on a single machine; it is therefore necessary to load the machine, carry out the treatments following a predetermined cycle, unload the machine and finally wash it thoroughly before starting a new cycle. This working process is extremely flexible and is suitable for processing small lots: for example, it is possible to a carry out a scouring treatment on a single machine, then a bleaching one followed by a dyeing process. For the production of large lots, the discontinuous process is labor-intensive, *i.e.* it requires many operators to load and unload the material; it also entails long processing times and results that can vary from one batch to another.

2.3.1.2 Continuous Systems

The operations are carried out by means of a series of machines; every machine carries out always and solely the same process. Every machine is assembled according to specific production requirements. A system like this entails high start-up costs and a complex setup but once the system has started, it requires a smaller staff and grants excellent repeatability and high output rates; continuous systems are therefore suitable for manufacturing large lots of products with the highest cost-efficiency.

2.3.1.3 Semi-continuous Systems

In these mixed systems several operations are carried out with both continuous and discontinuous machines. For example, a continuous pad-batch machine is used to wet the fabric and a discontinuous system is then used for other treatments. These mixed systems are suitable for processing small and medium lots; they require reasonable start-up costs and grant quite good reproducibility.

2.3.2 Application of the Fnish

The operations to be carried out when applying the finish to a textile substrate are mostly conditioned by the structural and hygroscopic properties of the material to be processed, by the desired effects, by the physical and chemical nature of the elements that make up the finishing substance and by the machines output rate. In textile finishing, can distinguish between four main application techniques.

2.3.2.1 Padding

This is certainly the most popular process for both the most conventional and innovative finishing treatments. The machine used for this process can be referred to with various definitions such as padding unit, squeezing unit, etc. After ensuring that the textile substrate can be padded by evaluating its mechanical and structural properties, this technique can be applied to carry out all wet finishing operations, except for some cases. For application, the fabric is conveyed to spreading and stretching units which prevent the formation of creases, then into beck containing the finishing chemical and finally to heavy rollers (called padding unit) which squeeze out the excess liquor. The fabric feeding speed must be constant.

2.3.2.2 Spraying

The application of finishing substances by spraying is used for carrying out gentle finishing processes which leave on the textile material a small concentration of products, and is particularly indicated for applying softening, anti-static and anti-mildew agents. For a good and homogeneous penetration and diffusion of the finish in the textile material, it is better to let the sprayed and wound fabric rest for some hours before drying. In the last few years, a very important field has been developed in the textile sector, *i.e.* the production of webs made of synthetic fibers. For this particular type of product, the resin coating process is carried out only by spraying the finish directly on the fibrous substrate and by generally applying synthetic resins in aqueous emulsion.

2.3.2.3 Exhaustion

Exhaustion uses for treatment staple fiber, yarns and fabrics. This process suitable for chemical that stable when in liquid form, in finishing process, the finishing dissolved in the liquor is first adsorbed on the surface , then penetrates in the core of the fiber, and finally migrates thus allowing good uniformity and consistency (the process is affected by operating temperature and time).

2.3.2.4 Coating

The basic method of producing a coated fabric is the direct spreading of a polymer, in the form of a thick liquid or paste, using a sharpened length of metal called a doctor blade or knife. The doctor blade is set up over a fabric, which is held flat by the application of tension - both lengthways and, ideally with the use as stenter pins, width ways - in a coating machine. The thickened polymer fluid is placed in front of the doctor blade and is spread over the surface of the fabric by the forward movement of the fabric into a heated oven. The thickened liquid can be either water based or solvent based, and evaporation of the water or solvent leaves behind a polymer deposit on the fabric.

2. 4 Literature Reviews

Giglia *et al.* [1983] studied producing a protective clothing by using breathable material in stead of impermeable materials such as plastics or oilskin. An air and water vapor permeable, toxic vapor absorptive non-woven fabric material comprising a web-laid sheet containing fibrillated acrylic fiber, activated carbon fiber and activated carbon particles, wherein the concentration of fibrillated acrylic fiber ranges from about 5 - 25 % by weight based on the total weight of fibers and particles, the concentration of activated carbon fiber ranges from about 10 - 75% by weight, same basis, and the concentration of activated carbon particles ranges from about 15-80% by weight, same basis. [12]

Hubert von Blucher [1987] studied about a material for protection against chemical pollutants is produced by spraying onto a base fabric a mixture of granular activated carbon and a polymer binder, with AIRLESS spraying devices wherein a pump supplies the pressure to 250 bars, to produce an open-pored foam structure which does not materially reduce the air permeability. The binder is permeable to the pollutant so the latter can reach the carbon, The binder should be of high molecular weight or precrosslinked to the extent that it does not penetrate into the mesopores of the activated carbon. [13]

Hoshino *et al.* [1991] invented relates to deodorizing resin compositions and formed deodorizing articles that suitable especially for use in packaging or wrapping. In order to package various malodorous articles, plastic films, aluminum foils, glass containers and the like which do not have air permeability, can prevent emission of offensive odors of contents. However, inconvenient because offensive odors filling the interiors of the packages come out at once when the packages are opened. Formed articles of the deodorizing resin composition of this invention are composed of 99.9-50 parts by weight of a thermoplastic resin and 0.1-50 parts by weight of a deodorizing ingredient. The deodorizing ingredient is a white or substantially colorless deodorizing agent, which may preferably be composed of 10-90 parts by weight of a zinc compound and 90-10 parts by weight of a polycarboxylic acid or a salt thereof, an acidic polymer or a salt thereof, or a sulfate of aluminum. A formed deodorizing article can be obtained by forming or molding the deodorizing resin composition into a desired shape. Formed deodorizing resin composition can have desired beautiful colors and are adapted to absorb offensive odors. [14]

Kurata *et al.* [2000] studied about an absorbent article adapted for preventing a deodorization function of the absorbent article from being degraded to fully achieve the deodorization effect. A main object of an absorbent article including the sanitary napkins, the pantiliners, and the like is to absorb the excrements discharged from the body, and to retain the excrements in the absorbent article without flow back. An absorbent article produced by coating a mixture of the antibacterial silica gel, the acrylic resin and the deodorant on a hydrophobic sheet of product. the deodorant is granular or powdery, and is at least one selected from activated carbon, zeolite, silica gel and bentonite or may be a combination of the agent. Accordingly, the unpleasant odors generated from the excrements can be effectively adsorbed and the leakage of the liquid can be prevented as well. [15]

Sun *et al.* [2004] studied about a fibrous material, which can be an absorbent material, includes a plurality of natural fibers treated with a carboxylic acid-based odor control agent, which are able to withstand insults with an aqueous liquid without dissolving the odor control agent. The acid-based odor control agent is bound to the natural fibers by an organosilicone polymer binder. The silicone polymers have a unique ability to protect the acidic odor control agents from being dissolved or otherwise passed into solution by aqueous liquids, while at the same time permitting odoriferous gases such as ammonia to reach the odor control agents. Put another way, the silicone polymers are water insoluble, and at the same time are highly porous. The combination of odor control system and silicone polymer are applied to the natural fibers, desirably absorbent fibers such as cellulose, by spray coating, brushing, printing, dipping, extrusion, or the like. [16]

CHAPTER III

EXPERIMENTAL

3.1 Materials and Chemicals

- Activated carbon obtained from coconut shell was supplied by Department of Chemical Technology, Faculty of Science Chulalongkorn University, Ground activated carbon powder was sieved using print screen fabric of mesh no. 120T prior to use.
- Bleached, knitted cotton fabric was obtained from A.N.Y. Products Co., Ltd.
- Acramin BA-N, commercial binder from Bayer Thailand Company Limited
- Acrafix CN, commercial crosslinking agent from Bayer Thailand Company Limited
- Diammonium phosphate, $(NH_4)_2HPO_4$, A.N.Y. Products Co., Ltd.
- Iodine solution, I_2, BDH Limited Company
- Potassium Iodide, KI, BDH Limited Company
- Potassium Iodate, KIO_3, BDH Limited Company
- Sodium thiosulphate, $Na_2S_2O_4 \bullet 5\ H_2O$, BDH Limited Company
- Formaldehyde, HCOH, 40 % solution, BDH Limited Company
- Acetylacetone, $C_5H_8O_2$, S.D. Fine Chem. Limited.
- Ammonium acetate, CH_3COONH_4, BDH Limited Company
- Glacial acetic acid, CH_3COOH, J.T. Braker Company
- Hydrochloric acid, HCl, Merck Ltd.
- Sodium hydroxide, NaOH, Merck Ltd.
- Sulphuric acid, H_2SO_4, BDH Limited Company
- Sodium carbonate, Na2CO3, Merck Ltd.
- Methylene blue trihydrate, $C_{16}H_{18}ClN_3S \bullet 3\ H_2O$, Ajax Fine Chem.
- Soluble starch, BDH Limited Company
- Multifiber adjacent fabric (D.W. type), Sumet Labtest Co., Ltd.
- Cotton rubbing cloth, Sumet Labtest Co., Ltd.
- ECE Phosphate Reference Detergent FBA free, Union TSL Co., Ltd.

3.2 Apparatus

- Padder Laboratory Machines, Tsuji Dyeing Machine Co., Ltd.
- Mini dryer stenter, Tsuji Dyeing Machine Co., Ltd.
- Soft Incubator Eyela SLT-600ND
- Oven Memmert ULE 600
- Analytical Balance: Superba-series, Precisa 205 A
- Shirley Stiffness Tester, Shirley Development Limited
- Crock Meter type rubbing tester, Daiei Kagaku Seiki MFG.Co., Ltd.
- Air Permeability Tester, SDL International Limited.
- Ahiba Nuance Machine TS Serial NO. 99250, Data Color International.
- Cabinet Light Box: Model CAC150 Verivide
- UV-Visible Spectrophotometer Model 160A, Shimadzu
- Grey Scale for Assessing Change: SDC Standard Method ISO 105-A03:1993
- Grey Scale for Assessing Staining: SDC Standard Methods ISO 105-A02:1993
- Desiccator

3.3 Experimental Process

3.3.1 Preliminary Investigation of the Effect of Binders on Absorption Efficiency of Activated Carbon Powder

Activated carbon powder of 50 g (particle size < 45 µm) was dispersed in 1,500 ml of binder solutions with various concentrations as shown in Table 3.1. The binder solution was comprised Acramin BA-N (Binder), Acrafix CN (Crosslinking agent) and Diammonium phosphate (Catalyst). The liquor ratio of activated carbon to binder solution was 1:30 (g:ml). The dispersion was stirred for 5 minutes and then followed by filtration to collect the binder coated activated carbon. The treated activated carbon powder was dried at 90 $^{\mathrm{O}}$C in oven for 10 minutes. The dried powder was then cured at the temperature ranges 140 $^{\mathrm{O}}$C - 170 $^{\mathrm{O}}$C and curing time 1- 5 minutes in order to fix binder film onto the powder particle. The absorption efficiency of activated carbon powder was determined for iodine number and methylene blue number as follows

Table 3.1 Amount of Chemical

In all case, the amount of activated carbon was fixed at 10 g.

Sample Number	Amount of Chemical (g/l)			Curing Time (min.)
	Acramin BA-N	Acrafix CN	Diammonium phosphate	
1	10	1	1	1
2				2
3				3
4				4
5				5
6	20	2	2	1
7				2
8				3
9				4
10				5
11	30	3	3	1
12				2
13				3
14				4
15				5
16	40	4	4	1
17				2
18				3
19				4
20				5
21	50	5	5	1
22				2
23				3
24				4
25				5

3.3.1.1 Determination of Iodine Number

Iodine number of treated activated carbon with ASTM D 4607-94 (1999) - standard test method for determination of iodine number of activated carbon.

Dry the ground sample, and cool to room temperature in a desiccator. Three dosages of sample must be estimated to determine the iodine number. Weigh the three dosages (M) of dry carbon to the nearest milligram. Transfer each to one of three clean, dry 250 ml erlenmeyer flasks equipped with ground glass stoppers.

Pipette 10.0 ml of hydrochloric acid solution into each flask, stopper each flask, and swirl gently until the carbon is completely wetted. Loosen the stoppers to vent the flasks, place on a hot plate in a fume hood, and bring the contents to a boil. Allow to boil gently for 30±2 sec to remove any sulphur which may interfere with the test results. Remove the flasks from the hot plate and cool to room temperature. Standardize and then pipette 100 ml of standardized iodine solution into each flask.

Immediately stopper the flasks, and shake the contents vigorously for 30 ±1 sec quickly filter each mixture by gravity through one sheet of folded filter paper into one of three beakers. For each filtrate, use the first 20 to 30 ml to rinse a pipette, and discard the rinse portions. Use clean beakers to collect the remaining filtrates. Mix each filtrate by swirling the beaker, and pipette 50.0 ml of each filtrate into one of three clean 250 ml erlenmeyer flasks.

Titrate each filtrate with standardized sodium thiosulphate solution until a pale yellow color develops. Add 2 ml of starch solution, and continue the titration with standardized sodium thiosulphate solution until 1 drop produces a colorless solution. Record the volume (S) of standardized sodium thiosulphate solution used, perform the following calculations for each carbon dosage

$$A = (N_2)(12{,}693.0)$$

$$B = (N_1)(1{,}26.93)$$

In which N_2 is the normality of the standardized iodine solution and N_1 is the normality of the standardized sodium thiosulphate solution. And calculate the dilution factor (DF) using the equation

$$DF = (I + H)/F$$

In which I is the volume in milliliters of iodine solution used in the standardization procedure, H is the volume in milliliters of hydrochloric acid solution used and F is the volume in milliliters of filtrate used.

Calculate the weight, in milligrams, of iodine adsorbed per gram of sample (X/M) by the equation

$$X/M = [A - (DF)(B)(S)]/M$$

In which S is the volume, in milliliters, of standardized sodium thiosulphate solution used, and M is the weight in grams of the sample. And calculate the normality of the residual filtrate (C) as follows

$$C = (N_1 \: X \: S)/F$$

Using logarithmic paper, plot X/M (as the ordinate) versus C (as the abscissa) for each of the three carbon dosages. Calculate the least squares fit for the three points and plot. The iodine number is the X/M value at a residual iodine concentration (C) of 0.02 N.

3.3.1.2 Determination of Methylene Blue Number

Various concentrations of methylene blue solutions ranging from 0.1 - 1 mg/l were prepared by dissolving methylene blue crystals in distilled water. A calibration curve of absorbance versus concentration was constructed, using a UV-Visible Spectrophotometer at maximum wavelength of 665 nm.

Adsorption of methylene blue were obtained by adding 1 g of treated activated carbon powder to a 250 ml flask containing 100 ml of 1 mg/l aqueous solution of methylene blue. The flask was kept in a room temperature for 2 hours. The samples were withdrawn from the flask, and then the methylene blue solution was separated from the adsorbent and centrifuged for 2 minutes.

The absorbance of the supernatant solution was then measured vessels. The amount of adsorbed at time t, Y (mg/g), was calculated by

$$Y = \frac{(C_0 - C_t)V}{W} \; X \; 1{,}000$$

Where C_0 and C_t are the liquid-phase concentrations initially, respectively (g/ml), V is the volume of solution (ml) and W is the weight of dry activated carbon (g).

3.3.2 Application and Fixation of Activated Carbon onto Knitted Cotton Fabrics

3.3.2.1 Effect of the Binder on Absorption Efficiency of Treated Fabrics

The application of activated carbon onto knitted cotton fabric was carried out in two steps using a padder. The formulas of padding liquors were given in Table 3.2. First, the cotton fabric was padded with 10 g/l activated carbon dispersion. The pressure nip of squeeze roller was adjusted to enable a wet pick up of 80%. Subsequently, the activated carbon treated fabric was repadded with the binder solution using the same padder. In order to study the effect of the binder, 5 pieces of activated carbon treated fabrics were padded with binder solutions with concentrations of 10, 20, 30, 40 and 50 g/l respectively. The treated fabric was dried at 100 ^{O}C for 5 minutes and cured at 150 ^{O}C for 5 minutes to fix activated carbon onto cotton. After that, the treated fabric was soaped off at with 100 ^{O}C for 5 minutes in boiling water containing 2 g/l standard soap and dried in air. Treated fabric properties was determined by formaldehyde adsorption as in 3.3.4 (Observation time is 4 hours)

Table 3.2 Amount of Chemical in Experiment

Sample Number	**Amount of Chemical (g/l)**			**Curing Time (min.)**
	Acramin BA-N	**Acrafix CN**	**Diammonium phosphate**	
1	10	1	1	5
2	20	2	2	
3	30	3	3	
4	40	4	4	
5	50	5	5	

3.3.2.2 The Optimization of Fixation Temperature and Curing Time

In order to study the effect of temperature and curing time in fixation, the application of activated carbon onto knitted cotton fabric was carried out in two steps using a padder. The formulas of padding liquors were given in Table 3.3.

First, the cotton fabric was padded with activated carbon dispersion with concentrations of 10, 20, 30, 40 and 50 g/l respectively. The pressure nip of squeeze roller was adjusted to enable a wet pick up of 80%. Subsequently, the activated carbon treated fabric was repadded with the binder solution using the same padder, pieces of activated carbon treated fabrics were padded with binder solutions with concentrations of 10, 20, 30, 40 and 50 g/l respectively.

The treated fabric was dried at 100 ^{O}C for 5 minutes and was then cured at the temperature ranges 140 ^{O}C - 160 ^{O}C and curing time 1- 5 minutes to fix activated carbon onto cotton. After that, the treated fabric was soaped off at with 100 ^{O}C for 5 minutes in boiling water containing 2 g/l standard soap and dried in air. Treated fabric properties was determined by formaldehyde adsorption, colorfastness to washing, colorfastness to rubbing, stiffness, fabric wicking rate and air permeability

Figure 3.1 Padder Laboratory Machines.

3.3.2.3 Determination of Percent Add-on after Finishing

Dry and determined constant weight of treated fabric. Weighing to the nearest 0.1 mg using the analytical balance and record the weight. Place the specimen in an oven maintained at 105 ^{O}C for 60 minutes. At the end of the period move the specimen from the oven, immediately and put it in the desiccator. When it cooled to room temperature, remove it from the desiccator and reweighing. Repeat the heating and reweighing process for periods of 30 minutes until the weight is constant to within ± 0.001 g and record constant weight. Calculate add-on percent of fabric weight as follow

$$\% \; Add - on \; = \frac{W_i \; - W_0}{W_i} \; x \; 100$$

When W_t is weight of treated fabric and W_o is weight of untreated fabric

Table 3.3 Amount of Chemical Used in Finishing Process

Amount of Chemical (g/l)			**Amount of Activated Carbon (g/l)**	**Curing Temperature (^{O}C)**	**Curing Time (min.)**				
Acramin BA-N	**Acrafix CN**	**Diammonium phosphate**			**Sample Number**				
					1	**2**	**3**	**4**	**5**
10	1	1	10	140	1	2	3	4	5
20	2	2	20		6	7	8	9	10
30	3	3	30		11	12	13	14	15
40	4	4	40		16	17	18	19	20
50	5	5	50		21	22	23	24	25
10	1	1	10	150	1	2	3	4	5
20	2	2	20		6	7	8	9	10
30	3	3	30		11	12	13	14	15
40	4	4	40		16	17	18	19	20
50	5	5	50		21	22	23	24	25
10	1	1	10	160	1	2	3	4	5
20	2	2	20		6	7	8	9	10
30	3	3	30		11	12	13	14	15
40	4	4	40		16	17	18	19	20
50	5	5	50		21	22	23	24	25

3.3.3 Study of Surface Morphology of Treated Fabrics by SEM

Scanning electron microscope (SEM) was used to study surface morphology of treated fabrics. The samples of fabrics were coated with gold by sputtering at room temperature. Scanning electron micrographs of fabrics were taken by scanning electron microscope (JSM-5410LV). The instrument was operated at 15 kv.

3.3.4 Evaluation of Absorption Efficiency of Treated Fabrics

The specimens were cut into 70 x 70 mm. One of the thus-cut specimens was then suspended in a 500 ml erlenmeyer flask, followed of an addition of 50 ml of 15 ppm formaldehyde solution. The outlet of the flask was sealed with paraffin and then places them in the oven at $49\pm1^{O}C$. When reach observation time, remove and cool the flasks for at least 30 minutes. Remove the fabric, recap the flask and shake them to mix any condensation formed on the flask sides.

Pipette 5 ml of Nash reagent into test tubes and pipette 5 ml of the reagent into at least one additional tube for a reagent blank. Add 5 ml aliquots from each of the sample incubation flasks to the tubes and 5 ml of distilled water to the tube which is used as a reagent blank. Mix and place the tubes in a $58\pm1\ ^{O}C$ water bath for 6 minutes. Remove and cool.

Read the absorbance in the spectrophotometer against the reagent blank using a wavelength of 412 nm. And determined formaldehyde absorption (%) of specimens as follow

$$Formaldehyde\ adsorption = \frac{Blank\ \ Concentration - Residual\ Concentration}{Blank\ \ Concentration}\ X\ 100$$

3.3.5 Fastness Testing

With regard to the optimal condition for fixation and the properties of treated fabric is the important requirement. Obviously, this relates to the fastness properties, which normally test according to ISO standard test. These tests including, colorfastness to washing and colorfastness to rubbing. The test results were expressed in grey scale terms. According to the standard grey scale for color change [18] and grey scale for color staining [19]. The fastness grades are given in Table 3.4 and Table 3.5

Table 3.4 Grey Scale Terms for Color Change

Grade	Colorfastness
5	Excellent (no change)
4	Good (Slightly change)
3	Fair (noticeably change)
2	Poor (considerably change)
1	Very poor (much change)

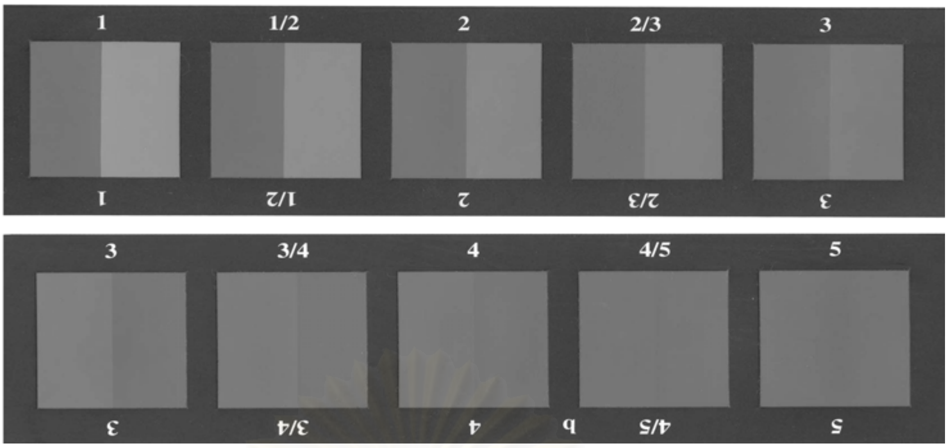


Figure 3.2 Grey Scale for Color Change.

Table 3.5 Grey Scale Terms for Color Staining

Grade	**Colorfastness**
5	Excellent (no staining)
4	Good (Slightly staining)
3	Fair (noticeably staining)
2	Poor (considerably staining)
1	Very poor (much staining)

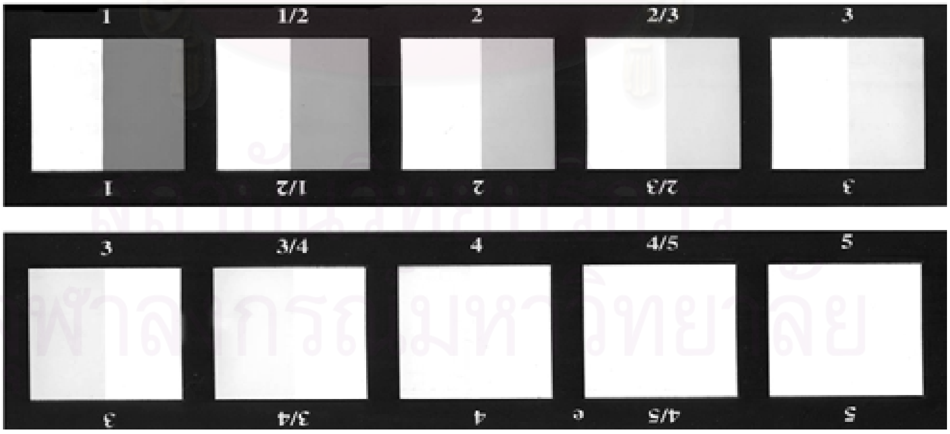


Figure 3.3 Grey Scale for Color Staining**.**

3.3.5.1 Sample Preparation

Fabric was usually tested in the form of a composite specimen (10 cm x 4 cm), made up of the test specimen placed in contact with undyed fabric, usually in the form of multifiber strip, of the same size. The purpose of the undyed fabric is to measure the staining effect of any dye that has been lost from the test fabric.

There are two types of multifiber adjacent fabric, one with wool which was type DW and one without wool which was given in Table 3.6

Table 3.6 Multifiber Strip

Multifiber DW	**Multifiber TV**
Secondary acetate	Triacetate
Bleached cotton	Bleached cotton
Polyamide	Polyamide
Polyester	Polyester
Acrylic	Acrylic
Wool	Viscose

3.3.5.2 Colorfastness to Washing (ISO 105-C06 A1S: 1994) [20]

The specimen of the fabric was put in contact with multifiber adjacent fabric. The specimen was treated in an aqueous solution containing 4 g/l of ECE detergent at 40 ^{O}C for 30 minutes. After that, the colorfastness property of the tested sample was evaluated using the grey scale for color change and grey scale for color staining.

3.3.5.3 Colorfastness to Rubbing (ISO 105-X12: 2001) [21]

The specimen of the fabric was rubbed with a dry rubbing cloth and with a wet rubbing cloth. For dry rubbing, with the dry rubbing cloth flat in place over the end of the finger of crock meter, rub to end fro in a straight line along a track 10 cm. long on the dry specimen, 10 times to and fro in 10 seconds with a drawn ward force of a Newton. For wet rubbing, repeat the test described in dry rubbing with a fresh dry specimen and with a rubbing cloth that has been wetted with water, using take-up of about 95 - 100%. After rubbing, dry the cloth at room temperature. The staining of the rubbing cloths is assessed with the grey scale.

Figure 3.4 Crock Meter.

3.3.6 Evaluation of Stiffness Properties [22]

The stiffness of a treated fabric was evaluated by the Shirley Stiffness Tester SDL 3B complies with ASTM D 1388-96 for the determination of bending length and flexural rigidity of fabrics.

The test specimens are each 25 mm wide and 200 mm long, three are cut parallel to the warp and three parallel to the weft. Condition the specimens for at the standard atmosphere (65±2 % Relative Humidity and a temperature of 20±2 ^{O}C) until the mass of a specimen does not change by more than 0.25% at intervals of 2 hours.

Place the stiffness tester on a level table with the minor towards the observer. Lay the specimen on the platform with one end coincident with the front upper edge of the platform. The slide is placed on the specimen so that the zero of the scale is in line with the mark. The slide is now pushed forward at a uniform rate, carrying the specimen with it, until by locking in the mirror it is seen that the end edge of the specimen is in line with the two scribed lines. Four readings are taken from each specimen, one face up and one face down on the first end, and then the same for the second end. Calculate bending length and flexural rigidity of fabrics, using the equation

$$C = \frac{l}{2}$$

$$G = MC^{3}$$

Where l is length of fabric projecting, C is bending length of tested fabric in centimeters, M is mass of tested fabric in milligrams and G is flexural rigidity of tested fabric.

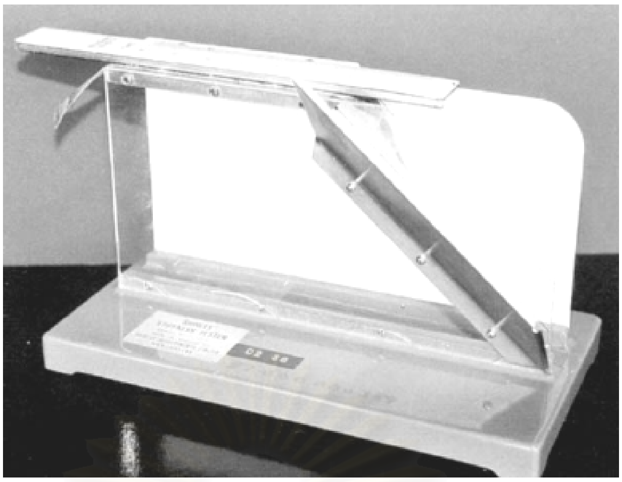


Figure 3.5 Shirley Stiffness Tester.

3.3.7 Determination of Fabric Wicking Rate (Vertical Suspension)

Cut specimens approximate 2.5 x 15 cm. Then**,** pour 200 ml of distilled water in a 500 ml Erlenmeyer flask and hank the fabric on the top edge of flask, fabric should just slightly touched water surface. Determination of fabric wicking rate by check and record wet area of specimens in centimeters at 1 and 5 minutes

3.3.8 Determination of Air Permeability [23]

A method for measuring the permeability of fabrics to air was evaluated by ISO EN 9237. Conditions for the test in this experiment : Test surface area 500 mm^2 and Pressure drop 100 Pa for apparel fabrics.

Mount the test specimen in the circular specimen holder with sufficient tension to eliminate wrinkles. Turn on the vacuum pump unit to force the air through the test specimen and adjust the flow of air gradually till a pressure drop reach 100 Pa, is achieved across the test area of the fabric. Record the air flow after at least 1 minutes or until steady conditions are achieved. Repeat the test at different locations on the sample at least 10 times under the same conditions.

Calculate the arithmetic mean of the individual readings and the coefficient of variation (to the nearest 0.1%) stating the 95% confidence interval. Calculate the air permeability (R) expressed in millimeters per second, using the equation

$$R = \frac{\overline{q_v}}{A} \, x \, 167$$

Where q_v is the arithmetic mean flow rate of air, in cubic decimeters per minute (liters per minute), A is the area of fabric under test, in square centimeters and *167* is the conversion factor from cubic decimeters (or liters) per minute per square centimeter to millimeters per second.

Figure 3.6 Air Permeability Tester.

CHAPTER IV

RESULTS AND DISCUSSION

4.1 The Effect of Binders on Absorption Efficiency of Activated Carbon Powder

In this study, binders, Acramin BA-N, was employed to fix activated carbon powder onto cotton fabric. Binders under thermofixation temperature were transformed into polymer network film where activated carbon powder particles performance of activated carbon. Therefore, the prelimary investigation of adsorption performance of binder treated activated carbon was carried out as follows:

4.1.1 Iodine Number

Table 4.1 and Figure 4.1 show the iodine number obtained from Acramin treated activated carbon. The results show that when the amount of binder increases the iodine number decreases, indicating that binder exhibited the negative effect on the adsorption capability of activated carbon. When the amount of binder, temperature and curing time were increased, the iodine number was decreased. It is because of the coverage of some binder film on the surface of activated carbon as a result to decrease the adsorption capacity.

Considered the effect of curing temperature, at 140 ^{O}C the iodine number is approximately 1,120 mg/g when the amount of binder and curing time was varied. When the fixation temperature increased from 150, 160 and 170 ^{O}C, at all case of the amount of binder applied the iodine number decreased. Example from 160 to 170 ^{O}C, the iodine number decreased from 1,133 mg/g to 992 mg/g. The reduction in iodine number indicated the reduction of the adsorption capacity of activated carbon due to the barrier effect of binder film. At the high temperature and prolong curing, it would lead to the better film formation which exhibited the effective coverage on the surface of activated carbon and its consequent adsorption performance.

Table 4.1 Iodine Number of Treated Activated Carbon Powder by Varying Amount of Binder, Time and Temperature in Fixation

Temperature (^{O}C)	**Amount of Binder (g/l)**	**Iodine Number (mg/g)**				
		Curing Time (min.)				
		1	**2**	**3**	**4**	**5**
-	-	1,210				
140	10	1,133	1,132	1,127	1,127	1,125
	20	1,133	1,130	1,128	1,125	1,120
	30	1,134	1,132	1,131	1,131	1,126
	40	1,132	1,131	1,131	1,129	1,125
	50	1,126	1,136	1,133	1,128	1,130
150	10	1,076	1,047	1,020	976	956
	20	1,007	982	954	950	921
	30	964	952	928	910	885
	40	953	932	898	863	853
	50	940	910	887	846	813
160	10	1,029	1,027	1,015	1,013	987
	20	1,016	987	974	971	960
	30	985	979	944	895	852
	40	959	930	891	830	821
	50	936	912	876	814	804
170	10	991	977	958	960	960
	20	985	977	950	952	925
	30	888	881	848	847	827
	40	871	851	859	800	630
	50	821	748	747	725	588

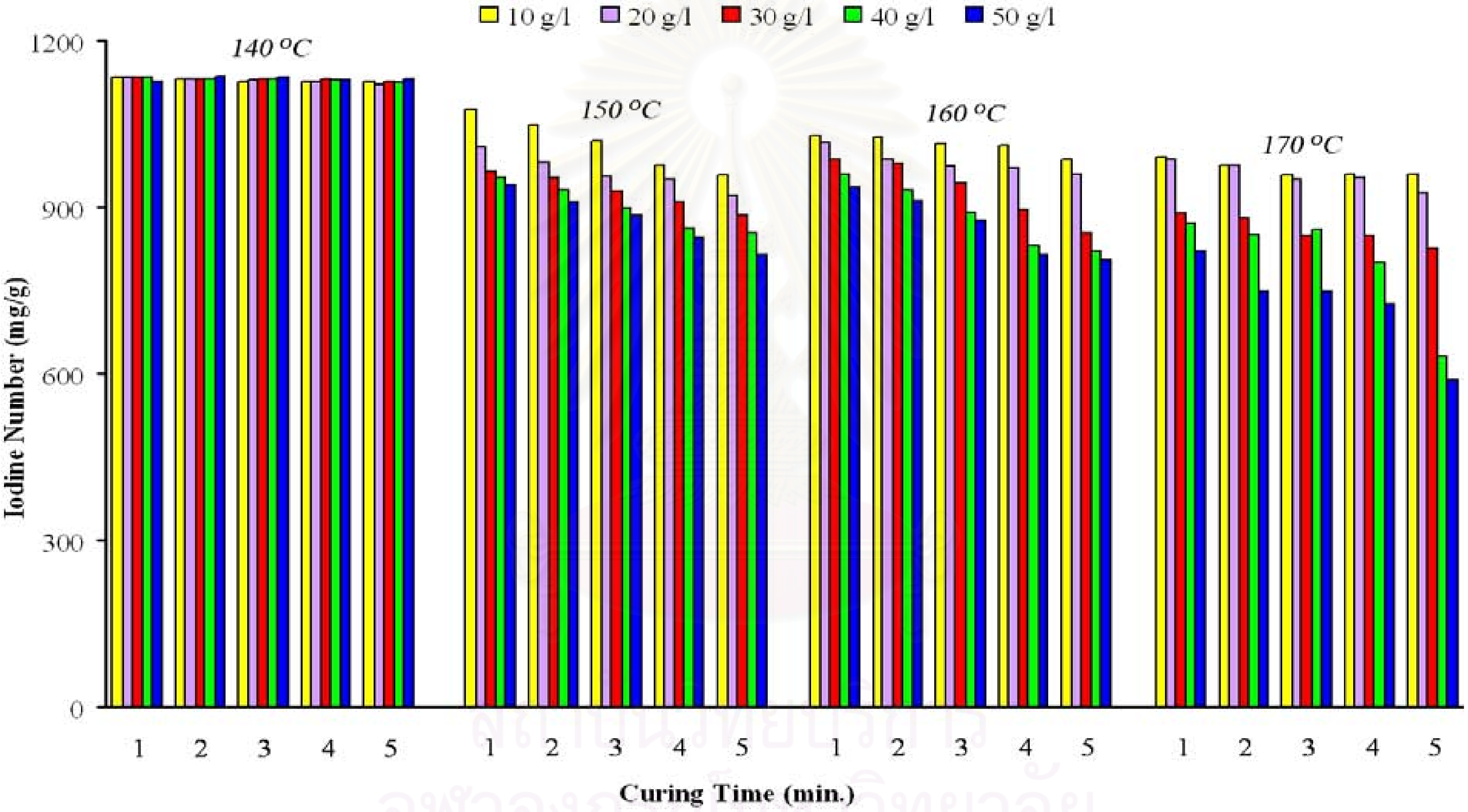


Figure 4.1 Iodine Number of Treated Activated Carbon Powder by Varying Amount of Binder, Time and Temperature in Fixation.

4.1.2 Methylene Blue Number

Table 4.2 and Figure 4.2 show the methylene blue number obtained from Acramin treated activated carbon. The results indicated, binder exhibited the negative effect on the adsorption capability of activated carbon, when the amount of binder, temperature and curing time were increased, the methylene blue number was decreased.

Considered the effect of curing temperature, when the fixation temperature increased from 140, 150, 160 and 170 ^{O}C, at all case of the amount of binder applied the methylene blue number decreased. Example from 140 to 150 ^{O}C, the methylene blue number decreased from 65.6 mg/g to 62.9 mg/g, (about 4% reduction) and from 160 ^{O}C to 170 ^{O}C, the methylene blue number decreased from 62.8 mg/g to 57.6 mg/g, (about 8% reduction). The reduction of performance in terms of methylene blue number indicated the reduction of the adsorption capacity of activated carbon due to the barrier effect of binder film. It is suggested that the high temperature and prolong curing in fixation process, it would lead to the better film formation which revealed the effective coverage on the surface of activated carbon and its consequent adsorption performance.

Table 4.2 Methylene Blue Number of Treated Activated Carbon Powder by Varying Amount of Binder, Time and Temperature in Fixation

Temperature (^{O}C)	**Amount of Binder (g/l)**	**Methylene Blue Number (mg/g)**				
		Curing Time (min.)				
		1	**2**	**3**	**4**	**5**
-	-	95.2				
140	10	65.6	65.3	65.1	56.0	54.9
	20	60.0	58.5	55.6	52.8	47.3
	30	57.2	55.8	53.8	51.1	45.2
	40	53.3	49.2	45.2	40.6	42.8
	50	50.8	48.5	46.0	43.0	37.4
150	10	62.9	61.4	57.8	55.6	50.5
	20	57.4	54.2	49.7	48.1	46.6
	30	56.0	53.5	48.2	47.8	47.0
	40	48.7	46.8	42.7	45.1	46.8
	50	45.4	43.5	41.8	44.2	40.5
160	10	62.8	60.9	64.7	62.7	61.1
	20	53.1	56.4	62.0	53.6	55.7
	30	49.7	52.4	49.7	52.0	53.0
	40	47.2	45.3	45.3	50.6	51.6
	50	43.5	42.0	42.5	45.7	47.4
170	10	57.6	55.6	52.4	49.2	49.8
	20	48.4	46.2	47.1	47.3	48.6
	30	47.7	43.2	45.7	43.8	46.1
	40	43.5	41.8	43.0	43.0	43.5
	50	41.3	40.9	41.1	41.1	40.1

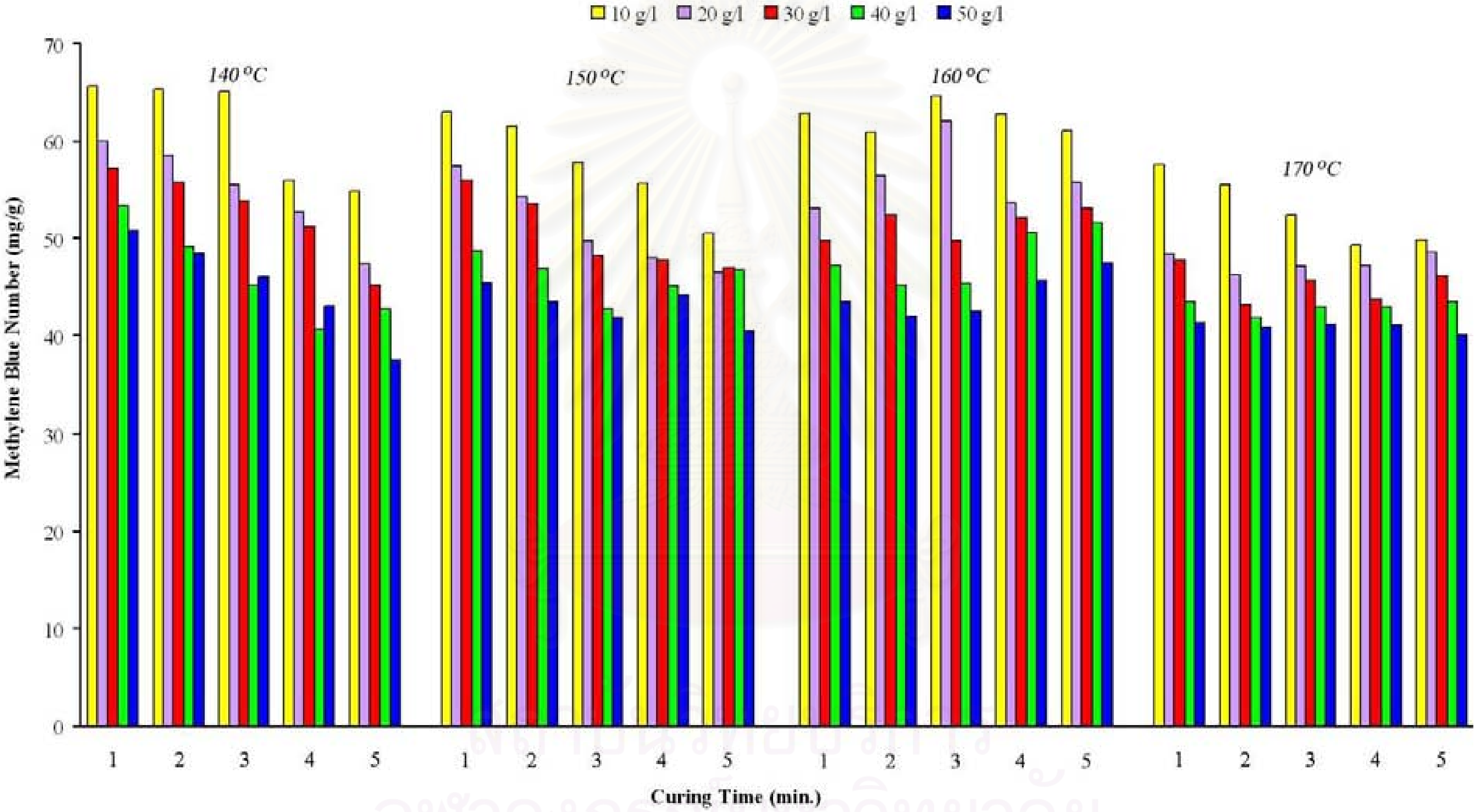


Figure 4.2 Methylene Blue Number of Treated Activated Carbon Powder by Varying Amount of Binder, Time and Temperature in Fixation.

4.2 Application and Fixation of Activated Carbon onto Knitted Cotton Fabrics

4.2.1 Effect of the Activated Carbon and Binder Ratio on Percent Reduction of Formaldehyde

The activated carbon to binder concentration ratios of 1:1, 1:2, 1:3, 1:4 and 1:5 were used to study the fixation of activated carbon onto knitted fabric. Table 4.3 showed the results of the percent formaldehyde adsorption. It was found that the percent formaldehyde adsorption decreased when the amount of binder increased. As seen from the table, the activated carbon to binder ratios of 1:1, 1:2, 1:3, 1:4 and 1:5 yielded the successive reduction of the percent formaldehyde adsorption from 10.79%, 9.87%, 7.85%, 8.76% and 3.04% respectively. At the ratio of 1:1, the percent formaldehyde adsorption was the highest value in this study. It is suggested that the lowest ratio of the activated carbon to binder is recommended in order to achieve the optimum performance of the adsorption capacity of the activated carbon.

Table 4.3 Formaldehyde Adsorption of Treated Fabrics at Various Ratio of Binder

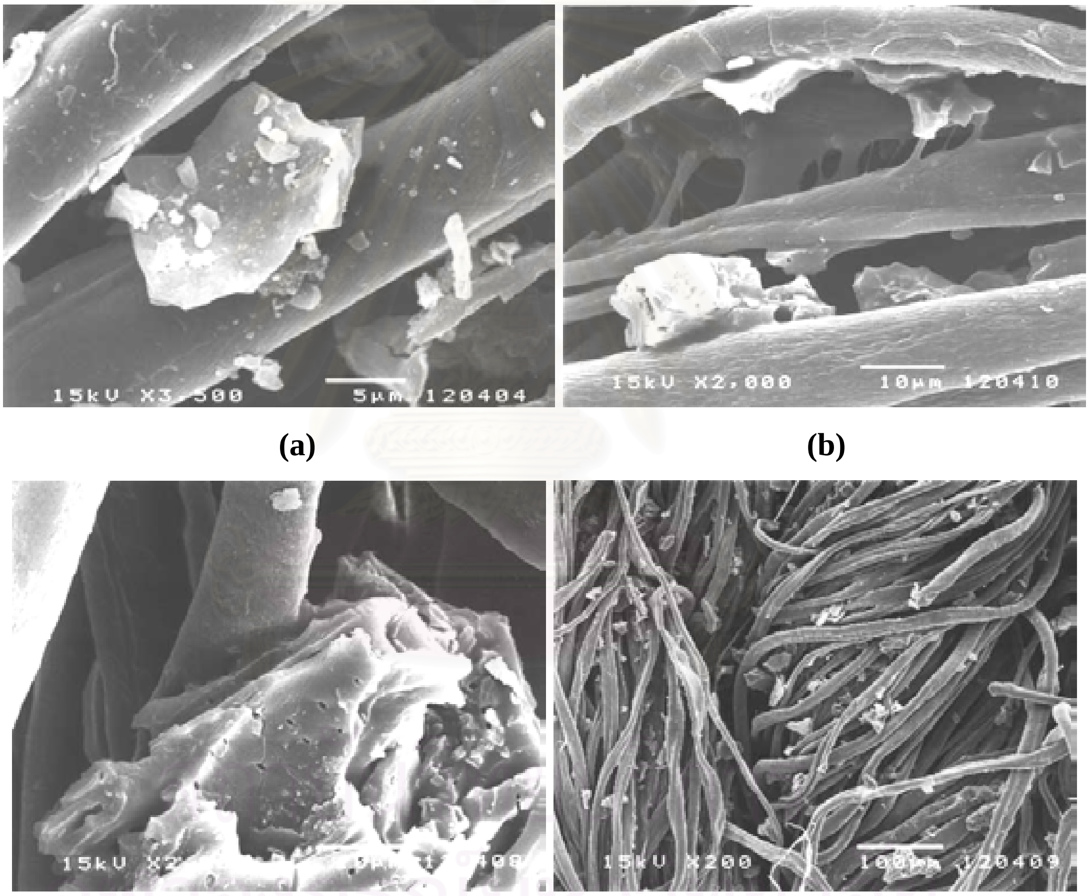
Activated Carbon : Binder	Formaldehyde Adsorption (%)
1 : 1	10.79
1 : 2	9.87
1 : 3	7.85
1 : 4	8.76
1 : 5	3.04

4.2.2 Calculation of Add-on Percent of Fabric Weight after Finishing

Percent add-on of fabric weights after padding with activated carbon solution ranging from 10, 20, 30, 40 and 50 g/l is 1.0784 %, 2.1021 %, 3.0615%, 3.9883 % and 5.0270 %, respectively. The results show that the percent add-on increases with an increase in the concentration of activated carbon.

4.3 Surface Morphology of Treated Fabrics

The surface morphology of treated fabrics shows in Figure 4.3. Micrograph (a) in Figure 4.3 show the surface of fabric treated with activated carbon only. In micrograph (b) exhibited binder cover on the activated carbon and fabric surface, as the activated carbon powder was fixed onto fabric through binder film. Micrograph (c) showed surface of activated carbon on fabric after treated with binder, there are some pores of activated carbon still visible, indicating that binder film not fully cover on fabric surface after finishing.



(a) **(b)**

(c) **(d)**

Figure 4.3 Scanning Electron Micrograph

(a) Treated Fabric Surface with out Binder

(b) Treated Fabric with Binder

(c) Surface of Activated Carbon after Coated with Binder

(d) Surface of treated fabric

4.4 Result of Adsorption Efficiency of Treated Fabrics

It was desirable in this study that the good fixation of activated carbon onto knitted cotton fabric with excellent adsorption performance was achieved. Several factors especially the amount of binder, fixation temperature and curing time were found to influence those requirements. Effects of temperature and time were further studied in details and the results were given as follows:

Table 4.4 and 4.5 showed the effect of fixation temperature and time on the adsorption capacity. Considered at fixed curing temperature when curing time increased; the percent formaldehyde adsorption of fabric decreased. It was possible that the formation of binder film on fabric surface would hinder the adsorption performance of treated fabric and activated carbon on fabric surface.

From Table 4.5, it was found after washing process, percent formaldehyde adsorption of treated fabric at curing temperature of 140 ^{O}C was reduced. Meanwhile, formaldehyde adsorption capacity of treated fabric at curing temperatures of 150 and 160 ^{O}C was increased. It was possible that the binder film not full adhesion at 140 ^{O}C curing temperature, during washing some activated carbon was easily removed from fabric surface, as a result of the poor fixation of binder film at the insufficient curing temperature leading to poor fastness of activated carbon. For 150 and 160 ^{O}C curing temperature, the binder film was sufficiently created and strongly adhered onto fabric surface. As a consequent, during washing process, most of activated carbon powder was retained inside fabric due to its resistance to washing. Also during washing, crack and peel-off of binder film might occur, allowing an increase in the formaldehyde adsorption capacity observed after washing

Considered the adsorption efficiency of treated fabric compare with untreated fabric, formaldehyde adsorption capacity of untreated fabric at 2 hour and 4 hour was higher than treated fabric (at all case). But after 24 hour, found the adsorption efficiency of treated fabric was higher than untreated fabric. Example, at 150 ^{O}C curing temperature, activated carbon 50 g/l and curing time 4 minute, the adsorption efficiency of treated fabric was increased about 38 percent when compared with untreated fabric

Table 4.4 Percent of Formaldehyde Adsorption of Treated Fabrics

Curing Temperature (^{O}C)	Amount of Chemical (g/l)	Formaldehyde Adsorption (%)														
		2 h					4 h					24 h				
		Curing Time (min.)														
		1	2	3	4	5	1	2	3	4	5	1	2	3	4	5
-	-	8.27					17.85					24.23				
140	10	7.68	7.12	4.78	3.70	1.35	7.79	6.30	5.55	3.78	2.52	18.22	12.84	12.84	12.51	7.85
	20	7.48	7.25	5.18	4.64	3.37	11.07	7.16	5.67	4.91	2.66	22.31	20.81	17.80	15.09	8.21
	30	8.86	7.34	5.71	5.32	3.89	12.38	10.68	6.19	5.54	3.30	23.01	21.09	19.07	16.58	10.34
	40	8.77	8.23	6.12	5.27	4.37	12.41	12.08	11.21	9.72	3.63	23.47	22.96	19.50	18.08	15.71
	50	8.84	8.19	6.21	5.30	4.77	15.16	13.76	13.06	11.98	5.31	31.35	31.54	22.05	22.79	18.91
150	10	7.26	6.48	5.45	4.89	3.94	8.59	8.21	7.86	7.51	7.13	12.34	10.6	10.33	8.46	7.86
	20	7.51	6.78	5.83	5.14	4.33	8.63	8.46	8.07	7.73	7.17	14.12	11.66	11.26	9.26	9.22
	30	7.86	6.87	6.52	5.36	4.84	8.79	8.46	8.25	7.90	7.47	16.7	14.83	13.22	12.19	11.46
	40	8.16	7.38	6.70	5.49	4.93	9.02	8.72	8.34	8.16	7.60	22.75	20.14	17.36	16.98	12.99
	50	8.34	7.56	7.51	6.39	6.21	9.15	8.81	8.50	8.46	7.78	27.58	25.3	23.10	21.25	20.13
160	10	2.76	1.43	1.02	0.82	0.57	4.31	1.84	2.81	2.55	2.01	11.10	10.98	9.82	5.63	4.12
	20	1.91	1.50	1.24	1.02	0.81	5.18	3.99	3.24	3.51	2.25	13.24	13.98	9.30	9.82	6.42
	30	2.83	1.96	1.35	1.18	1.23	6.04	4.02	3.56	6.58	3.99	15.79	14.95	12.46	10.83	6.42
	40	3.12	2.04	1.61	1.22	1.70	6.58	5.42	6.69	7.09	3.79	17.68	16.11	13.64	11.33	9.87
	50	3.72	2.76	3.07	2.30	2.00	7.32	6.27	6.13	7.74	5.61	21.02	17.43	13.98	13.92	11.24

Table 4.5 Percent of Formaldehyde Adsorption of Treated Fabrics after Wash

Curing Temperature (^{O}C)	**Amount of Chemical (g/l)**	**Formaldehyde Adsorption (%)**														
		2 h					**4 h**					**24 h**				
		Curing Time (min.)														
		1	**2**	**3**	**4**	**5**	**1**	**2**	**3**	**4**	**5**	**1**	**2**	**3**	**4**	**5**
-	-	8.95					18.14					25.29				
140	10	5.30	4.82	3.45	2.45	1.50	7.66	6.23	4.67	3.12	2.24	17.28	11.56	11.56	11.26	7.07
	20	6.20	5.77	4.20	3.63	1.50	11.02	6.98	4.30	4.87	2.96	20.08	18.73	16.02	13.58	7.07
	30	7.84	6.94	4.58	4.11	1.97	12.11	11.87	6.02	6.04	3.67	20.71	18.98	17.16	14.92	9.31
	40	8.53	7.54	5.87	5.15	3.56	11.91	11.23	11.01	10.80	4.03	21.65	20.66	17.55	16.27	14.14
	50	8.65	8.03	6.68	5.25	4.09	14.84	13.51	12.90	11.20	6.73	30.18	28.39	19.85	20.51	17.02
150	10	9.07	8.10	6.81	6.11	4.92	10.85	10.26	9.82	9.39	8.91	15.43	13.25	12.91	10.58	9.82
	20	9.39	8.48	7.29	6.43	5.41	11.44	10.38	10.09	9.66	8.96	17.65	14.57	14.08	11.57	11.53
	30	9.82	8.59	8.15	6.70	6.05	10.79	10.58	10.31	9.88	9.34	24.87	21.54	16.52	15.24	14.32
	40	10.20	9.23	8.37	6.86	6.16	10.74	10.69	10.42	10.20	9.50	28.19	26.69	24.20	23.72	20.24
	50	10.42	9.45	9.39	7.99	6.27	11.33	11.01	10.63	10.58	9.72	38.23	35.37	32.62	30.31	27.66
160	10	2.39	1.79	1.20	1.02	0.34	5.39	2.30	3.51	3.19	2.29	18.88	17.37	11.63	7.04	5.15
	20	3.45	1.88	1.55	1.04	0.39	6.47	4.99	4.05	3.51	2.81	19.05	17.52	12.28	12.28	8.02
	30	3.54	2.45	1.69	1.47	0.54	7.55	4.99	4.45	8.22	3.99	19.74	18.69	15.58	13.54	8.03
	40	3.90	2.55	2.01	1.52	1.85	8.22	5.53	8.36	10.11	4.59	22.10	20.14	17.05	14.16	12.34
	50	4.65	3.45	3.84	2.87	2.12	11.27	11.32	11.05	10.78	7.01	28.78	20.54	17.47	17.40	14.04

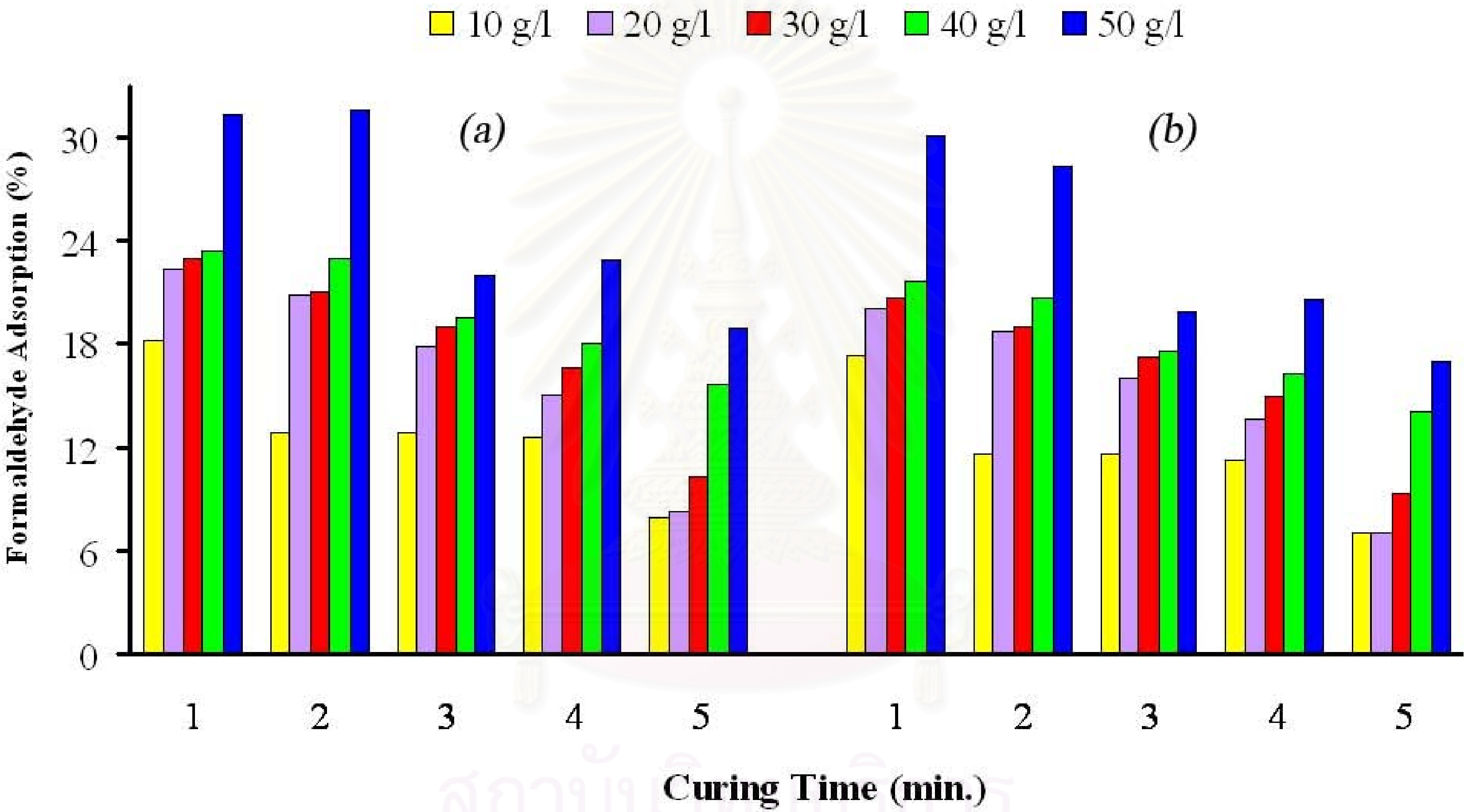


Figure 4.4 Formaldehyde Adsorption of Treated Fabrics at 140 ^{O}C Curing Temperature (Observation Time : 24 hour) (a) Before washing (b) After washing.

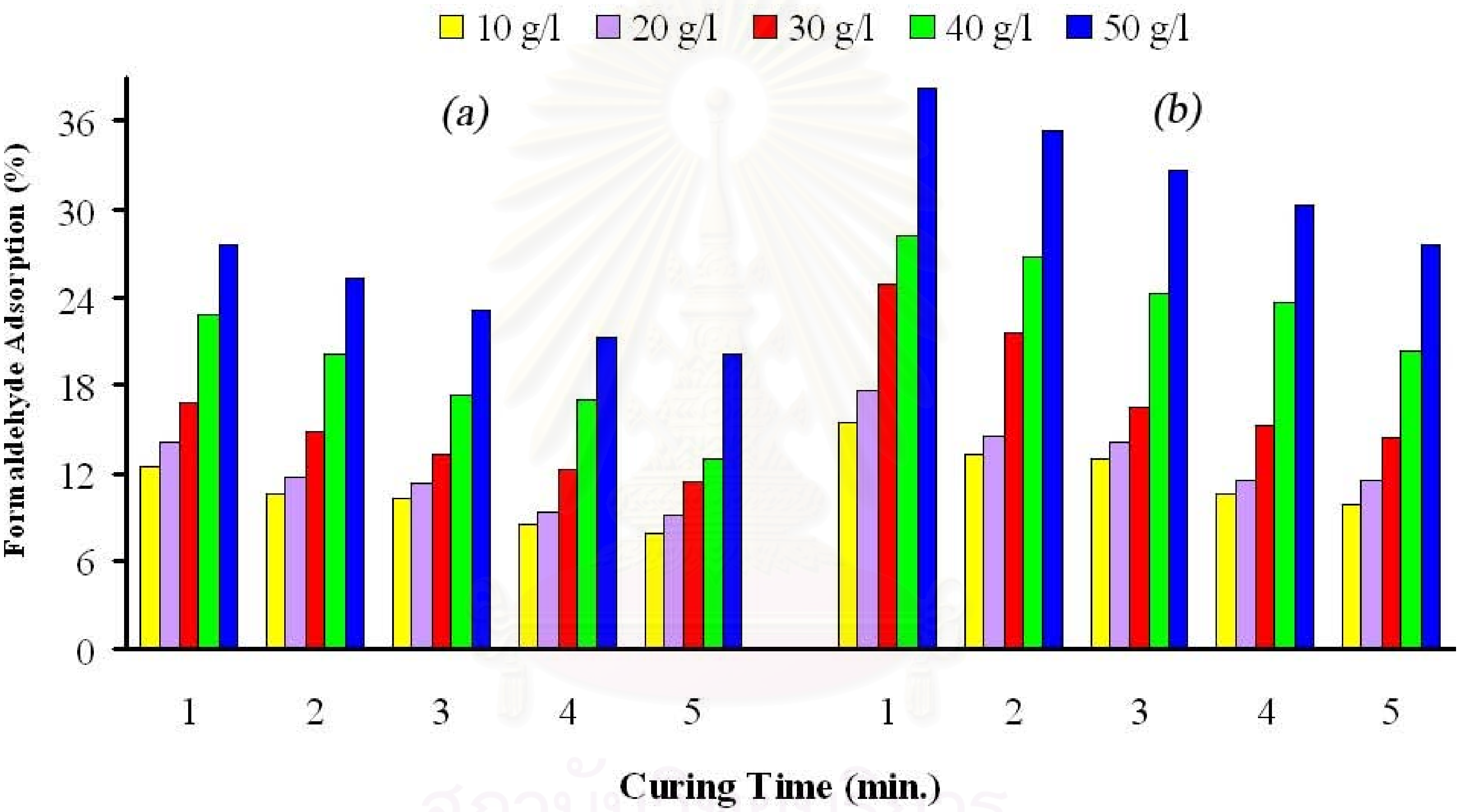


Figure 4.5 Formaldehyde Adsorption of Treated Fabrics at 150 ^{O}C Curing Temperature (Observation Time : 24 hour) (a) Before washing (b) After washing.

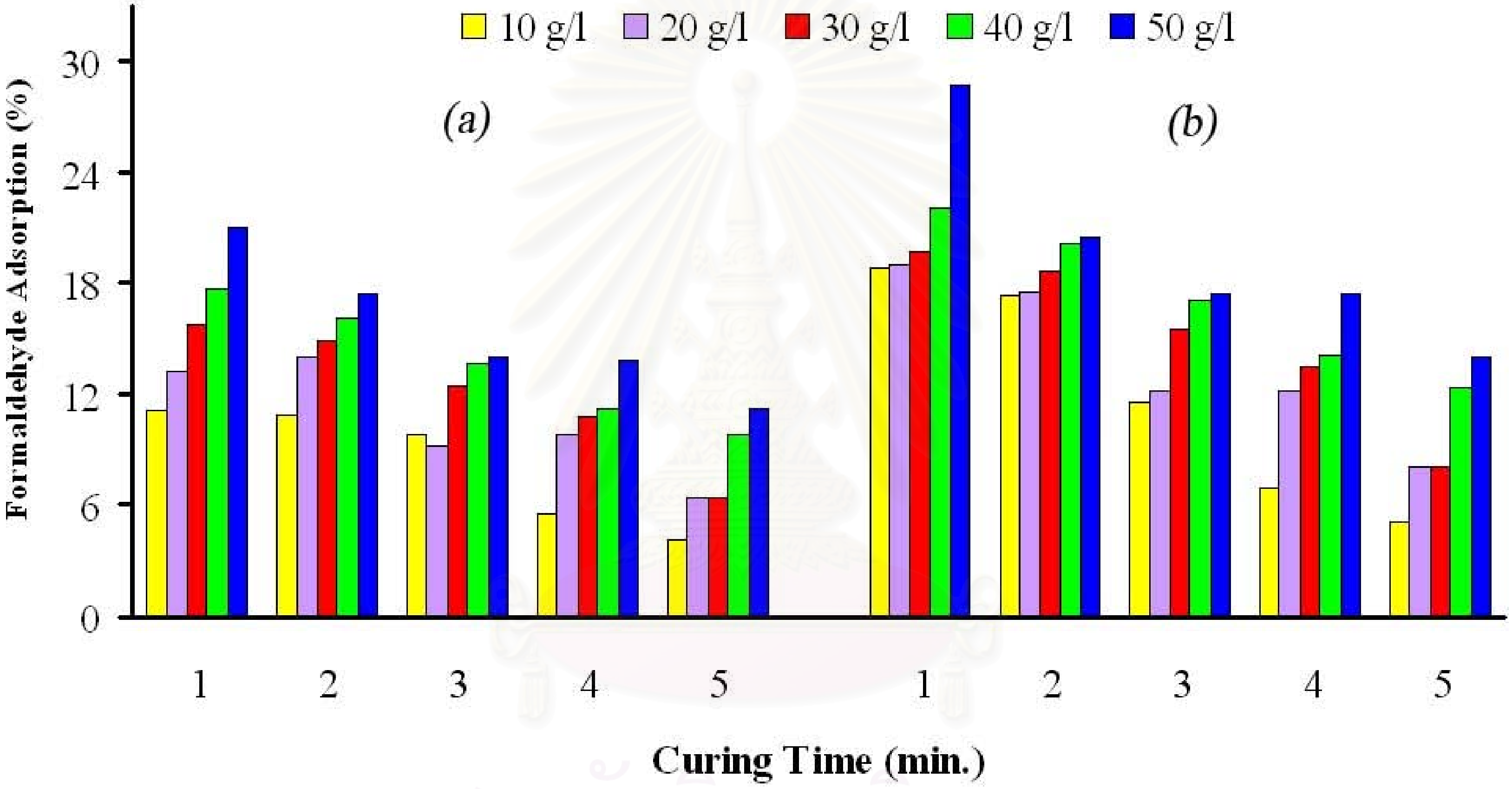


Figure 4.6 Formaldehyde Adsorption of Treated Fabrics at 160 ^{O}C Curing Temperature (Observation Time : 24 hour) (a) Before washing (b) After washing.

4.5 Fastness Testing

The fastness properties including wash fastness and rubbing fastness were carried out according to section 3.3.5. Results are presented as follows

4.5.1 Colorfastness to Washing

Table 4.6 The Result of Colorfastness to Washing of Treated Fabrics at 140 ^{O}C Curing Temperature (Standard: ISO 105-CO6 A1S: 1994)

Amount of Chemical (g/l)	**Sample Number**	**Color Change**	**Color Staining**					
			Acetate	**Cotton**	**Nylon**	**Polyester**	**Acrylic**	**Wool**
10	1	3-4	4-5	4	4-5	4-5	4-5	4
	2	3-4	4-5	4	4-5	4-5	4-5	4
	3	4	4-5	4	4-5	4-5	4-5	4
	4	4	4-5	4-5	4-5	4-5	4-5	4
	5	4-5	4-5	4-5	4-5	4-5	4-5	4
20	6	4-5	4-5	4	4-5	4-5	4-5	4
	7	4-5	4-5	4	4-5	4-5	4-5	4
	8	4-5	4-5	4	4-5	4-5	4-5	4
	9	4-5	4-5	4-5	4-5	4-5	4-5	4
	10	4	4-5	4-5	4-5	4-5	4-5	4
30	11	4	4-5	4	4-5	4-5	4-5	4
	12	4	4-5	4	4-5	4-5	4-5	4
	13	4	4-5	4-5	4-5	4-5	4-5	4
	14	4	4-5	4-5	4-5	4-5	4-5	4
	15	4	4-5	4-5	4-5	4-5	4-5	4
40	16	4	4-5	4	4-5	4-5	4-5	4
	17	4	4-5	4	4-5	4-5	4-5	4
	18	4	4-5	4-5	4-5	4-5	4-5	4
	19	4-5	4-5	4-5	4-5	4-5	4-5	4
	20	4-5	4-5	4-5	4-5	4-5	4-5	4
50	21	4	4-5	4	4-5	4-5	4-5	4
	22	4	4-5	4	4-5	4-5	4-5	4
	23	4-5	4-5	4-5	4-5	4-5	4-5	4
	24	4-5	4-5	4-5	4-5	4-5	4-5	4
	25	4-5	4-5	4-5	4-5	4-5	4-5	4

Change in color and color staining of treated fabrics after washing were rated using grey scale. From the treated fabric at 140 ^{O}C curing temperature, color change was rated in range 3-4 to 4-5 on grey scale and color staining in range 4-5. Change in color was moderated when compared to the maximum rate of 5 on grey scale. The results indicated that activated carbon was not effectively fixed by Acramin binder. Positively, an improvement in wash fastness was observed with an increase in the amount of binder applied, curing temperature and time. The staining ratings are quite good since activated carbon as pigment usually exhibits poor substatantivity on textile materials.

Table 4.7 The Result of Colorfastness to Washing of Treated Fabrics at 150 ^{O}C Curing Temperature (Standard: ISO 105-CO6 A1S: 1994)

Amount of Chemical (g/l)	**Sample Number**	**Color Change**	**Color Staining**					
			Acetate	**Cotton**	**Nylon**	**Polyester**	**Acrylic**	**Wool**
10	1	4	4-5	4	4-5	4-5	4-5	4
	2	4	4-5	4	4-5	4-5	4-5	4
	3	4	4-5	4-5	4-5	4-5	4-5	4
	4	4-5	4-5	4-5	4-5	4-5	4-5	4
	5	4-5	4-5	4-5	4-5	4-5	4-5	4
20	6	4	4-5	4	4-5	4-5	4-5	4
	7	4	4-5	4-5	4-5	4-5	4-5	4
	8	4	4-5	4-5	4-5	4-5	4-5	4
	9	4-5	4-5	4-5	4-5	4-5	4-5	4
	10	4-5	4-5	4-5	4-5	4-5	4-5	4
30	11	4	4-5	4	4-5	4-5	4-5	4
	12	4	4-5	4	4-5	4-5	4-5	4
	13	4	4-5	4-5	4-5	4-5	4-5	4
	14	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	15	4-5	4-5	4-5	4-5	4-5	4-5	4-5
40	16	4	4-5	4	4-5	4-5	4-5	4
	17	4	4-5	4	4-5	4-5	4-5	4
	18	4	4-5	4-5	4-5	4-5	4-5	4
	19	4	4-5	4-5	4-5	4-5	4-5	4-5
	20	4-5	4-5	4-5	4-5	4-5	4-5	4-5
50	21	4	4-5	4-5	4-5	4-5	4-5	4
	22	4-5	4-5	4-5	4-5	4-5	4-5	4
	23	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	24	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	25	4-5	4-5	4-5	4-5	4-5	4-5	4-5

From the treated fabrics at 150 ^{O}C curing temperature, color change was rated in range 4 to 4-5 on grey scale and color staining in range 4-5. Change in color was quite good when compared to the maximum rate grey scale. The results indicated that activated carbon was effectively fixed by binder. An improvement in wash fastness was observed with an increase in the amount of binder applied, curing temperature and time. The staining ratings are good.

Table 4.8 The Result of Colorfastness to Washing of Treated Fabrics at 160 ^{O}C Curing Temperature (Standard: ISO 105-CO6 A1S: 1994)

Amount of Chemical (g/l)	**Sample Number**	**Color Change**	**Color Staining**					
			Acetate	**Cotton**	**Nylon**	**Polyester**	**Acrylic**	**Wool**
10	1	4-5	4-5	4-5	4-5	4-5	4	4-5
	2	4-5	4-5	4-5	4-5	4-5	4	4-5
	3	4-5	4-5	4-5	4-5	4-5	4	4-5
	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
20	6	4	4-5	4-5	4-5	4-5	4-5	4
	7	4	4-5	4-5	4-5	4-5	4-5	4-5
	8	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	9	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	10	4-5	4-5	4-5	4-5	4-5	4-5	4-5
30	11	4	4-5	4-5	4-5	4-5	4-5	4
	12	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	13	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	14	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	15	4-5	4-5	4-5	4-5	4-5	4-5	4-5
40	16	4	4-5	4-5	4-5	4-5	4-5	4
	17	4	4-5	4-5	4-5	4-5	4-5	4-5
	18	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	19	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	20	4-5	4-5	4-5	4-5	4-5	4-5	4-5
50	21	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	22	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	23	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	24	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	25	4-5	4-5	4-5	4-5	4-5	4-5	4-5

From the treated fabrics at 160 ^{O}C curing temperature, color change was rated in range 4-5 on grey scale and color staining in range 4-5. Change in color was good when compared to the maximum rate of 5 on grey scale. The results indicated that an improvement in wash fastness was observed with an increase in the amount of binder applied, curing temperature and time. The staining ratings are good.

4.5.2 Colorfastness to Rubbing

Table 4.9 The Result of Colorfastness to Rubbing of Treated Fabrics at 140 ^{O}C Curing Temperature (Standard: ISO105-X12: 2001)

Amount of Chemical (g/l)	**Sample Number**	**Color Staining**			
		Warp Direction		**Weft Direction**	
		Dry	**Wet**	**Dry**	**Wet**
10	1	4	2-3	4	3
	2	4	3-4	4-5	3-4
	3	4-5	3-4	5	3
	4	5	4	5	4
	5	5	4-5	5	4-5
20	6	5	2-3	5	3
	7	5	2-3	4	2-3
	8	4-5	3	4-5	3
	9	5	3-4	5	3-4
	10	5	4	5	4-5
30	11	4-5	3	4-5	3
	12	5	3	5	3
	13	5	3-4	5	4
	14	5	3-4	4-5	3-4
	15	4-5	3-4	5	4
40	16	4-5	3	4-5	3
	17	5	3	5	3
	18	5	3-4	5	4
	19	5	3-4	4-5	3-4
	20	4-5	3-4	5	4
50	21	5	3	5	3-4
	22	5	3	4	3
	23	5	3-4	5	4
	24	4-5	3-4	4-5	4
	25	5	4	5	4

Color staining of treated fabrics after rubbing was rated using grey scale. For the treated fabrics at 140 ^{O}C curing temperature tested in wet condition color staining observed along the warp and weft directions were rated in range of 2-3 up to 4 on grey scale and in dry condition color staining evaluated along the warp and weft directions are in the range from 4-5 to 5. The staining ratings are good when evaluated in dry condition.

Table 4.10 The Result of Colorfastness to Rubbing of Treated Fabrics at 150 ^{O}C Curing Temperature (Standard: ISO105-X12: 2001)

Amount of Chemical (g/l)	Sample Number	Color Staining			
		Warp Direction		Weft Direction	
		Dry	Wet	Dry	Wet
10	1	4	3	5	3-4
	2	5	3-4	4	4
	3	4-5	3-4	5	4
	4	4-5	4	4-5	4-5
	5	4	4	5	4
20	6	4-5	3	5	3
	7	5	3	4-5	3-4
	8	5	3	5	3
	9	5	4	5	4
	10	5	3-4	5	4
30	11	5	3	5	3
	12	4-5	3	5	3
	13	5	3-4	5	3-4
	14	5	3-4	5	4
	15	5	3-4	5	4
40	16	4-5	3	5	3
	17	4-5	3	5	3
	18	5	3	5	3-4
	19	5	3	5	3
	20	5	4	5	4
50	21	4	3	5	3
	22	5	3	5	3
	23	4-5	3	5	3-4
	24	5	3-4	5	3
	25	5	4-5	5	4

From the treated fabrics at 150 ^{O}C curing temperature in wet condition color staining observed along the warp and weft directions are in the range from 3 to 4-5 on grey scale and in dry condition color staining evaluated along the warp and weft directions were rated in range of 4-5 up to 5. The staining ratings are good when evaluated in dry condition. The results show that as the curing time increases the rubbing fastness increases, indicating that prolong curing time resulted in the better film formation of the binder.

Table 4.11 The Result of Colorfastness to Rubbing of Treated Fabrics at 160 ^{O}C Curing Temperature (Standard: ISO105-X12: 2001)

Amount of Chemical (g/l)	**Sample Number**	**Color Staining**			
		Warp Direction		**Weft Direction**	
		Dry	**Wet**	**Dry**	**Wet**
10	1	4-5	4	4-5	4
	2	5	4	4-5	4-5
	3	5	4-5	5	4
	4	5	4-5	5	4
	5	5	4	5	4-5
20	6	4-5	3-4	5	4
	7	4-5	3-4	4.5	3-4
	8	5	4	5	4
	9	5	4-5	5	4-5
	10	5	4.5	5	4-5
30	11	5	3	5	3-4
	12	5	3-4	5	3-8
	13	4-5	4	4-5	4
	14	5	4	5	4-5
	15	5	4-5	5	4-5
40	16	5	3-4	5	3-4
	17	4-5	3-4	5	3
	18	5	4-5	5	4-5
	19	5	4-5	4-5	4
	20	5	4-5	5	4-5
50	21	5	3	5	3-4
	22	5	3	4-5	3-4
	23	4-5	3-4	5	4
	24	5	4-5	5	4
	25	5	4-5	5	4

From the treated fabrics at 160 ^{O}C curing temperature in wet condition color staining observed along the warp and weft directions were rated in range of 3 up to 4-5 and in dry condition color staining evaluated along the warp and weft directions were rated in range of 4-5 up to 5. The staining ratings are good when evaluated in dry condition. The results indicated that an improvement in rubbing fastness was observed with an increase in the amount of binder applied, curing temperature and time.

4.6 Fabric Stiffness Test

The stiffness of a fabric was determined by bending length and flexural rigidity of fabrics. The results are displayed Table 4.12 - 4.14

Table 4.12 Bending Length and Flexural Rigidity of Treated Fabrics at 140 ^{O}C Curing Temperature (Standard : ASTM D 1388-96)

Sample Number	Amount of Chemical (g/l)	Curing Time (min.)	Stiffness			
			Warp direction		Weft direction	
			Bending Length (cm)	Flexural Rigidity (mg cm)	Bending Length (cm)	Flexural Rigidity (mg cm)
-	-	-	1.025	868	0.750	328
1	10	1	1.113	1,070	0.963	516
2		2	1.175	1,206	0.963	541
3		3	1.206	1,335	0.975	562
4		4	1.219	1,274	0.975	590
5		5	1.225	1,519	1.025	630
6	20	1	1.150	1,230	0.975	471
7		2	1.200	1,378	0.988	543
8		3	1.206	1,347	1.000	582
9		4	1.213	1,375	1.025	588
10		5	1.225	1,542	1.050	646
11	30	1	1.194	1,317	0.975	636
12		2	1.206	1,403	1.013	706
13		3	1.213	1,393	1.013	693
14		4	1.219	1,514	1.038	780
15		5	1.225	1,624	1.063	809
16	40	1	1.194	1,352	0.963	751
17		2	1.200	1,432	1.025	783
18		3	1.213	1,416	1.050	809
19		4	1.231	1,546	1.050	861
20		5	1.256	1,671	1.063	874
21	50	1	1.206	1,431	0.775	670
22		2	1.225	1,595	0.800	751
23		3	1.231	1,452	1.063	804
24		4	1.250	1,584	1.088	808
25		5	1.269	1,737	1.100	887

Table 4.12 shows the effects curing temperature, curing time and amount of binder on the stiffness property (flexural rigidity) of treated fabric. The results show that the flexural rigidity expressed in mg/cm increase with an increase in curing temperature, the amount of binder and curing time. For example, at the amount of binder of 50 g/l, the flexural rigidities are 1,737 mg cm when correspond to the curing temperatures of 140 ^{O}C and curing time 5 minutes, respectively. The higher values indicate the poor hand properties of treated fabric such as stiffness as a result of the presence of binder film on fabric surface.

Table 4.13 Bending Length and Flexural Rigidity of Treated Fabrics at 150 ^{O}C Curing Temperature (Standard : ASTM D 1388-96)

Sample Number	Amount of Chemical (g/l)	Curing Time (min.)	Stiffness			
			Warp direction		Weft direction	
			Bending Length (cm.)	Flexural Rigidity (mg cm.)	Bending Length (cm.)	Flexural Rigidity (mg cm.)
-	-	-	1.025	868	0.750	328
1	10	1	0.888	1,196	0.913	662
2		2	1.013	1,232	0.925	673
3		3	1.038	1,285	0.938	695
4		4	1.238	1,312	0.938	722
5		5	1.288	1,541	0.963	781
6	20	1	1.050	1,331	0.875	650
7		2	1.100	1,424	0.925	676
8		3	1.138	1,500	0.925	724
9		4	1.150	1,505	0.925	771
10		5	1.150	1,562	0.963	804
11	30	1	1.138	1,443	0.963	691
12		2	1.163	1,573	0.963	725
13		3	1.188	1,650	0.975	764
14		4	1.238	1,754	1.000	821
15		5	1.250	1,784	1.025	891
16	40	1	1.150	1,504	1.000	646
17		2	1.225	1,742	1.025	787
18		3	1.225	1,761	1.025	862
19		4	1.275	2,146	1.038	898
20		5	1.275	1,894	1.038	964
21	50	1	1.200	1,732	0.888	733
22		2	1.275	2,244	0.988	859
23		3	1.300	2,219	1.013	951
24		4	1.313	2,393	1.013	999
25		5	1.363	2,447	1.050	1,046

Table 4.13 the stiffness property of treated fabric at 150 ^{O}C curing temperature. The results show that the flexural rigidity increase with an increase in curing temperature, the amount of binder and curing time. For example, at the amount of binder of 50 g/l, the flexural rigidities are 2,447 mg cm when correspond to curing time 5 minutes. The higher values indicate the poor hand properties of treated fabric as a result of the presence of binder film on fabric surface.

Table 4.14 Bending Length and Flexural Rigidity of Treated Fabrics at 160 ^{O}C Curing Temperature (Standard : ASTM D 1388-96)

Sample Number	Amount of Chemical (g/l)	Curing Time (min.)	Stiffness			
			Warp direction		Weft direction	
			Bending Length (cm.)	Flexural Rigidity (mg cm.)	Bending Length (cm.)	Flexural Rigidity (mg cm.)
-	-	-	1.025	868	0.750	328
1	10	1	1.325	2,311	1.088	967
2		2	1.388	2,612	1.100	1,138
3		3	1.400	2,626	1.113	1,203
4		4	1.400	2,756	1.175	1,350
5		5	1.456	3,233	1.175	1,374
6	20	1	1.338	2,385	1.088	1,063
7		2	1.338	2,560	1.125	1,114
8		3	1.356	2,641	1.138	1,143
9		4	1.419	2,941	1.138	1,132
10		5	1.431	2,944	1.163	1,157
11	30	1	1.394	2,774	1.113	1,098
12		2	1.400	2,799	1.125	1,093
13		3	1.425	3,323	1.125	1,111
14		4	1.463	3,310	1.138	1,177
15		5	1.506	3,971	1.163	1,242
16	40	1	1.375	2,828	1.088	1,037
17		2	1.500	3,916	1.150	1,203
18		3	1.506	3,724	1.188	1,350
19		4	1.544	4,381	1.188	1,463
20		5	1.606	4,896	1.225	1,470
21	50	1	1.431	2,913	1.063	999
22		2	1.481	3,606	1.125	1,145
23		3	1.563	4,032	1.150	1,274
24		4	1.600	4,573	1.175	1,281
25		5	1.600	5,078	1.200	1,288

Table 4.14 shows the stiffness property of treated fabric at 160 ^{O}C curing temperature. The results show that the flexural rigidity increase with an increase in the amount of binder and curing time. For example, at the amount of binder of 50 g/l and curing time 5 minutes, the flexural rigidities are 5,078 mg cm. The higher values indicate the poor hand properties of treated fabric such as stiffness as a result of the presence of binder film on treated fabric surface.

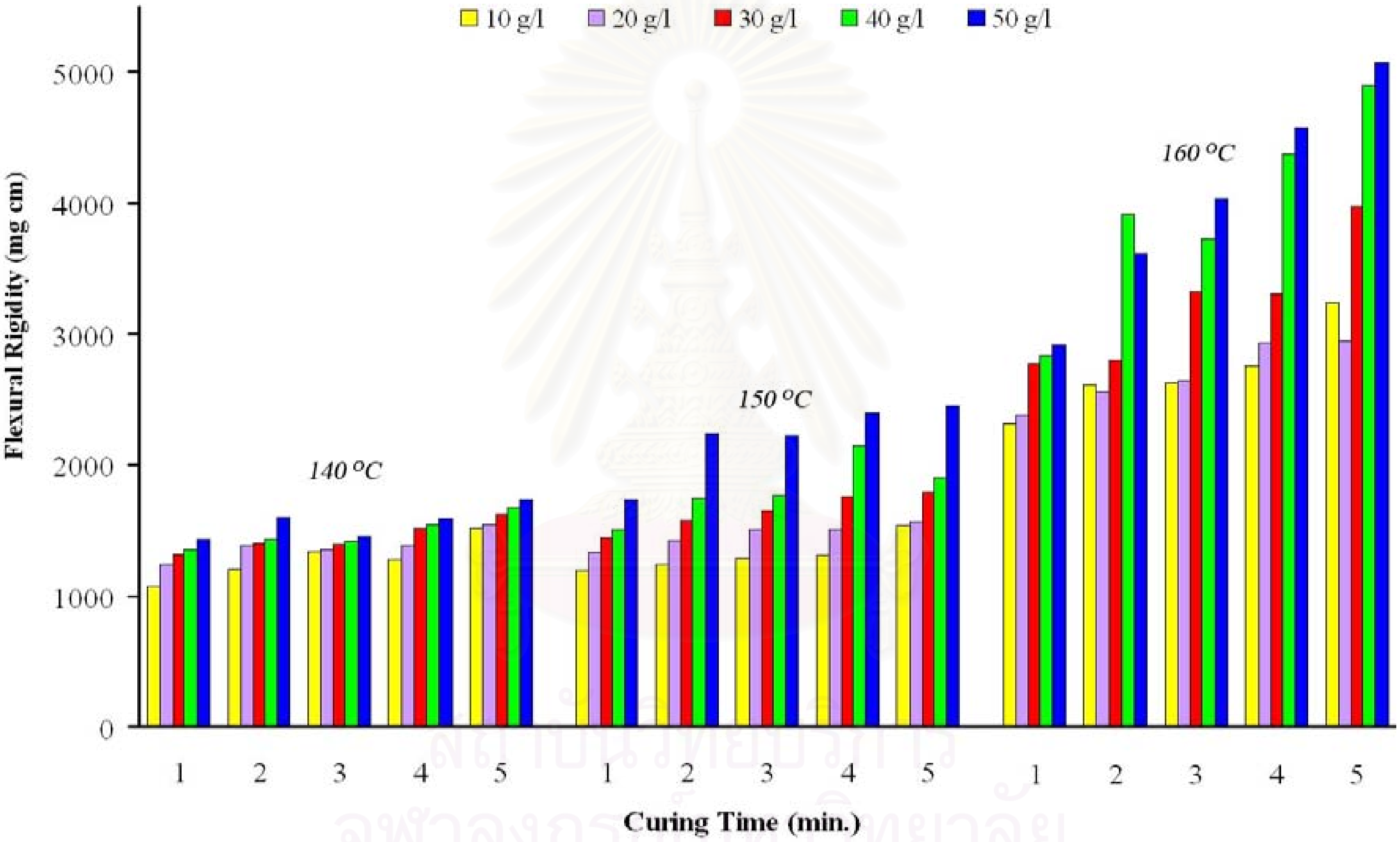


Figure 4.7 Flexural Rigidity of Treated Fabrics in Warp Direction**.**

4.7 Fabric Wicking Rate

Fabric wicking rate of treated fabrics are illustrated as following

Table 4.15 Fabric Wicking Rate of Treated Fabrics at 1 minute and 5 minutes

Curing Temperature (^{O}C)	**Amount of Chemical (g/l)**	**Fabric Wicking Rate (cm.)**									
		1 min.					**5 min.**				
		Curing Time (min.)									
		1	**2**	**3**	**4**	**5**	**1**	**2**	**3**	**4**	**5**
-	-	9.2					13.7				
140	10	7.2	6.8	6.5	6.4	6.1	12.6	12.4	12.3	12.0	11.9
	20	6.9	6.7	6.5	6.1	6.0	12.4	12.3	12.0	12.0	11.8
	30	6.8	6.6	6.5	6.3	6.3	12.5	12.3	12.2	12.0	11.8
	40	6.5	6.4	6.2	6.1	6.0	12.3	12.2	12.0	11.8	11.7
	50	6.3	6.0	6.0	5.8	5.7	12.2	11.7	11.5	11.4	11.2
150	10	7.0	6.8	6.6	6.3	6.0	12.7	12.3	12.1	12.0	12.0
	20	6.8	6.5	6.4	6.2	6.0	12.5	12.3	12.3	12.0	11.8
	30	6.5	6.4	6.3	6.2	6.0	12.3	12.1	12.0	11.8	11.6
	40	6.3	6.3	6.1	6.1	5.9	12.2	12.0	11.8	11.8	11.6
	50	6.2	6.1	6.0	6.0	5.8	12.0	11.8	11.7	11.7	11.5
160	10	6.8	6.6	6.3	6.2	6.0	12.4	12.2	12.1	12.0	11.9
	20	6.5	6.3	6.0	6.0	5.8	12.3	12.2	12.0	11.8	11.5
	30	6.3	6.2	6.0	5.8	5.7	12.2	12.0	11.8	11.6	11.3
	40	6.3	6.0	5.8	5.6	5.5	12.0	11.9	11.7	11.5	11.4
	50	6.0	5.8	5.7	5.5	5.3	11.9	11.7	11.5	11.5	11.3

From Table 4.15, the fabric wicking rate was decreased when curing temperature, curing time and amount of finishing agent increased. Due to prolong curing and high fixing temperature were develop binder's film full adhesion to fabric and coverage of binder's film on the surface of treated fabric as a result to decrease the adsorption properties of fabric.

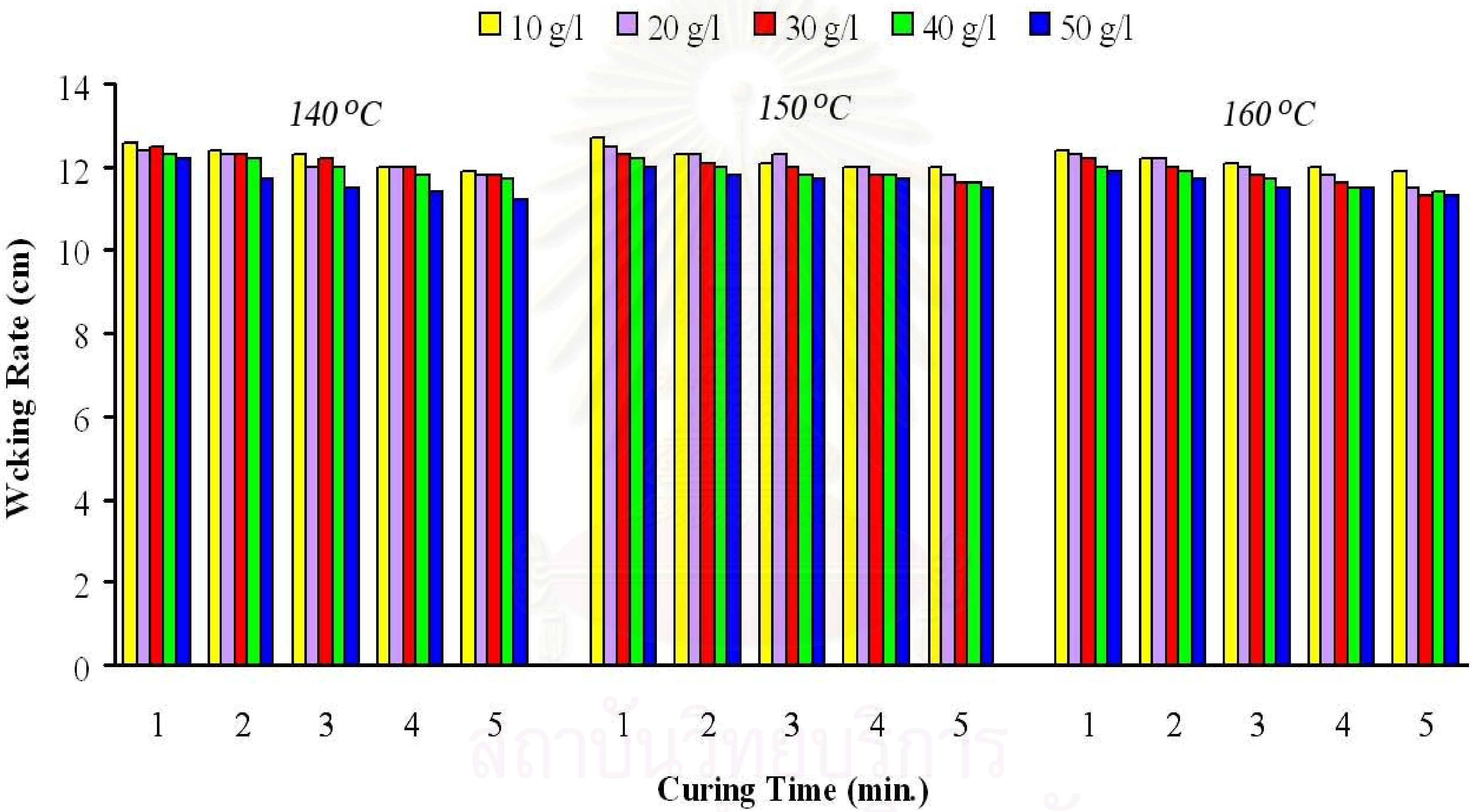


Figure 4.8 Fabric Wicking Rate of Treated Fabrics after 5 Minutes.

4.8 Air Permeability Test

Air permeability of treated fabrics are displayed Table 4.16

Table 4.16 Air Permeability of Treated Fabrics

Curing Temperature	Amount of Chemical (g/)	Air Permeability (mm/sec)				
		Curing Time (min.)				
		1	2	3	4	5
-	-	312.4				
140	10	257.1	286.7	245.2	265.0	270.9
	20	221.5	237.3	218.3	221.5	213.6
	30	215.5	217.5	213.6	207.2	187.9
	40	213.6	214.8	211.6	192.2	185.9
	50	183.5	174.4	178.0	162.2	168.1
150	10	253.1	239.3	209.6	212.0	225.4
	20	243.2	212.8	195.8	195.4	217.1
	30	192.2	195.4	195.0	189.0	166.9
	40	185.1	194.6	188.3	187.9	166.5
	50	168.1	170.1	180.3	174.8	165.3
160	10	220.3	208.0	213.6	206.8	222.7
	20	209.6	194.2	194.6	191.4	215.5
	30	198.1	191.0	192.6	185.1	202.1
	40	196.6	189.0	191.8	183.5	187.5
	50	187.9	175.6	159.0	153.1	168.1

The result indicated all specimen no wind protection (air permeability of fabric > 50 mm/sec). The amount of binder, fixation temperature and curing time were found to influence on air permeability. When the amount of binder, temperature and curing time were increased, air permeability of fabric was decreased. It is because of the coverage of binder film on the surface of fabric as a result to decrease the air permeability.

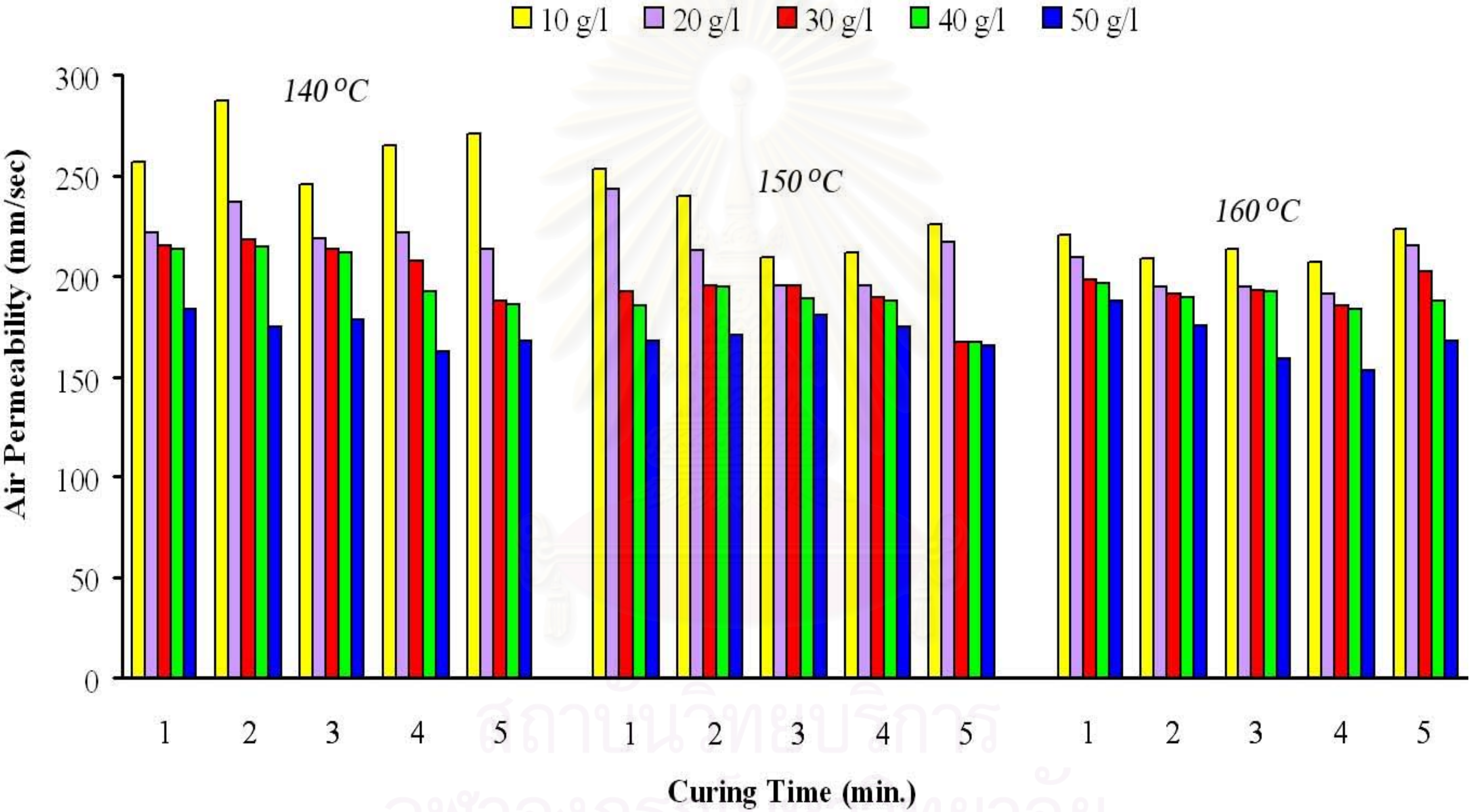


Figure 4.9 Air Permeability of Treated Fabrics.

CHAPTER V

CONCLUSIONS AND SUGGESTION

5.1 Conclusion

The research studied feasibility in fixation of activated carbon powder from coconut shell on knitted cotton fabrics using butadiene-acrylonitrile copolymer binder. The application was carried out by using a pad-dry-cure technique and used Acramin BA-N (Butadiene-acrylonitrile copolymer as a binder in ratio activated carbon powder per binder equal 1:1 The fixation was performed at the temperature ranging from 140 - 160 ^{O}C. The adsorption efficiency of treated fabrics was evaluated by percent formaldehyde absorption. Wash fastness and rubbing fastness as well as properties evaluation of treated fabrics were performed.

The results showed, in fixation process, binders were transformed into polymer network film where activated carbon powder particles resided. The binder exhibited the negative effect on the adsorption capability of activated carbon. When the amount of binder, temperature and curing time were increased, judged by the reductions of iodine number and percent formaldehyde absorption. After washing, the absorption efficiency of fixed activated carbon was recovered. This phenomenon was believed that during washing crack and peel-off of binder film might occur, allowing an increase in the adsorption capacity observed.

The appropriate conditions for fixation of activated carbon on cotton fabric were recommended as follows : the amount of binder of 50 grams per liter, fixation temperature of 150 ^{O}C and curing time of 4 minutes. Another condition such as at 160 ^{O}C, treated fabric would have good fastness properties but adsorption capacity of treated fabric was lower than 150 ^{O}C and some properties the fabric was not suitable for use

5.2 Suggestion for further work

1. To study with another type of binder and various type of activated carbon powder.
2. To study with another technique for finishing process such as coating, printing.
3. To study about durability of treated fabric and anitmicrobial properties of treated fabrics

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APPENDICES

APPENDIX A

PRODUCTION OF THE CALIBRATION CURVE FOR FORMALDEHYDE DETERMINATIONS

Purpose

The calibration curve is used to calculate the concentration of formaldehyde in the unknown liquors produced from textiles.

Reagents

1. Distilled water
2. Acetyl acetone reagent. Dissolve 150 g ammonium acetate in about 800 ml water, add 3.0 ml glacial acetic acid and 2.0 ml acetyl acetone. Transfer to a 1000 m1 volumetric flask and make up to the mark with water. Store in a brown bottle (See Note 1).
3. Formaldehyde solution. Approximately 37% (W/W)

Apparatus

1. Glass pipette 10.0ml.
2. Burette 50.0m1 with graduations at intervals of 0.1 ml.
3. Volumetric flasks 50 ml, 250 ml, 500 m1 and 1,000 ml.
4. Pipette 5.0 m1 (see Note 2).
5. Thermostatically controlled water bath capable of maintaining uniform temperature of 40 ±1°C throughout.
6. Test tubes (150 x 18mm) and suitable means of support for immersion in water bath at 40°C.
7. Filter photometer or spectrophotometer, which gives readings to three decimal places at wavelength 412 nm.

Method

1. Prepare a stock solution containing approximately 2,500 µg/ml formaldehyde accurately measure 6.7 ml of 37% formaldehyde solution into a 1 liter volumetric flask and make up to volume with distilled water. Allow the stock solution to stabilize for at least 48 hours before use. The stock solution is stable for at least 6 weeks if refrigerated at 4 ^{O}C. See Note 3.

2. Determine accurately the concentration of formaldehyde in the stock solution by the standardization method given in Appendix B (see Note 3).

3. Prepare an intermediate standard containing a nominal concentration of 100 µg/ml formaldehyde: Pipette 10.0 m1 of the standardized stock solution into a 250ml volumetric flask using a clean, single mark glass pipette, and make up to volume with distilled water. The actual concentration is calculated from the standardizations of the stock solution. The intermediate standard is not stable and should be freshly prepared before use (see Note 4).

4. Prepare a series of calibration standards using a clean 50 ml burette. Dispense the freshly prepared intermediate standard accurately into volumetric flasks according to the following table, and make up to the mark with distilled water (see Note 5).

Intermediate Standard Solution (a) (ml)	**Dilution Volume (a) (ml)**	**Calibration Standard Solution (Nominal µg/ml)[b, c]**	**Equivalent level of Formaldehyde on Fabric (for information only)**	
			Free & Hydrolvzed (mg/kg)	**Released (mg/kg)**
2.0	1000	0.20	8	10
2.5	500	0.50	20	25
5.0	500	1.00	40	50
12.5	500	2.50	100	125
5.0	100	5.00	200	250
7.5	100	7.50	300	375
10.0	100	10.00	400	500

(a) Volume of intermediate standard solution and dilution volume may be adjusted to give required calibration standard concentration.

(b) The nominal concentrations should be adjusted to give actual concentrations derived by standardization of the stock solution (see Appendix B).

(c) The calibration standards are not stable and should be prepared freshly before use

5. Mix each flask thoroughly. Pipette 5.0 ml of each calibration standard into a clean, dry test tube, add 5.0 ml acetyl acetone reagent and shake to mix.

6. To prepare a reagent blank, pipette 5.0 ml distilled water into a clean, dry test tube, add 5.0 ml acetyl acetone reagent and shake to mix.

7. Place the tubes containing the analysis solution and reagent blank in the water bath at 40 ± 1^{O}C for 30 ± 5 minutes so that liquid is below the level of the water in the water bath, remove and allow to cool for 30 minutes.

8. Measure the absorbance of the color-developed solution using a spectrophotometer in a clean 10mm cell at 412nm against the reagent blank (see Notes 6 and 7).

9. Plot a calibration curve of actual formaldehyde concentration (obtained by reference to the standardized stock solution) (µg/ml) against measured absorbance, and calculate the first order regression curve

Calculation

The concentration corresponding to the absorbance of a color-developed solution is derived from the calibration data:

(a) by reading directly from the calibration curve or

(b) by calculating from the first order regression equation $y = ax + b$ (see Note 8).

For liquors requiring dilution to bring within calibration, the dilution factor is used to calculate the concentration in the original undiluted liquor.

The method for calculating the amount of formaldehyde contained in the fabric is given in the individual analytical procedures.

Notes

1. Acetyl acetone reagent: Allow to age for at least 12 hours before use. The reagent is stable for up to six weeks if stored in a brown bottle.

2. 5.0 m1 pipette: An automatic graduated pipette is suitable for the 5.0 ml aliquots of calibration standards and acetyl acetone reagent. Manual graduated pipettes are unacceptable.

3. Stock solution: In order that accurate volumes are dispensed, the refrigerated stock solution must be allowed to reach room temperature before use.

4. Intermediate standard: Ensure the 10 ml glass pipette is thoroughly rinsed at least 3 times with stock solution before preparing the intermediate standard.

5. Calibration standards: Ensure burette is thoroughly rinsed with at least 3 full burettes of the intermediate standard solution before preparing calibration standards.

6. Color developed solution: The color-developed solution may fade if exposed to direct sunlight. If there is appreciable delay (say longer then 1 hour) before reading absorbance, keep the tubes covered or place in a dark cupboard. Otherwise, the color is stable for up to 16 hours and reading may be delayed if desired.

7. Maximum absorbance: The upper limit of absorbance for color-developed solutions prepared from the top calibration standard containing 10 µg/ml is around 1.3 absorbance units for a 10 mm cell at a wavelength of 412 nm

8. Calibration curve: Typical calibrations have been found to be linear up to at least 1.3 absorbance units according to the following equation:

$$\text{Formaldehyde concentration } (\mu g/ml) = \text{absorbance} \times 7.5 \pm 0.2$$

9. Frequency of calibration: To ensure good internal quality control, personnel and laboratories new to the methods should carry out the standardization and calibration procedures every 3 months for 1 year, and then once a year unless a new batch of acetyl acetone is purchased, or the spectrophotometer is changed or recalibrated.

APPENDIX B

STANDARDIZATION OF FORMALDEHYDE SOLUTION

Purpose

The standardized stock formaldehyde solution is used in Appendix 1 to produce the calibration curve that determines the concentration of formaldehyde in aqueous liquors derived from textiles.

Reagents

1. Distilled water
2. Standard sodium thiosulphate solution 0.1 mol/l (0.1 N) Commercially available concentrate. Using a volumetric flask, make up accurately to the volume specified by the chemical supplier.
3. Standard iodine solution 0.05 mol/1 (0.1N) Commercially available concentrate. Using a volumetric flask, make up accurately to the volume specified by the chemical supplier.
4. Sodium hydroxide solution 2.0 mol/1 (2.0N) Carefully dissolve sodium hydroxide solid (80 g) in approximately 900 ml water, allow to cool, pour into a measuring cylinder and make up to 1 liter with distilled water.
5. Sulphuric acid solution: 1.0 mol / I (2.0N) Carefully add concentrated Anala R grade sulphuric acid (100 g or 55 ml) to approximately 900ml water, allow to cool, pour into a measuring cylinder and make up to 1 liter with distilled water.
6. Starch indicator: Use one of the following *alternative methods:*
    - (i) Paste soluble starch (1.0g) with a few drops of distilled water. Add 100 ml boiling distilled water, stir and boil for one minute, Cool and add potassium iodide (3.0 g). Do not store.
    - (ii) A stable solution of starch is prepared from a dispersion of soluble starch (10 g) in 180 ml formamide. Pour whilst stirring into 130 ml hot formamide (100 – 110 ^{O}C) to dissolve the starch. The solution is allowed to cool before use. Prepare this starch indicator in a fume cupboard.
7. Formaldehyde solution. Approximately 37% (W/W) commercially available.
8. Stock solution containing approximately 2500 μg/ml formaldehyde: Accurately measure 6.7m1 of 37% formaldehyde solution into a 1 liter volumetric flask and make up to volume with distilled water. Allow the stock solution to stabilize

for at least 48 hours before to standardization. The stock solution is stable for at least 6 weeks if refrigerated at 4°C (see Note 1).

Apparatus

1. Measuring cylinder 1 liter
2. Glass pipettes 10.0 and 50.0 ml.
3. Burette 50.0 ml with graduations at intervals of 0.1 ml.
4. Volumetric flasks 500 ml and 1,000 ml.
5. Graduated pipettes up to 10 ml (see Note 2).
6. Conical flasks 250 ml

Method

1. Pipette 50.0m1 of 0.05 mo/l iodine into a 250 m1 flask. Add accurately 10.0 ml of the stock formaldehyde solution followed by 10.0 ml 2.0 mol/l sodium hydroxide. Shake the flask and allow to stand at room temperature for 15 minutes. The solution becomes colorless.
2. Add 15m1 1.0 mol/l sulphuric acid. The solution becomes brown due to liberated iodine (see Note 3).
3. Fill the burette with 0.1 mol/l sodium thiosulphate and titrate into the liberated iodine until the solution in the flask becomes a pale yellow color (see Note 4).
4. Add starch indicator (4-5 drops). The solution becomes dark blue. Continue titrating slowly and carefully (drop wise) until the solution becomes just colorless. Record the volume of sodium thiosulphate used.I5. Repeat steps 1 to 4 until three consecutive liters agree to within 0.1ml. Calculate the average value T_1.
6. Carry out three blank titrations in a similar manner, but omitting the stock formaldehyde solution. Calculate the average blank value T_b.

Calculation

1.0 m1 of 0.1 mol/l sodium thiosulphate solution (titrant) is equivalent to 1.0 m1 of 0.05 mol/l iodine solution, and this is equivalent to 0.0015015 g formaldehyde.

$$\text{Formaldehyde in stock solution} = (T_b - T_1) \times 150.15\ \mu g/ml$$

Where: T_b = Blank Titer and Sample Titer

Notes

1. Stock solution: In order that accurate volumes are dispensed, the refrigerated stock solution must equilibrate to room temperature before use.

2. Graduated pipettes: Automatic or glass pipettes graduated at 0.1 ml intervals are suitable for dispensing the 37% formaldehyde solution, and for adding the sodium hydroxide and sulphuric acid reagents during the titration procedure.

3. Liberated iodine: In standardizing the stock formaldehyde solution, it is essential that the solution in the flask is acidic before the titration takes place, to ensure liberation of all unreacted iodine.

4. Titrant: Ensure burette is thoroughly rinsed with at least 3 full burettes of the standard sodium thiosulphate solution before titrating.

APPANDIX C

DETERMINATION OF IODINE NUMBER

Reagents

1. *Hydrochloric Acid Solution (5% by weight)*

Add 70 ml of concentrated hydrochloric acid to 550 ml of water, and mix well.

2. *Potassium Iodate Solution (0.1000N)*

Dry 4 or more grams of primary standard-grade potassium iodate ($KI0_3$) at 110° ± 5° for 2 h, and cool to room temperature in a desiccator. Dissolve 3.5667 ± 0.1 mg of the dry potassium iodate in about 100 ml of water. Quantitatively transfer to a 1-L volumetric flask, dilute to volume with water, and mix thoroughly. Store in a glass-stoppered bottle.

3. *Starch Solution*

Mix 1.0± 0.5 g of starch with 5 to 10 ml of cold water to make a paste. Continue to stir while adding an additional 25 ± 5 ml of water to the starch paste. Pour the mixture, while stirring, into 1 L of boiling water, and boil for 4 to 5 min. Make this solution fresh daily.

4. *Sodium Thiosulphate Solution (0.100 N)*

Dissolve 24.820 g of sodium thiosulphate in approximately 75 ± 25 ml of freshly boiled water, and add 0.10 ± 0.01 g of sodium carbonate. Quantitatively transfer the mixture to a 1-L volumetric flask, and dilute to volume with water. Allow the solution to stand for a minimum of 4 days before standardizing. Store the solution in an amber bottle. To standardize the solution, perform the following in triplicate: Pipette 25.0 ml of 0.1000 N Potassium Iodate Solution into a wide-mouthed Erlenmeyer flask. Add 2.00 ± 0.01 g of potassium iodide, and shake the flask to dissolve the potassium iodide crystals. Pipette 5.0 ml of concentrated hydrochloric acid into the flask, and titrate the free iodine with Sodium Thiosulphate Solution to a light yellow color. Add a few drops of Starch Solution, and continue the titration until 1 drop produces a colorless solution. Determine the Sodium Thiosulphate Solution normality using the following formula:

$$(P \times R)/S$$

in which P is the volume, in milliliters, of 0.1000 N Potassium Iodate Solution; R is the normality of the 0.1000 N Potassium Iodate Solution; and S is the volume, in milliliters, of Sodium Thiosulphate Solution. Average the three normality results; repeat the test if the range of values exceeds 0.003 N.

5. *Standard Iodine Solution (0.100 ± 0.001 N)*

Transfer 12.700 g of iodine and 19.100 g of potassium iodide (KI), accurately weighed, into a beaker, and mix. Add 2 to 5 ml of water, and stir well. While stirring, continue to add small increments, approximately 5 ml each, of water until the total volume is 50 to 60 ml. Allow the solution to stand a minimum of 4 h to ensure crystal dissolution, stirring occasionally. Quantitatively transfer the solution to a 1-L volumetric flask, and dilute to volume with water. The iodide-to-iodine weight ratio must be 1.5:1. Store the solution in an amber bottle.

Note : Standardize this solution just before use. To standardize this solution, perform the following in triplicate. Pipette 25.0 ml into a 250-ml wide-mouthed Erlenmeyer flask. Titrate with the standardized Sodium Thiosulphate Solution until a light yellow color develops. Add a few drops of Starch Solution, and continue the titration until 1 drop produces a colorless solution. Determine the Iodine Solution normality using the following formula:

(S x N,)/I,

in which S is the volume, in milliliters, of the standardized Sodium Thiosulphate Solution; N is the normality of the standardized Sodium Thiosulphate Solution; and I is the volume, in milliliters, of Iodine Solution. Average the three normality results; repeat the test if the range of values exceeds 0.003 N. The standardized Iodine Solution concentration must be 0.100 ± 0.001 N. If it is not, repeat all of the steps starting from, "Transfer 12.700 g of iodine......"

Procedure

This procedure applies to both powdered and granular sample. A representative sample of powdered sample may need additional grinding until 60 wt % (or more) passes through a 325-mesh screen and 95 wt % (or more) passes through a 100-mesh screen. Grind a representative sample sufficiently to pass through the screens as described above. Dry the ground sample, and cool to room temperature in a desiccator. Three dosages of sample must be estimated to determine the iodine number. Weigh the three dosages (M) of dry carbon to the nearest milligram. Transfer each to one of three clean, dry 250-ml Erlenmeyer flasks equipped with ground glass stoppers. Pipette 10.0 ml of Hydrochloric Acid Solution into each flask, stopper each flask, and swirl gently until the carbon is completely wetted. Loosen the stoppers to vent the flasks, place on a hot plate in a fume hood, and bring the contents to a boil. Allow to boil gently for 30 ± 2 s to remove any sulfur (which may interfere with the test results). Remove the flasks from the hot plate and cool to room temperature. Standardize and then pipette 100.0 ml of Iodine Solution into each flask.

Note : Stagger the addition of standardized Iodine Solution to the three flasks so that no delays are encountered in handling.

Immediately stopper the flasks, and shake the contents vigorously for 30 ± 1 s. Quickly filter each mixture by gravity through one sheet of folded filter paper (Whatman No. 2V, or equivalent) into one of three beakers.

Note : Prepare the filtration equipment in advance to avoid delays in filtering the samples. For each filtrate, use the first 20 to 30 ml to rinse a pipette, and discard the rinse portions. Use clean beakers to collect the remaining filtrates. Mix each filtrate by swirling the beaker, and pipette 50.0 ml of each filtrate into one of three clean 250-ml Erlenmeyer flasks. Titrate each filtrate with standardized Sodium Thiosulphate Solution until a pale yellow color develops. Add 2 ml of Starch Solution, and continue the titration with standardized Sodium Thiosulphate Solution until 1 drop produces a colorless solution. Record the volume (S) of standardized Sodium Thiosulphate Solution used.

Calculation

The capacity of a carbon for any adsorbate depends on the concentration of the adsorbate. The concentrations of the standard iodine solution and filtrate must be known to determine an appropriate carbon weight to produce final concentrations agreeing with the definition of iodine number. The amount of sample to be used in the determination is governed by the activity of the sample. If filtrate normalities (C) are not within the range of 0.008 N to 0.040 N, repeat the procedure using different sample weights. Once filtrate normalities are set within the specified range, perform the following calculations for each carbon dosage:

$$A = (N_2)(12693.0)$$

in which N_2 is the normality of the standardized Iodine Solution.

$$B = (N_l)(126.93)$$

in which N_1 is the normality of the standardized Sodium Thiosulphate Solution. Calculate the dilution factor (DF) using the equation

$$DF = (I + H)/F$$

in which I is the volume, in milliliters of Iodine Solution used in the standardization procedure, H is the volume in milliliters of Hydrochloric Acid Solution used and F is the volume in milliliters of filtrate used.

Calculate the weight in milligrams of iodine adsorbed per gram of sample (X/M) by the equation

$$X/M= [A - (DF)(B)(S)]/M$$

in which S is the volume, in milliliters, of standardized Sodium Thiosulphate Solution used, and M is the weight, in grams, of the sample.

Calculate the normality of the residual filtrate (C) as follows:

$$C = (Ni\ X\ S)/F.$$

Using logarithmic paper, plot X/M (as the ordinate) versus C (as the abscissa) for each of the three carbon dosages. Calculate the least squares fit for the three points, and plot. The iodine number is the X/M value at a residual iodine concentration (C) of 0.02 N. The regression coefficient for the least squares fit should be greater than 0.995. Carbon dosages may be estimated initially by using three values of C (usually 0.01, 0.02, and 0.03) as follows:

$$M= [A - (DF)(C)(126.93)(50)]/E$$

in which M is the weight, in grams, of the carbon dosage and E is the nominal iodine number of the sample. If new carbon dosages have been determined, repeat the Procedure and Calculations.

VITA

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