

ADSORPTION STUDIES OF SYNTHETIC BATIK
DYE (SBD) USING GRANULAR ACTIVATED
CARBON (GAC): BATCH PROCESS

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BORANG PENGESAHAN STATUS TESIS

JUDUL : ADSORPTION STUDIES OF SYNTHETIC BATIK DYE (SBD) USING GRANULAR ACTIVATED CARBON (GAC): BATCH PROCESS

SESI PENGAJIAN : 2011/2012

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GRANULAR ACTIVATED CARBON (GAC): BATCH PROCESS

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Thesis submitted in fulfillment of the requirements
for the award of the degree
of Bachelor of Chemical Engineering in Chemical Engineering

Faculty of Chemical & Natural Resources Engineering
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JUNE 2012

SUPERVISOR DECLARATION

I hereby declare that I have been read through this project report and to my opinion this report is adequate in term of scope and quality for the purpose of awarding the degree of Bachelor of Chemical Engineering.

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I declare that this report entitled “Adsorption Studies of Synthetic Batik Dye (SBD) Using Granular Activated Carbon (GAC): Batch Process” is the result of my own research except this as cited in the references. The report has not been accepted for any degree and is not currently submitted in candidature of any other degree.

Signature : _____

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*Special dedication to my mum and dad that always inspire, love and stand beside me,
and to my beloved friend.*

Thank you for all your love, care and support.

ACKNOWLEDGEMENT

In the name of Allah S.W.T the Most Beneficent and the Most Merciful. The deepest sense of gratitude to the Almighty for the strength and ability to complete this project. Infinite thanks we brace upon Him.

I would like to take this opportunity to extend my deepest gratitude to all the parties involved in this project. First of all a special appreciation to my supervisor, Dr Anwaruddin Hisyam for his tireless efforts and on-going support, advise as well as encouragement, guidance, and critics in bringing this project to be successful. Without their outstanding support and interest, this report would not been at the best it would right now.

My special thankful goes to my beloved parents, Mr Ibrahim bin Mohamad @ Mohd and Mrs Norasiah Binti Junoh for their encouraged and supported me spiritually and financially during my year of study leading to completion of the thesis project. I know they always pray for my successful.

I would like to extend my heartfelt gratitude to the project coordinator, Miss Noraziah Bt Yazid and to all FKKSA lectures especially panels for presentation who offer tips, advice and endless cooperation. It is a special thanks to all teaching engineer and also lab assistant towards the accomplishment of my experiment. And sincere appreciation also extends to all my colleagues, housemates, and friends whom had provided assistance at various occasions.

I acknowledge my sincere indebtedness and gratitude to my loved ones for his love, dream and sacrifice throughout my life. My sincere thanks also go to all my friends including everyone who help me directly or indirectly in completing this thesis.

ABSTRACT

Batik industry is a very famous textile industry in Malaysia especially in East Coast of Peninsular Malaysia. Started in small scale, today it has become one of the highest economic growths in Malaysia due to high demands locally and from abroad. However, the wastewater from textile industry contributes to water pollution since it utilizes a lot of chemicals. Preliminary studies show that the wastewater from this homemade textile industry contains grease, wax, heavy metal, surfactant, suspended solid, and dyes (organic and inorganic). This research was done to study the potential of granular activated carbon (GAC) for the adsorption of synthetic batik dye (SBD) from aqueous solution by the variation of solution with different contact time, adsorbent dosage and initial concentration of SBD. Activated carbon (AC) is commonly known as one of the great adsorbents in adsorption process. For this study, granular activated carbon (GAC) has been used since it is preferred for all adsorption of gases and vapors as their rate of diffusion are faster. The synthetic batik dye has been chose because the research about this topic has not been done yet. There are three parameters that will be investigated; effect of adsorbent dosage (w), effect of contact time and effect of initial synthetic batik dye concentration. All results from the experiment were being analyzed using UV-Visible Spectrophotometer (UV-Vis). The results show that the color of the dye changes with different contact time, adsorbent dosage and also different initial concentration of synthetic batik dye. The data collected could be used to improve the effectiveness of dye removal from the batik industry wastewater using granular activated carbon (GAC). The research showed the applicability of GAC to adsorb the color of SBD. For SBD initial concentration shows the highest percentage concentration difference which is 69.75 % occurring at 0.2 g/L. While for adsorbent dosage, the highest dosage which is 60 gram get the highest percentage; 94.77 %. And for contact time, the longest contact time which is 20 minutes gets the highest percentage where the percentage is 95.06 %.

Key words: Granular Activated Carbon (GAC), Adsorption, Synthetic batik dye, Adsorbent, Batik Industries

ABSTRAK

Industri batik adalah industri tekstil yang sangat terkenal di Malaysia terutamanya di Pantai Timur Semenanjung Malaysia. Bermula secara kecil-kecilan, kini telah menjadi salah satu daripada pertumbuhan ekonomi tertinggi di Malaysia disebabkan oleh permintaan yang tinggi di dalam dan luar negara. Walau bagaimanapun, air sisa daripada industri tekstil menyumbang kepada pencemaran air kerana menggunakan bahan kimia yang banyak. Kajian awal menunjukkan bahawa air sisa daripada industri tekstil buatan sendiri ini mengandungi gris, lilin, logam berat, surfaktan, pepejal terampai, dan pewarna (organik dan tak organik). Kajian ini dilakukan untuk mengkaji potensi butiran karbon teraktif untuk penjerapan pewarna batik sintetik daripada larutan akueus oleh variasi penyelesaian dengan masa sentuhan yang berbeza, dos penjerap dan kepekatan awal pewarna batik sintetik. Butiran karbon teraktif biasanya dikenali sebagai salah satu penjerap terbaik di dalam proses penjerapan. Untuk kajian ini, butiran karbon teraktif telah digunakan kerana ia lebih sesuai untuk semua proses penjerapan gas dan wap kerana kadar resapan adalah lebih cepat. Pewarna batik sintetik telah dipilih kerana penyelidikan tentang topik ini tidak pernah dilakukan lagi. Terdapat tiga parameter yang akan dikaji; kesan dos penjerap, kesan masa sentuhan dan kesan awal kepekatan pewarna batik sintetik. Semua sampel dari eksperimen tersebut telah dianalisis dengan menggunakan *UV-Visible Spectrophotometer (UV-Vis)*. Hasil kajian menunjukkan bahawa warna bagi pewarna berubah dengan kadar masa sentuhan, kadar dos penjerap dan kadar awal kepekatan pewarna batik sintetik yang berbeza. Data yang dikumpul dapat digunakan untuk meningkatkan keberkesanan penyingkiran pewarna yang terdapat dalam air sisa industri batik dengan menggunakan butiran karbon teraktif. Kajian menunjukkan kesesuaian butiran karbon teraktif dapat menjerap warna pewarna batik sintetik. Untuk kepekatan awal pewarna batik sintetik menunjukkan peratusan perbezaan kepekatan tertinggi iaitu 69.75% berlaku di 0.2 g/L. Manakala bagi dos penjerap, dos tertinggi iaitu 60 gram mendapat peratusan tertinggi; 94.77%. Dan bagi masa sentuhan, masa sentuhan yang paling lama ialah 20 minit mendapat peratusan yang tertinggi sebanyak 95.06%.

Kata Kunci: Butiran karbon teraktif, Penjerapan, Pewarna batik sintetik, Penjerap, Industri Batik

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LIST OF SYMBOLS

%	Percentage
°C	Degree celcius
h	Hour
mL	Mililitre
g/L	gram/Litre
t	time
<i>C</i>	Concentration
<i>C_o</i>	Initial Concentration
w	Adsorbent dosage, weight
A	Absorbance
kg	kilogram
gm	gram
s	seconds
min	minutes

LIST OF ABBREVIATIONS

SBD	Synthetic Batik Dye
GAC	Granular Activated Carbon
PAC	Powdered Activated Carbon

CHAPTER ONE

INTRODUCTIONS

1.1 Introduction

This chapter explains about the research background, research objective and research scope of study. This chapter also explains about the research flow to get the sample of the research.

1.2 Research Background

Adsorption is the process through which a substance, originally present in one phase, is removed from that phase by accumulation at the interface between that phase and a separate (solid) phase. In principle adsorption can occur at any solid fluid interface. This is where the atoms, ions, biomolecules or molecules of gas, liquid or dissolved solid are stick or attached to the surface. The surface here defined as surface of the adsorbent. So the process will create film of adsorbate on the surface of the adsorbent. Adsorbate is the molecule or atoms that are being accumulated.

The driving force for adsorption is the reduction in interfacial (surface) tension between the fluid and the solid adsorbent as a result of the adsorption of the adsorbate on the surface of the solid. The surface or interfacial tension, σ , is the change in free energy, G , resulting when the area between two phases, A , is increased. The definition of σ is:

$$\sigma = \left[\frac{\partial G}{\partial A} \right]_{T,P,n_j}$$

In a bulk material, all the bonding requirements (be they ionic, covalent, or metallic) of the constituent atoms of the material are filled by other atoms in the material. However, atoms on the surface of the adsorbent are not fully surrounded by other adsorbent atoms and therefore can attract adsorbates. The exact nature of the bonding depends on the details of the species involved, but the adsorption process is generally classified as physisorption which means characteristic of weak van der Waals forces or chemisorption defined as the characteristic of covalent bonding. It may also occur due to electrostatic attraction.

Adsorption has been divided into two main applications which are liquid-phase adsorption and gas-phase adsorption. Removal of organic compounds from water or organic solutions, colored impurities from organics, and various fermentation products from fermentor effluents are the examples of liquid-phase adsorption. While for gas-phase adsorption, the examples are removal of water from hydrocarbon gases, sulfur compounds from natural gas, solvents from air and other gases, and odors from air.

Adsorbent is another term that is commonly related to adsorption. An adsorbent is a substance, usually porous in nature and with a high surface area that can adsorb substances onto its surface by intermolecular forces. Many adsorbents have been developed for a wide range of separations. Typically, the adsorbents are in the form of small pellets, beads, or granules where the size ranges from about 0.1 mm to 12mm in size. Larger size of adsorbents normally has been used in packed beds. A particle of adsorbent has a very porous structure, with many fine pores and pore volumes up to 50% of total particle volume. To be simplified, adsorbent act like a sponge where the structure has many pores and can absorb either liquid or gases. The adsorption

often occurs as a monolayer on the surface of the fine pores, although several layers sometimes will occur. Physical adsorption, or van der Waals adsorption, usually occurs between the adsorbed molecules and the solid internal pore surface and is readily reversible. (*Geankoplis, 2003*).

There are many types of commercial adsorbents nowadays. For example, activated carbon, silica gel, activated alumina, molecular sieve zeolites, synthetic polymers or resin etc. All of these adsorbents have been characterized by very large pore surface areas of 100 to over 2000 m²/g. Only at very low concentrations is the adsorption isotherm linear, at higher concentrations the adsorption isotherm may be Langmuir or Freundlich in nature. Due to the fact that solutes can distribute between the adsorbent surface and a mobile phase, adsorbents are used as a stationary phases in gas-solid and liquid-solid chromatography. Adsorbents are also used for extraction purposes removing traces of organic materials from large volumes of water very efficiently.

The overall adsorption process consists of a series of steps in series. When the fluid is flowing past the particle in a fixed bed, the solute first diffuses from the bulk fluid to the gross exterior surface of the particle. Then the solute will diffuses inside the pore to the surface of the pore. Finally, the solute is adsorbed on the surface. That is why it is called a series of steps of adsorption.

Dyes are substances that can be used to impart color to other materials, such as textiles, foodstuffs, and paper. Unlike pigments, dyes are absorbed to a certain extent by the material to which they are applied. The colors from some dyes are more stable than others, however. A dye that does not fade when the material it was applied to is exposed to conditions associated with its intended use is called a fast dye. Contrariwise, a dye that loses its coloring during proper usage is referred to as a fugitive dye. Some of the conditions that could cause such a change in the properties of a dye include exposure to acids, sunlight, or excessive heat as well as various washing and cleaning procedures. Certain dyes may be considered both fast and fugitive, depending on the material with which they are used. (*Michael W. Davidson, 2004*).

1.3 Problem Statement

Wastewater from batik industry that is not well-treated can cause harm to the environment especially water pollution since the waste is disposing to the river. Most of textile factory did not treat first the waste before disposing it and aquatic life got the effect from the waste. Preliminary studies show that the wastewater from this homemade textile industry contains grease, wax, heavy metal, surfactant, suspended solid, and dyes (organic and inorganic). Since the advanced technology for waste treatment is high cost, there is some research need to be done by using natural ingredients to save the earths and protect living things.

1.4 Research Objective(s)

To study the potential of granular activated carbon (GAC) for the adsorption of synthetic batik dye (SBD) from aqueous solution by the variation of solution with different contact time, adsorbent dosage and initial concentration of SBD.

1.5 Scope of research

In order to prove that granular activated carbon is the best adsorbent for the equilibrium studies on the adsorption of organic compounds, the scope of research is more focusing on:

- To study adsorption process of synthetic batik dye (SBD) by using granular activated carbon (GAC)
- To observe these three parameters (contact time, adsorbent dosage, and initial concentration to the adsorption of SBD by using GAC
- To analyze the concentration of synthetic batik dye using UV-Vis Spectrophotometer.

- To analyze the turbidity and color of the dye before and after the experiment using turbidity meter.

1.6 Significance of research

The equilibrium adsorption studies were carried out to evaluate the effect of adsorbent, contact time, and the SBD initial concentration. This research also can turn from waste to wealth and strike towards better and healthier lifestyle. The data collected could be used to improve the effectiveness of dye removal from the batik industry wastewater using adsorption process by using granular activated carbon (GAC). The synthetic batik dye has been choosing because the research involving this chemical has not been done yet and the waste from batik industry is easy to get in Malaysia.

CHAPTER TWO

LITERATURE REVIEW

2.1 Introduction

Adsorption process is one of the separation process consist of liquid-phase adsorption and gas-phase adsorption process. For liquid-phase, is used to separate or remove the liquid compound in the water or oil etc. While for gas-phase, is commonly used to remove or separate liquid from the gas phase. But both types of adsorption still needed adsorbent as an agent to absorb the fluid that we want. This research was done to study the potential of granular activated carbon (GAC) for the adsorption of synthetic batik dye (SBD) from aqueous solution by the variation of solution with different contact time, adsorbent dosage and initial concentration of SBD. There are three parameters that will be investigated; effect of adsorbent dosage (w), effect of contact time and effect of initial synthetic batik dye concentration. All results from the experiment were being analyzed using UV-Visible Spectrophotometer (UV-Vis) and the color changes are tested using turbidity meter. The results show that the color of the dye changes with different contact time, adsorbent dosage and also different initial concentration of synthetic batik dye.

2.2 Overview of Adsorption Process

Nowadays adsorption is commonly used as one of the separation process. One or more components of a gas or liquid stream are being adsorbed. In commercial processes, the adsorbent is usually in the form of small particles in a fixed bed. The process of adsorption occurs when the fluid is passed through the bed and the solid particles will adsorb the components from the fluid. When the bed is almost reaching saturated state, the flow in this bed is being stopped and the bed is regenerated thermally or by other methods to make sure that desorption is occur. Desorption is the reverse of adsorption. The adsorbed material or also known as adsorbate is thereby recovered and the solid adsorbent is ready for another cycle of adsorption process. Adsorption process is commonly used especially in gas-phase adsorption. For example, removal of water from hydrocarbon gases, sulfur compounds from natural gas, solvents from air and other gases, and odors from air. (*Geankoplis, 2003*).

When a gas comes into contact with a solid surface, molecules of the gas will adsorb to the surface in quantities that are function of their partial pressure in the bulk. The measurement of the amount of gas adsorbed over a range of partial pressures at a single temperature results in a graph known as an adsorption isotherm. There are three types of adsorption isotherms that is commonly used; Langmuir, Freundlich and linear isotherms. These isotherms can have very different shapes depending on the type of adsorbent, the type of adsorbate, and the intermolecular interactions between the gas and the surface. (*Findenegg, 1984*).

2.2.1 Factors Affect the Effectiveness of Adsorption Process

There are many factors that affecting the effectiveness of adsorption process. Below are the factors with few explanations.

- **Surface area of adsorbent**

Larger sizes of adsorbent will imply a greater adsorption capacity.

- **Particle size of adsorbent**

Smaller particle sizes reduce internal diffusional and mass transfer limitation to the penetration of the adsorbate inside the adsorbent (i.e., equilibrium is more easily achieved and nearly full adsorption capability can be attained). However, wastewater drop across columns packed with powdered material is too high for use of this material in packed beds. Addition of powdered adsorbent must be followed by their removal.

- **Contact time or residence time**

The longer the time the more complete the adsorption will be. However, the equipment will be larger.

- **Solubility of solute (adsorbate) in liquid (wastewater)**

Substances slightly soluble in water will be more easily removed from water (i.e., adsorbed) than substances with high solubility. Also, non-polar substances will be more easily removed than polar substances since the latter have a greater affinity for water.

- **Affinity of the solute for the adsorbent (carbon).**

The surface of activated carbon is only slightly polar. Hence non-polar substances will be more easily picked up by the carbon than polar ones.

- **Number of carbon atoms.**

For substances in the same homologous series a larger number of carbon atoms is generally associated with a lower polarity and hence a greater potential for being adsorbed (e.g., the degree of adsorption increases in the sequence formic-aceticpropionic-butyric acid).

- **Size of the molecule with respect to size of the pores.**

Large molecules may be too large to enter small pores. This may reduce adsorption independently of other causes.

- **Degree of ionization of the adsorbate molecule.**

More highly ionized molecules are adsorbed to a smaller degree than neutral molecules.

- **pH.**

The degree of ionization of a species is affected by the pH (e.g., a weak acid or a weak basis). This, in turn, affects adsorption.

2.3 Overview of Synthetic Batik Dye

2.3.1 Introduction of Synthetic Batik Dye

William Henry Perkin, an eighteen-year-old English chemist, was searching for a cure for malaria, synthetic quinine, and accidentally discovered the first synthetic dye. He found that the oxidation of aniline could color silk. From a coal tar derivative he made a reddish purple dye. The brilliant purple was called mauve. The dye was not stable to sunlight or water and faded easily to the color presently named mauve, a pale purple. This discovery resulted in additional research with coal tar derivatives and other organic compounds and an entire new industry of synthetic dyes was born. In the twenty-first century, synthetic dyes are less expensive, have

better colorfastness, and completely dominate the industry as compared with natural dyes. Thousands of distinctly different synthetic dyes are manufactured in the world (*Aspland, 1997*).

Before synthetic dyes were discovered in 1856, dyestuffs were manufactured from natural products such as flowers, roots, vegetables, insects, minerals, wood, and mollusks. Batches of natural dye were never exactly alike in hue and intensity, whereas synthetic dyestuffs can be manufactured consistently. The use of computers and computer color matching (CCM) produces color that is identical from batch to batch (*Aspland, 1997*).

Dyes are classified by their chemical composition, the types of fibers to which they can be applied, by hue, or by the method of application. Dye molecules may attach to the surface of the fiber, be absorbed by the fiber, or interact with the fiber's molecules. Each fiber reacts differently to dyes. Fiber modifications will also react differently to the same dye. Within a dye classification, different hues will have different colorfastness.

The Society of Dyers and Colourists (SDC) and the American Association of Textile Chemists and Colorists (AATCC) classify dyes by their chemical composition. In the publication, *The Colour Index International*, dyes are listed by their generic name, which indicates the application class, and by a Colour Index constitution number (CI number) which indicates the chemical structure (*Society of Dyers and Colourists, 1992*).

Although it is only since the middle of the last century that man-made or synthetic dye substances were discovered and perfected for use by the textile industry, they have almost entirely replaced natural dyes. Except in some of the most remote areas of the world, methods for growing, processing, and applying natural dyes, perfected over many centuries, suddenly became obsolete. The new dyes were more brilliant, more permanent, less costly, and far easier to use. By increasing or decreasing the proportion of the dye to the weight of the fabric, any hue, tint, or shade could be obtained.

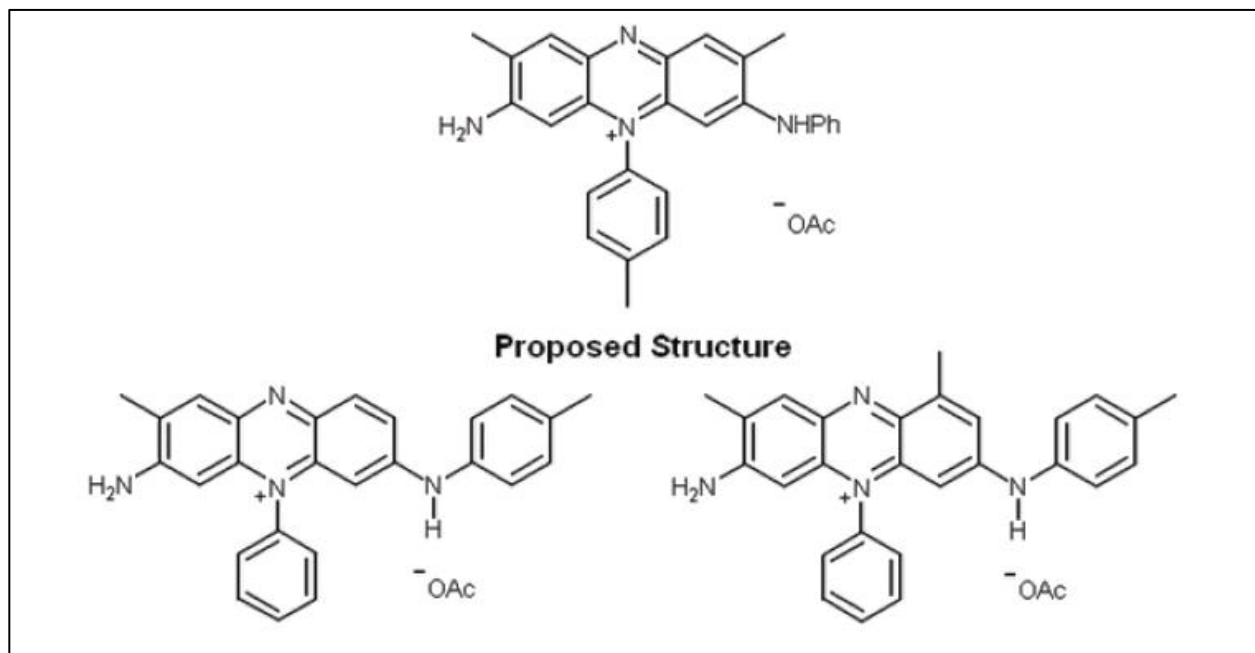


Figure 2.1: Structure of synthetic dye (*Perkin, 1896*)

As more and more synthetic dyes were formulated they became categorized into specific types or classes, based on their respective chemical components and their affinity for bonding with only certain designated fibers. There are many classes of dyes but since they are manufactured for the textile dyeing industry, not all types are available for distribution in the relatively small amounts appropriate for home or studio use. In addition, not all dye types can be used safely and effectively with the various resist-dye textile techniques presented here.

Fiber-reactive are historically the most recent of the synthetic dyes, first developed in England about thirty-five years ago for use by the textile industry, but they have proven to be the most useful and practical dyes for the textile artist. There are more than 100,000 commercially available dyes with over 7×10^5 tonnes of dyestuff produced annually. Due to their chemical structure, dyes are resistant to fading on exposure to light, water, and many chemicals. Due to their complex structure and synthetic origin, many dyes are difficult to be decolorized and decomposed biologically. There are many structure varieties such as acidic, basic, disperse, azo, diazo, anthroquinone based, and metal complex dyes.

2.3.2 Batik Industry

Homemade textile industry is very well known in Malaysia especially in the East Coast of Peninsular Malaysia and Sarawak. This industry is traditionally inherited from generation to generation. High skills coupled with the right equipment and tools are needed in order to produce batiks of high quality. Today, this industry has become very commercialized and contributed positively to the economic growth for some states such as Kelantan and Terengganu. Moreover, this industry has become the main attraction of foreign and local tourists to visit these states apart from other attractions such as an Islamic ruled government and many more. (*Kheng, 1998*) Traditionally, the process of making a batik industry is shown in Figure 2.1.

Batik or fabrics with the traditional batik patterns are found in (particularly) Indonesia, Malaysia, Japan, China, Azerbaijan, India, Sri Lanka, Egypt, Nigeria, Senegal, and Singapore. The techniques, symbolism and culture surrounding hand-dyed cotton and silk garments known as Indonesian Batik permeate the lives of Indonesians from beginning to end: infants are carried in batik slings decorated with symbols designed to bring the child luck, and the dead are shrouded in funerary batik. Clothes with everyday designs are worn regularly in business and academic settings, while special varieties are incorporated into celebrations of marriage and pregnancy and into puppet theatre and other art forms. The garments even play the central role in certain rituals, such as the ceremonial casting of royal batik into a volcano. Batik is dyed by proud craftspeople who draw designs on fabric using dots and lines of hot wax, which resists vegetable and other dyes and therefore allows the artisan to color selectively by soaking the cloth in one color, removing the wax with boiling water and repeating if multiple colors are desired. The wide diversity of patterns reflects a variety of influences, ranging from Arabic calligraphy, European bouquets and Chinese phoenixes to Japanese cherry blossoms and Indian or Persian peacocks. Often handed down within families for generations, the craft of batik is intertwined with the cultural identity of the Indonesian people and, through the symbolic meanings of its colors and designs, expresses their creativity and spirituality. (*Representative List of the Intangible Cultural Heritage of Humanity, 2009*).

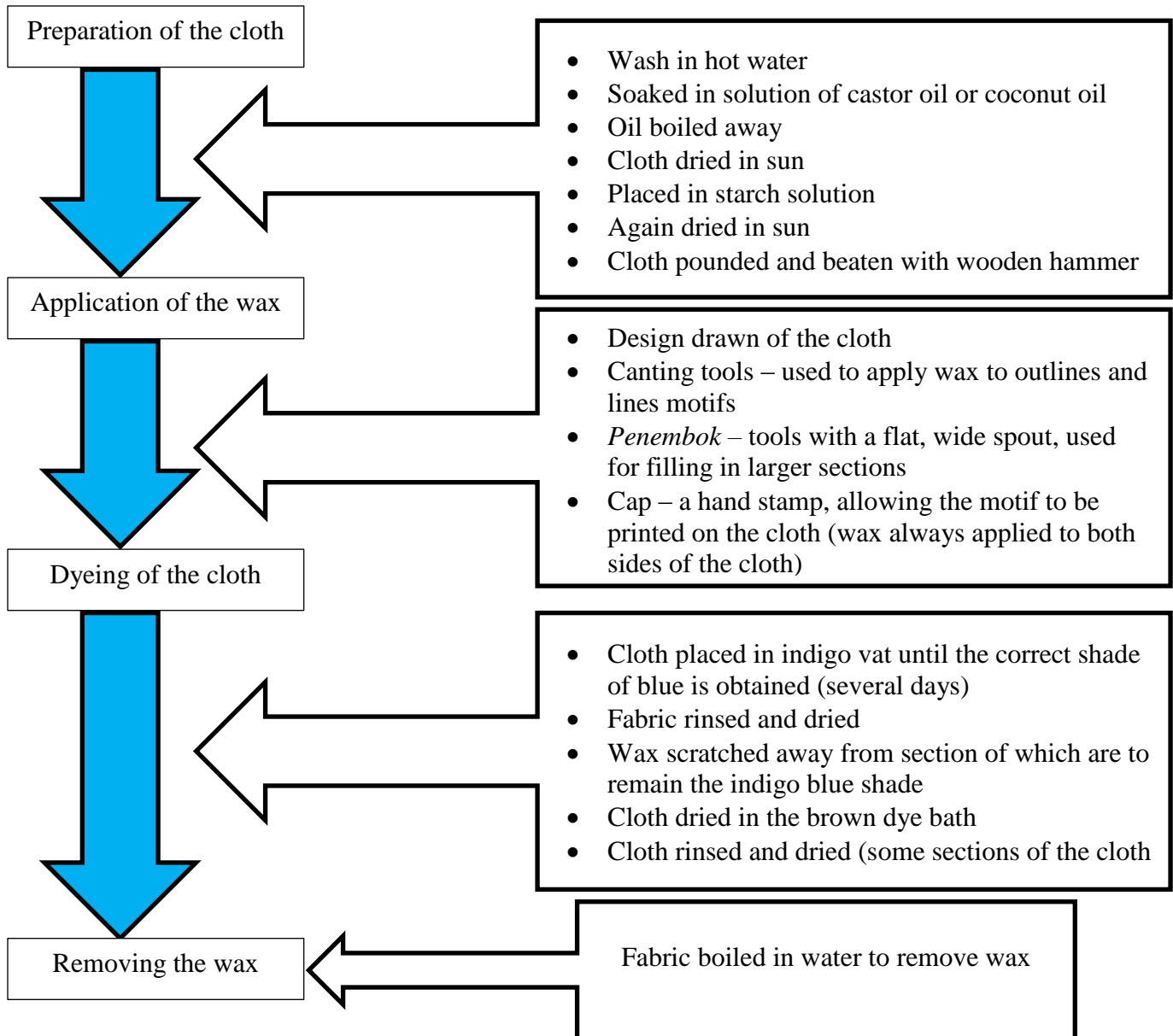


Figure 2.2: Schematic diagram of a batik process

It is started from the preparation of cloth and raw materials and the use of suitable equipment until drying process. The process of batik painting starts with the preparation of fabrics, dyes, waxes, chemicals (ludigol, natrium silicate, sodium carbonate, sodium alginate, and potassium aluminum sulfate) and the equipment and tools which are the canting tool and stove. The painting process consumes large volume of water for washing. The process is continued with the application of wax on the fabric. Conventionally, the dye obtained from natural resources like plant and insect were used for batik textile. Nowadays, dyeing process uses both organic and inorganic synthetic dye that available in variety of colors. It is first pulled through a chemical agent that helps the dye to bond to the cloth. The dye reacts with the chemical agent to produce the final color. Chemical substance such as ludigol is added into dilute dye to get a brighter and more vibrant color. Besides, alum (potassium aluminum sulfate) is used in fabric pretreatment process before marbling, so that the design can be applied well on the fabric. Sodium alginate is used to thicken the dye for screening, printing, and painting or to control spreading (*Siti Zainon, I. 1986*).

In batik industry, nowadays, synthetic and/or chemical dyes are used widely. Most of the batik producers use reactive dye as the coloring substance and the plain fabrics are imported from China, India, and Japan. By using pencil and stencil, variety of design can be applied on the fabrics. It is normally done by the skilled designer who in charge of producing new and attractive designs from time to time. The design and color combination are very important to have a good market value. Usually the design is based on nature such as flowers, trees, and sceneries.

“*Canting*” process using simple tools; the canting and small thin wall spouted copper container that is connected to a short bamboo handle. The copper container is filled with melted wax to draw the design on clothes. Next the cloth is dyed. Again, it requires skills in order to produce a good combination of colors. Finally the decorated and painted fabrics are boiled. This process is very important to remove all the wax that protects the dye from spreading during the painting process. In this process, a lot of chemicals and suspended solids including dyes may be removed from the surface of the batik. Eventually the batik is dried under mild sun to protect the colors.

Also, removing the wax consumes large volume of water. The boiled water after the boiling process is normally discharge and can be considered as wastewater from the batik industry without further treatment. It produces a variety of waste streams that vary widely with respect to parameters such as grease surfactant contains suspended solids and colors. Thus, it contributes water pollution due to the usage of many chemical substances.

2.4 Synthetic Dye and Its Impact on the Environment

Synthetic dye is usually made from aniline or chrome. Aniline's level of toxicity is not known but it does disrupt the oxygen distribution process in the body. Chrome is easily found in the land, air and even water but is most attracted to the land. Once soil is contaminated with high levels of chrome, it stays there as chrome does not enter the water easily, therefore its molecules cannot move around once it is in the soil. It stays. Workers in industries which utilize chrome have been prone to nasal irritations and have a heightened chance to develop lung and respiratory cancer. Imagine having organic cotton dyed with synthetic dye that has endangered the health of people. That will completely ruin the concept of having an eco-friendly piece of cloth. (*Sarah Yeoh, 2012*).

During the manufacturing process of such dyes, strong acids, alkalis and solvents are used along with certain heavy metals to get a particular compound. Besides posing a health hazard, it also generates a great amount of toxic waste. Plus, they usually need high temperatures to produce a certain compound. For example, creating a dye known as Mordant Blue 23 in the Color Index, part of its production includes combining a few ingredients with sulfur to produce Sulfur Trioxide (S_2O_3) at a temperature of $1300^{\circ}C$. It strains the environment because industries still depend on un-renewable energy sources such as petroleum and coal, especially since most dye mills are located in poorer regions of the world such as India and China. With a greater demand for synthetic dye, a greater carbon footprint will be left on our environment due to the depletion of coal and petroleum as well as the fumes emitted.

Even after the dye has been completed, there is wastage that gives a negative impact on the environment. For example, the grinding of materials or the dumping of dry, powdered materials; these actions can pollute the air, making it toxic to anyone who inhales it. Between each batch of materials processed, the equipment needs to be cleaned as well for quality control purposes and this uses a great deal of water. The water that is flushed from this process is not safe as it contains lots of chemicals such as the dye itself, remnants of dye intermediates (ingredients that kick start certain chemical processes) and minute solids. These are all highly toxic and till this day, not many companies treat this wastewater properly before disposing of it.

Due to the unavailability of an efficient waste disposal system, cases such as the Fuan textile factory in Guangdong which deals primarily with export textiles of brands such as Gap, Target and Walmart; dumped its dye waste into the Maozhou River, contaminating not just a direct water source but the underground water source as well. Underground water contamination lasts for many years which endanger the health of the current and future generation. This is just one of the many illegal cases of dye waste dumping in China; though it may be hard to fault the dye mills alone for doing so. American apparel companies which forms the largest customer base for China's dye mills demand lower and lower prices and to keep up with demand, the waste water is not treated to cut costs.

Synthetic dye and various clothing finishes have also been known to be a trigger for clothing dermatitis, which is a skin condition resulting in an allergic reaction from synthetic dye or any additives in clothing. This is commonly present in heavy-set and or individuals who sweat easily and is normally concentrated in the armpits, groin and the waistband area. Natural fibres such as angora, cotton and linen have never caused this condition though treated or starched cotton can be a trigger. (*Sarah Yeoh, 2012*).

2.5 Adsorbent

2.5.1 Introduction of Activated Carbon

Adsorbents are used usually in the form of spherical pellets, rods, moldings, or monoliths with hydrodynamic diameters between 0.5 and 10 mm. The adsorbent must have high abrasion resistance, high thermal stability and small pore diameters, which results in higher exposed surface area and hence high surface capacity for adsorption. The adsorbents must also have a distinct pore structure which enables fast transport of the gaseous vapors. Most industrial adsorbents fall into one of three classes:

- Oxygen-containing compounds – Are typically hydrophilic and polar, including materials such as silica gel and zeolites.
- Carbon-based compounds – Are typically hydrophobic and non-polar, including materials such as activated carbon and graphite.
- Polymer-based compounds - Are polar or non-polar functional groups in a porous polymer matrix.

Activated carbon can be considered as a material of phenomenal surface area made up of millions of pores - rather like a “molecular sponge”. It consists of highest volume of adsorbing porosity of any material known. This is why it is applicable for adsorption process. Granular activated carbons are versatile adsorbents with wide range of applications. They are most effective adsorbents in treating drinking water and industrial wastewater.

Activated carbon is one of the commercial adsorbents that has been processed to make it extremely porous and thus to have a very large surface area available for adsorption or chemical reactions. This is a microcrystalline material made by thermal decomposition of wood, vegetable shells, coal and so on, and has surface areas of 300 to 1200 m²/g with average pore diameters of 10 to 60 Å. Organics are generally adsorbed by activated carbon. Activated carbons are well known for their exceptional ability to adsorb some acidic gases. (*Geankoplis, 2003*).

Normally in the adsorption process, the common activated carbons used are powdered activated carbon (PAC) and granular activated carbon (GAC). Both types have their own advantages and disadvantages. PAC has smaller size compared to GAC. Normally the PAC size is less than 1.0 mm in size with an average diameter between 0.15 and 0.25 mm. Besides they can present a large surface to volume ratio with a small diffusion distance. The disadvantage of PAC is easy to occur the high head loss. PAC is generally added directly to other process units, such as raw water intakes, rapid mix basins, clarifiers, and gravity filters.

On the other hand, GAC has larger size that leads to small surface to volume ratio and also smaller external surfaces. Normally, the GAC size is divided into two parts according to different phase applications; liquid phase and vapor phase applications. For liquid applications, the sizes are 8×20, 20×40, or 8×30 while vapor phase applications are 4×6, 4×8 or 4×10.

2.5.2 Chemical Properties and Physical Characteristics of Activated Carbon

Activated carbon products can be characterized by physical properties and activity properties. (*TIGG Corporation, 2012.*) Activated carbon is a carbonaceous adsorbent with a highly developed porous structure. Its effectiveness in removing pollutants has been found superior to many other techniques because of the high quality of the effluent achieved, simplicity of design, ease of operation and insensitivity to toxic substances. Nevertheless, its high cost precludes its application in most cases. Recognizing this economic drawback, many investigators have made efforts in low-cost alternatives to activated carbon from a range of carbonaceous and mineral precursors, such as fruit stones, nut shells, fly ash, minerals and scrap tires. The efforts made in this direction have been extensively reviewed by *Pollard et al. (1992) and Heschel and Klose (1995).*

Important physical properties are surface area, product density; mesh size, abrasion resistance and ash content. In water treatment applications, carbon density is expressed as back washed and drained (BWD) or bulk density. This establishes the number of pounds of carbon required to fill a back washable filter, and is expressed in terms of pounds per cubic foot.

Mesh size (8x30, 12x40, 20x50, etc.) establishes the range of particle sizes and thus, the effective particle size that will be used in a filter. Particle size is an important parameter in specifying carbons for specific applications, affecting such operating conditions as pressure drop, filtration capabilities, backwash rate requirements and the rate of adsorption of contaminants. While a smaller particle size affects more pressure drop across a carbon bed, the rate of diffusion of an organic into the pore and its subsequent adsorption is significantly increased.

Another important characteristic that distinguishes different types of liquid phase carbons is abrasion resistance. Abrasion resistance refers to a carbon's ability to withstand degradation during handling and is expressed in terms of abrasion number. The higher the abrasion number, the more resistant the carbon is to abrasion.

The final important physical property of activated carbon is ash level, which reflects the purity of the carbon. It is the inorganic residue left after heating of the raw material. Common ash constituents of coal-based carbons are silica, alumina, iron, calcium and magnesium.

Activity characterizations are key indicators of a carbon's potential performance for removing contaminants from water. An important characterization tool used in determining the ability of a carbon to adsorb a particular adsorbent is the pore size distribution, which is usually depicted in the form of a curve (Figure 1). The pore size distribution is produced through adsorption of gases and liquids under pressure. It defines the available pore volume of a carbon over three pore size regions: The micropore region (less than 100 Angstroms. in size), mesopore region (between 100 and 1,000 Angstroms), and macropore region (greater than 1,000 Angstroms).

The molecules encountered in gas phase are generally smaller and more mobile than those in liquid phase applications. Therefore, a gas phase carbon has the majority of its pores concentrated in the micropore region. There is a wider range of molecular sizes in liquid phase work (taste and odor, color bodies, organics, pesticides), and adsorbates are less mobile in water. This means a broader range of pore sizes must be available, both for ease of movement of adsorbates through the carbon pores and for adsorption of particular molecular sizes. Inexpensive tests have traditionally been used to approximate the distribution of pores available for a carbon as just described. These tests include the adsorption of a single standard reference adsorbate, and give the ability to distinguish activity characteristics of different carbons.

Iodine is the most common standard adsorbate and is often used as a general measurement of carbon capacity. However, because of its small molecular size, Iodine more accurately defines the small pore or micropore volume of a carbon and thus reflects its ability to adsorb low molecular weight, small substances. Iodine number is defined as the milligrams of Iodine adsorbed by one gram of carbon, and it approximates the internal surface area (square meters per gram).

Molasses number is a measure of the degree of de-colorization of a standard molasses solution. Because color pigments are large and cannot penetrate into small pores, the molasses number defines the large pore or macropore volume of a carbon. It is used as a relative guideline for measuring the capacity of a carbon for larger adsorbate molecules.

CHAPTER THREE

MATERIALS AND METHODS

3.1 Introduction

In order to study the kinetics and equilibrium studies on the adsorption of organic compound or synthetic dye batik by using granular activated carbon, there are several methods that must be done. There are two parameters that must be considered before conduct the experiment which are first; batch adsorption study which includes effect of adsorbent dosage (w), effect of contact time, and effect of initial acetic acid concentration. Second parameter is adsorption equilibrium study. All the first three parameters will produce graph of the concentration which is then will be interpret by isotherm graph Langmuir, Freundlich or BET isotherm graph.

3.2 Materials and Methods

3.2.1 Experimental Apparatus

Experimental apparatus that has been used during the research are; pH meter to measure the pH of synthetic dye batik solution before next procedure of the experiment is being proceeded. From the testing, the pH of synthetic dye batik solution is getting to be 3.13. Then by using the turbidity meter to get the value of turbidity contained in the synthetic dye batik solution. The turbidity value is then determined 1000 NTU. Next apparatus used is stackable incubator shaker. This was used to make sure that the mix solution of synthetic dye batik solution and granular activated carbon (GAC) is well-mixed and become homogenous solution. Once done with the shaker, UV-Visible Spectrophotometer is being used to analyze the adsorbance value of the mix solution from previous experiments.



Figure 3.1: pH Meter



Figure 3.2: UV-Visible Spectrophotometer



Figure 3.3: Turbidity Meter



Figure 3.4: Stackable Incubator Shaker

3.2.2 Experimental Materials

There are two main materials used in the research; synthetic dye batik and granular activated carbon. The GAC is provided by Dr Anwaruddin Hisyam while the synthetic batik dye is bought from Natural Batik Village Sdn Bhd located at Balok where the price is RM20 for 1.5 litres. For the granular activated carbon, Dr Anwaruddin give me approximately 1 kilogram (kg) of granular activated carbon (GAC) while for the synthetic dye batik, Dr Anwaruddin gave me the dye in powdered form where the dilution must be made in order to get the solution of synthetic dye batik. The color is yellowish when diluted with deionized water. The concentration of the dye is approximately 4.0483 g/L.



Figure 3.5: Synthetic Batik Dye in powdered form



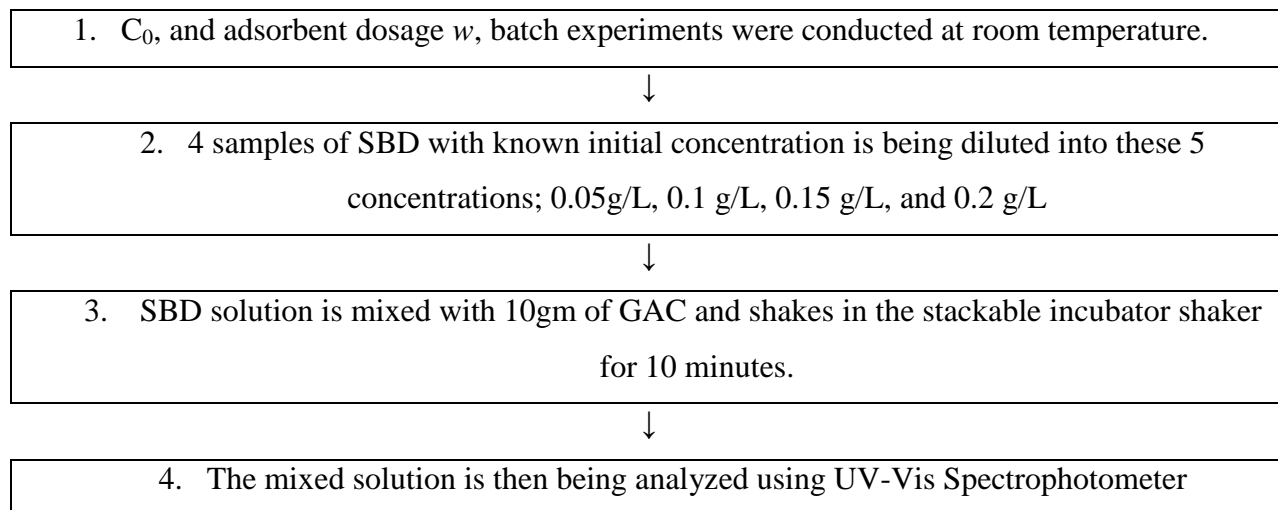
Figure 3.6: Granular Activated Carbon



Figure 3.7: Synthetic Batik Dye solution after dilution

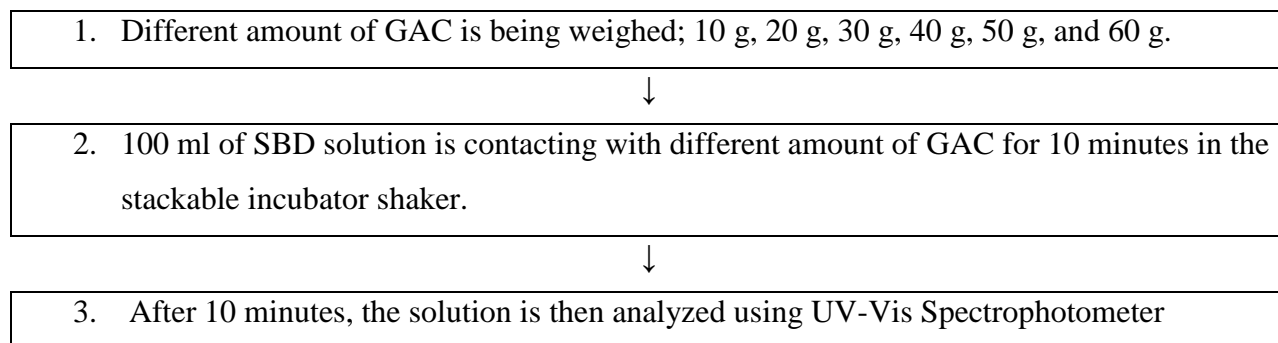
3.2.3 General Flow of Methodology

3.2.3.1 Effect of Initial Synthetic Batik Dye (SBD) Concentration (C_0 vs C)



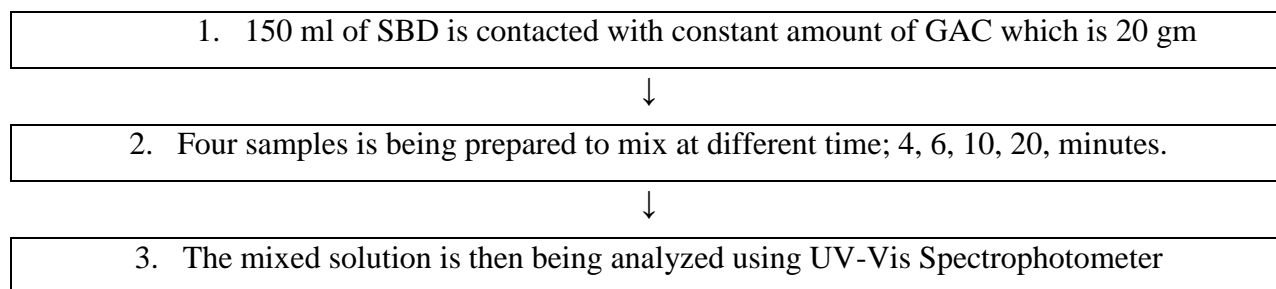
Scheme 3.1: Step by step Effect of SBD Initial Concentration

3.2.3.2 Effect of Adsorbent Dosage (w) (w vs C)



Scheme 3.2: Step by step Effect of Adsorbent Dosage (w)

3.2.3.3 Effect of Contact Time (time vs C)



Scheme 3.3: Step by step Effect of Contact Time

3.3 Overview of Parameters

3.3.1 Effect of Initial Synthetic Batik Dye Concentration (C_0 vs C)

Since the concentration of synthetic batik dye is known, this is 4.0483 g/L, the solution is then being diluted to five different concentrations; 0.05g/L, 0.1 g/L, 0.15 g/L, 0.2 g/L and 0.25 g/L. All five samples which volume is 100 ml is then mixed with 10 gram of GAC and being shake in the stackable incubator shaker for about 10 minutes at 150 rpm. Then about 5 ml solutions needed from each sample to analyze using UV-Visible Spectrophotometer. The graph is then plotted.

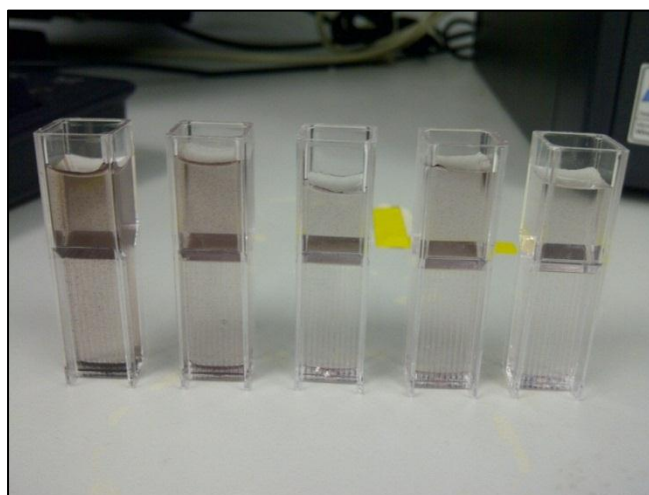


Figure 3.8: Mixed solution in the cuvette for UV-Vis analysis

3.3.2 Effect of Adsorbent Dosage, w (w vs C)

Six different amount of GAC; 10 g, 20 g, 30 g, 40 g, 50 g, and 60 g is mixed with 100 ml SBD in a conical flask with constant concentration and shake in the stackable incubator shaker at constant time; 10 minutes. The shaker must be set at 150 rpm. After 10 minutes, about 5 ml of the mixed solution is taken for analysis using UV-Visible Spectrophotometer. The graph is then plotted.



Figure 3.9: The solution in the stackable incubator shaker

3.3.3 Effect of Contact Time (time vs C)

100 ml of SBD solution is mixed with 20 gram of GAC but at different times; 4, 6, 10, 20, 30 minutes. These means five samples must be prepared. The mixed solution is then shaking at the magnetic stirrer at 150 rpm. 5 ml of the samples is taken for analysis using UV-Visible Spectrophotometer. The graph is then plotted.

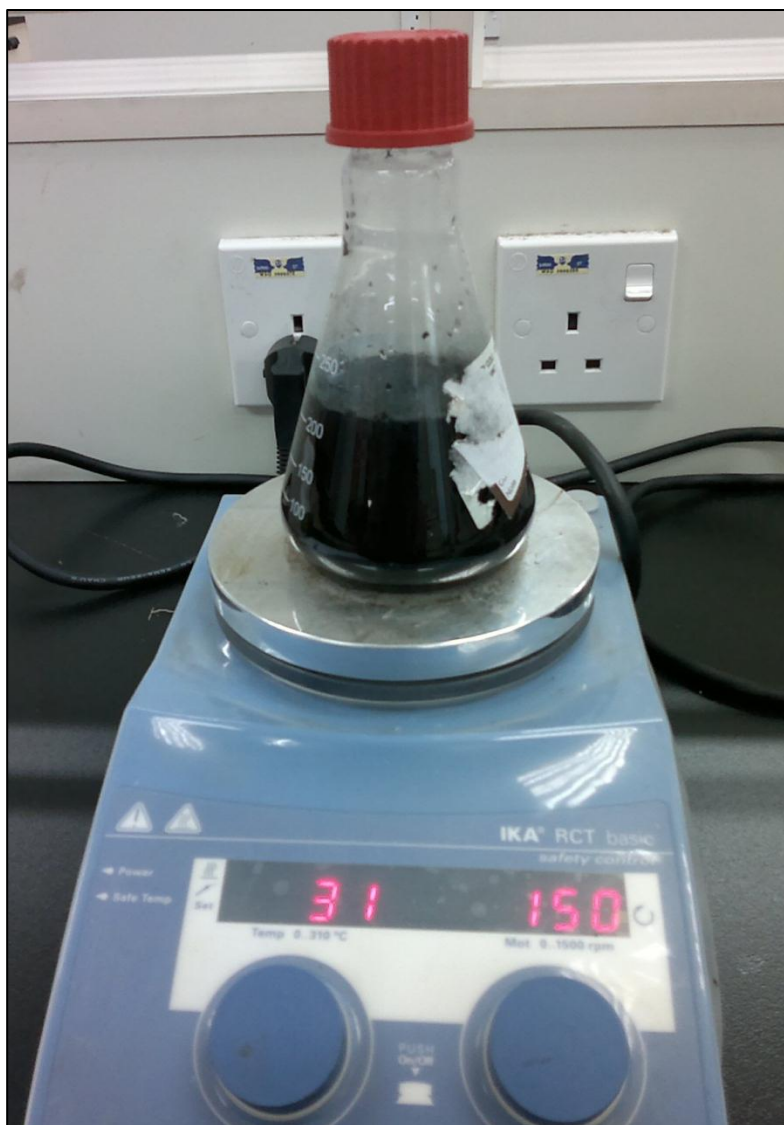


Figure 3.10: The solution is stirred on the magnetic stirrer

CHAPTER FOUR

RESULT AND DISCUSSIONS

4.1 Result and Discussion

After done with the experiments and analysis, the absorbance values getting from the analysis of UV-Visible Spectrophotometer is being plotted into the graph. The difference of the concentration was evaluated by its concentration difference given by the following equation:

$$\text{Concentration Difference \%} = \frac{C_0 - C}{C_0} \times 100\% \text{ ----- } \rightarrow (\text{Equation 4.1})$$

Where C_0 is the initial concentration while C is the final concentration.

The concentration difference getting from the analysis shows that by using granular activated carbon (GAC), the percentage of concentration difference calculated range from 50 % - 97 % at difference contact time, adsorbent dosage, and SBD initial concentration.

4.2 Standard Curve Analysis

Before analysis towards all samples has been done, the standard curve of the solution must be made. This will be a guidance to get the unknown concentrations of the solution. Since the concentration of SBD is 4.0483 g/L, the concentration taken to make the standard curve are 1 g/L, 2 g/L, and 3 g/L. This graph will be the references for the three parameters to get the final concentration, C after the experiments. The standard curve will be used as references if the concentration of the solution is not known. The solution is analyzed first using UV-Vis, and get the value of Absorbance, A . From the standard curve graph, the Absorbance getting from the analysis is then moved to the right until reach straight line, and moved downward and get the concentration. Below are the results and the graph plotted of the standard curve:

Concentration (g/L)	Absorbance (A)
Blank sample	0
1	0.074
2	0.148
3	0.205

Table 4.1: Standard Curve Absorbance Table

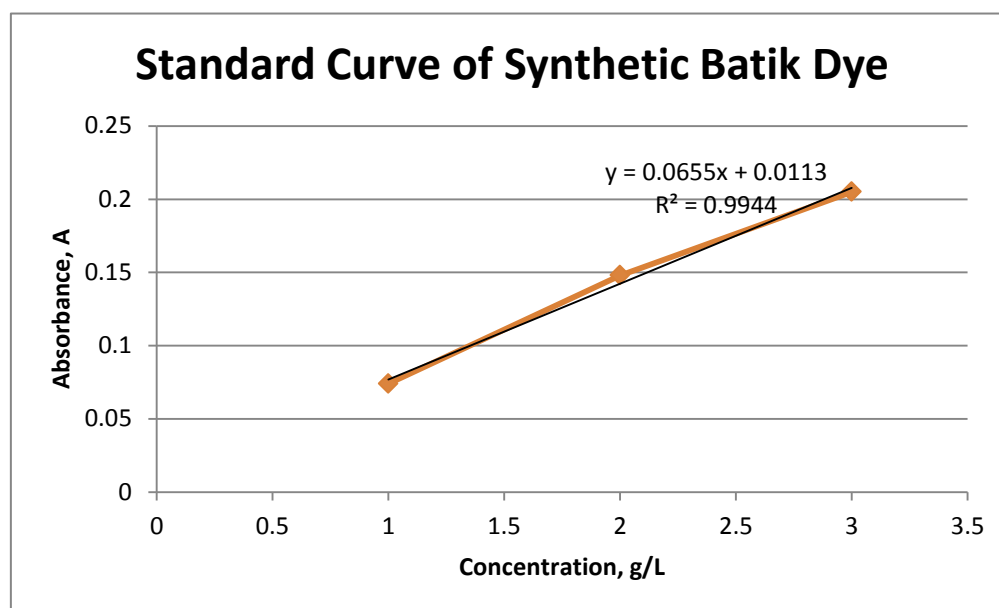


Figure 4.1: Graph Standard Curve-Absorbance vs Concentration

The graph above shows the standard curve of the solution. From the graph above, it shows that the absorbance value is increasing when the concentration increased. The concentration becomes manipulated variable while the volume of the each concentration used is constant. The standard curve of the solution can be used to get the concentration of the solution. The solution with unknown concentration is analyzed first using UV-Vis, then the value of absorbance get from the analysis is then used to get the final concentration.



Figure 4.2: The solution with different volume and concentration for standard curve analysis (10 ml, 25ml, and 100 ml)

4.3 Study the Effect of Synthetic Batik Dye Initial Concentration (C_0)

Experiment has been done to find the relationship between initial concentration, C_0 and final concentration, C . In this experiment, the initial concentration of synthetic batik dye is being manipulated while the mass of GAC is held constant. The time also constant to 10 minutes. Four solutions with different concentration are being prepared. Below are the results and the graph plotted of the study the effect of initial synthetic batik dye concentration (C_0):

Initial concentration, C_0 (g/L)	Absorbance, A	Final Concentration, C (g/L)	Concentration Difference, %
0.05	0.173	0.0244	51.2
0.1	0.274	0.045	55
0.15	0.387	0.058	61.33
0.2	0.404	0.0605	69.75

Table 4.2: Absorbance vs Initial Concentration Table

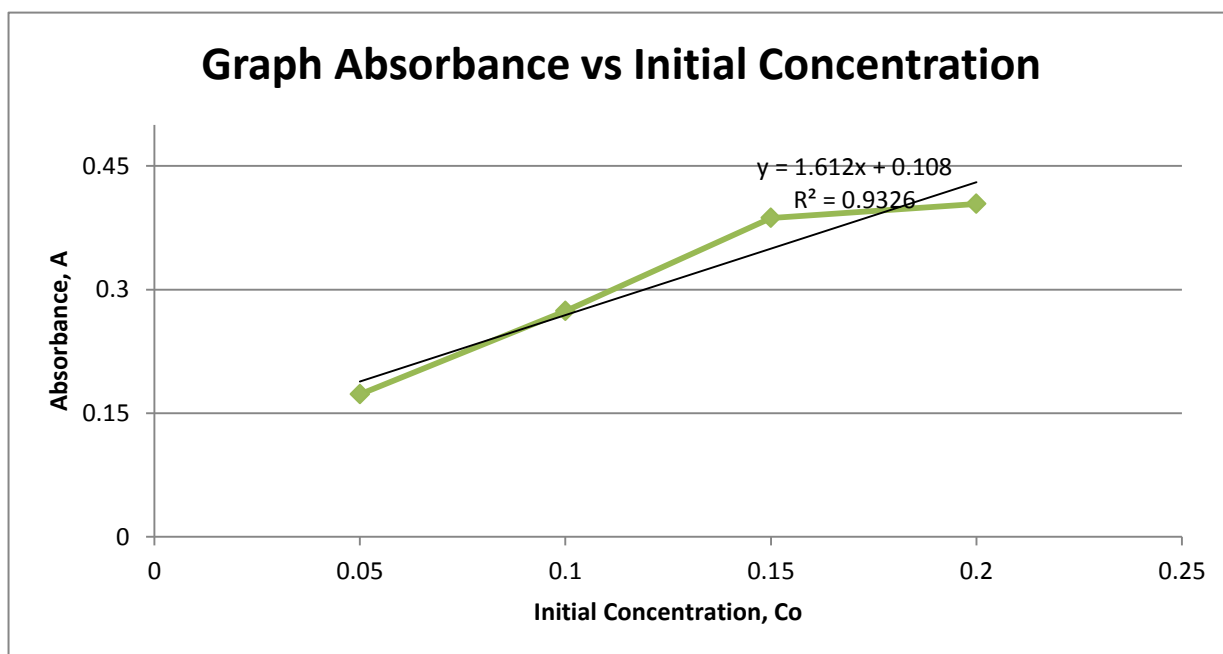


Figure 4.3: Graph Absorbance vs SBD Initial Concentration, C_0

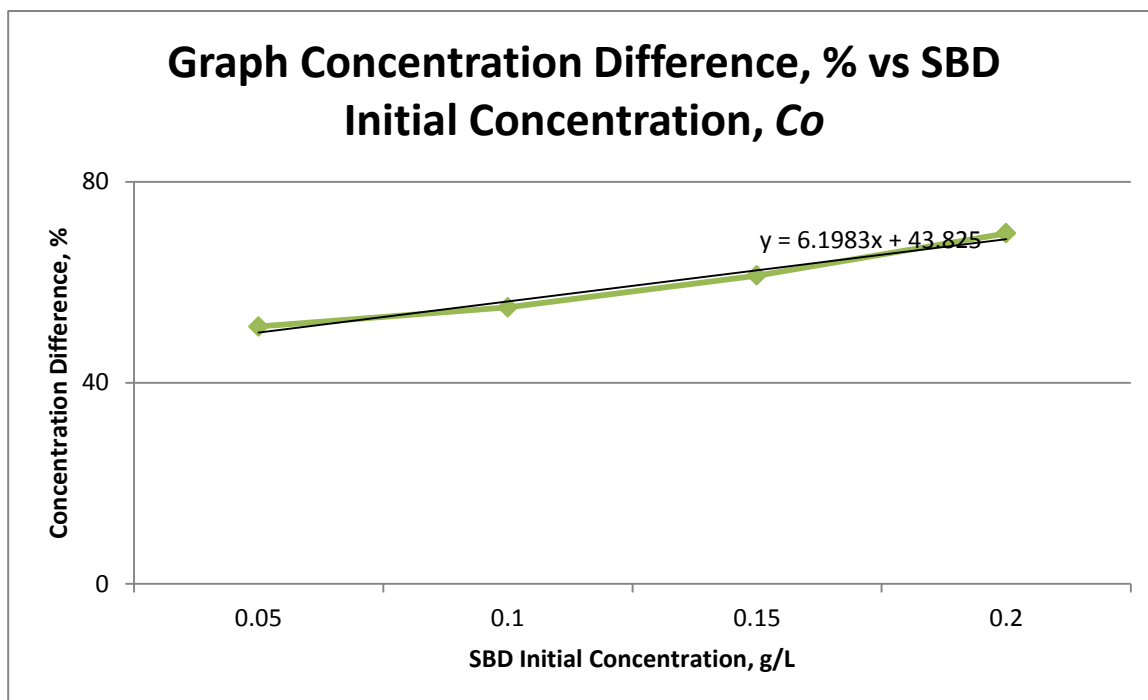


Figure 4.4: Graph Concentration Difference, % vs SBD Initial Concentration, C_o

For figure 4.3, the straight line was created and this shows that when the SBD initial concentration, C_o increased, the absorbance, A will also be increased. The highest absorbance value is at 0.2 g/L and the absorbance value is 0.404 A. And the lowest absorbance value is at 0.05 g/L while the absorbance value is 0.173 A. For figure 4.4, the graph also shows the straight line where the lowest initial concentration; 0.05 g/L gives 51.2 % of concentration difference. While the highest initial concentration; 0.2 g/L gives 69.75 % of concentration difference. The hypothesis can be made; the higher the initial concentration, the higher the absorbance and concentration difference percentage will be.

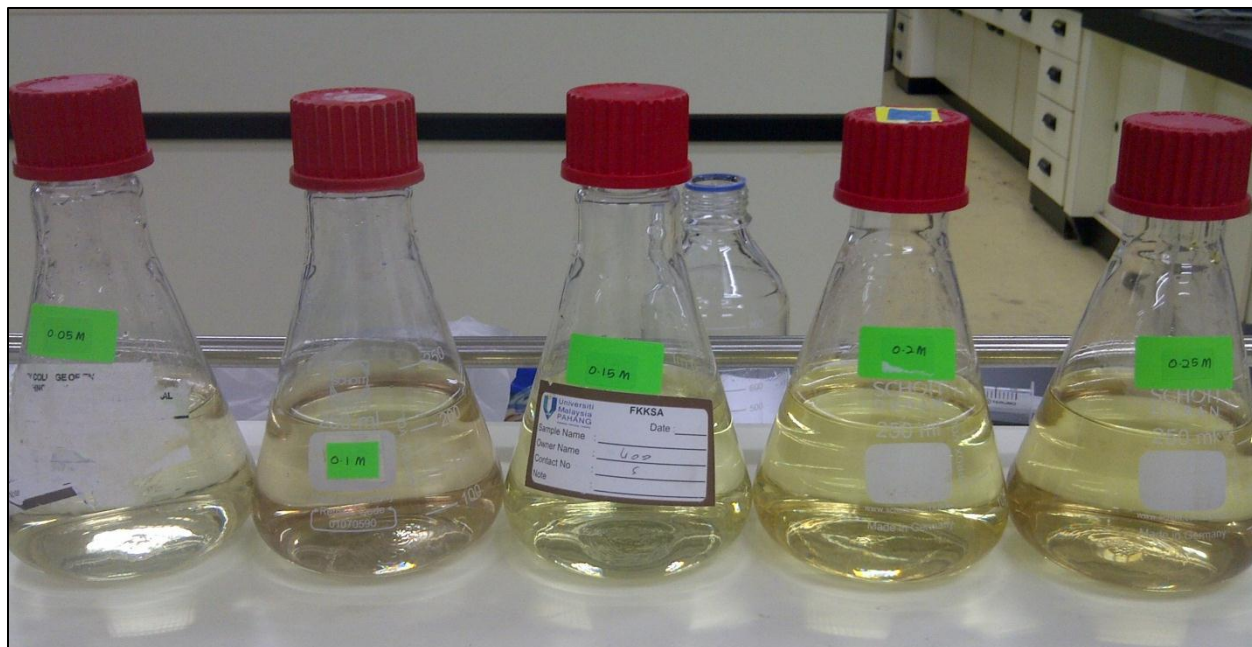


Figure 4.5: The SBD solution with different concentration before mixing

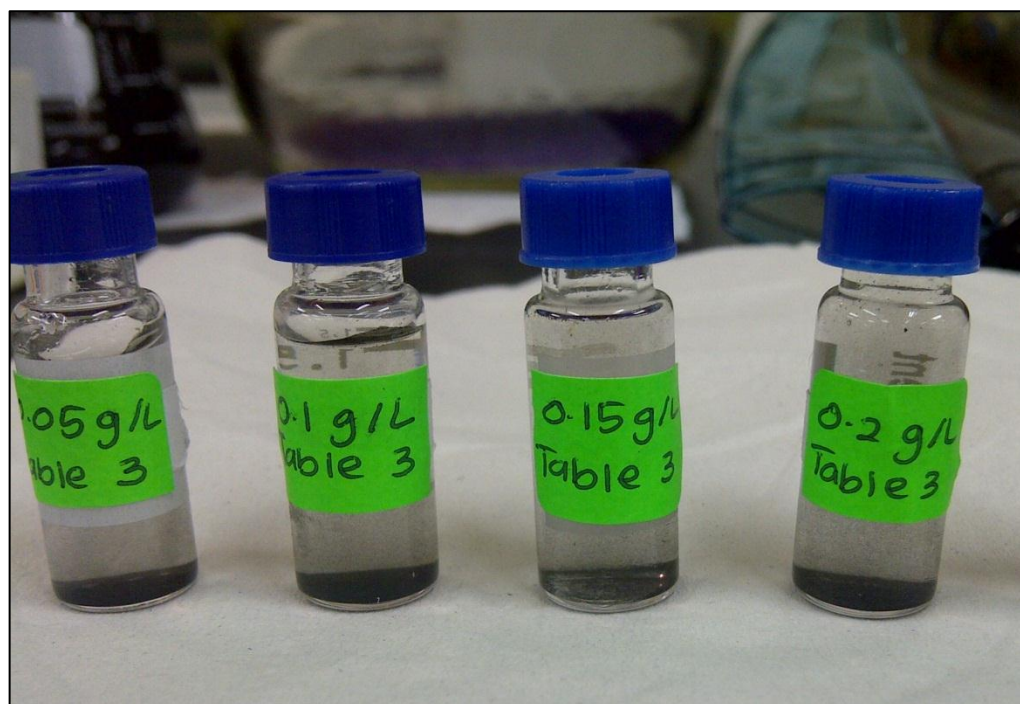


Figure 4.6: The SBD solution after mixing with GAC

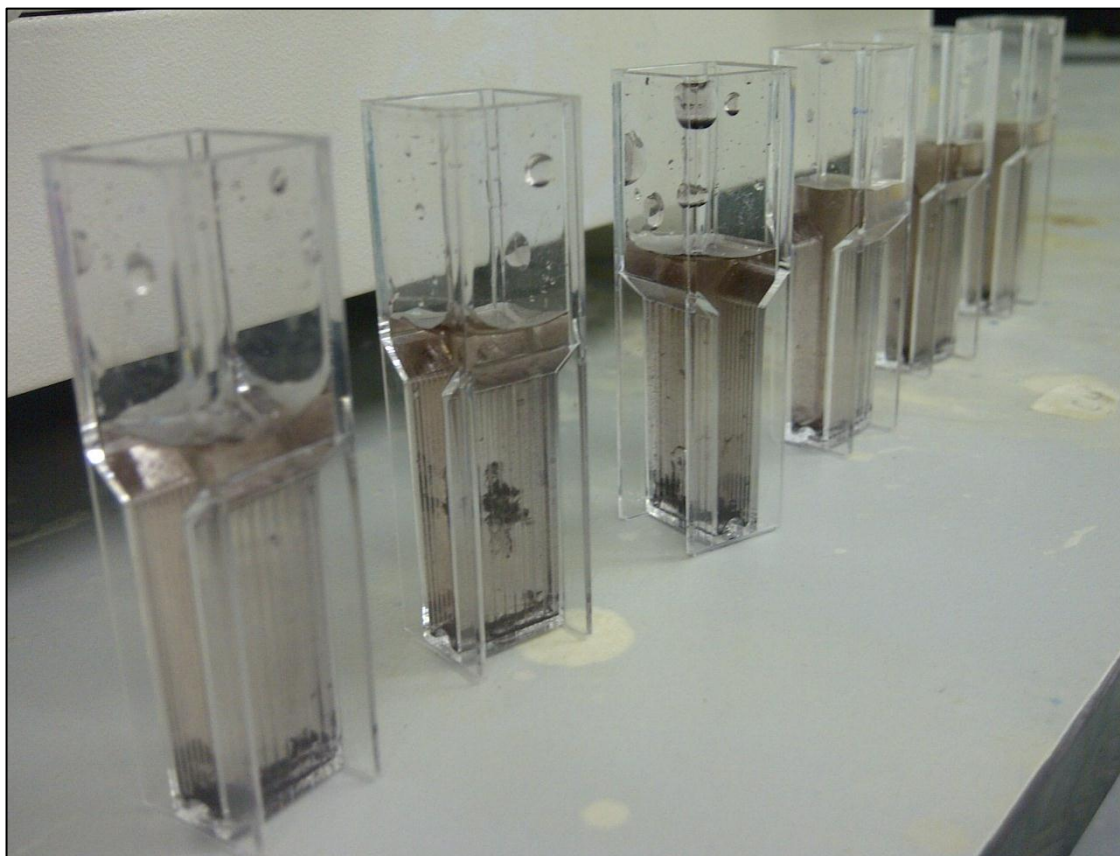


Figure 4.7: The mixing solution in the cuvette going through settling process

4.4 Study the Effect of Adsorbent Dosage (w)

In this experiment, the time for contacting mixed solution and the volume of synthetic batik dye is held constant. While the mass of GAC is being manipulated at six different mass; 10 g, 20 g, 30 g, 40 g, 50 g, and 60 g. Below are the results and the graph plotted of the study the effect of adsorbent dosage (w):

Adsorbent dosage, w (g)	Absorbance, A	Initial Concentration, C_0 (g/L)	Final Concentration, C (g/L)	Concentration Difference, %
10	0.355	133.33	18.20	86.35
20	0.456	133.33	17.60	86.80
30	0.746	133.33	8.75	93.44
40	0.849	133.33	8.28	93.79
50	2.921	133.33	6.97	94.77
60	2.921	133.33	6.97	94.77

Table 4.3: Absorbance vs Adsorbent Dosage (w) Table

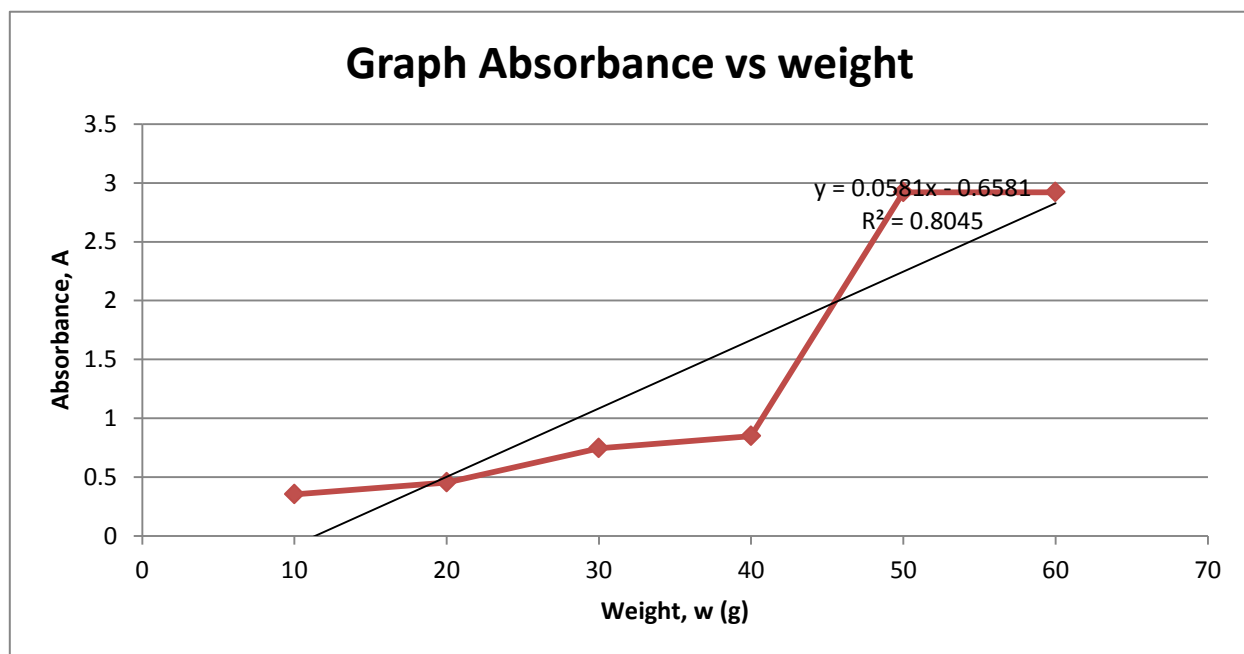


Figure 4.8: Graph Absorbance vs Adsorbent Dosage (w)

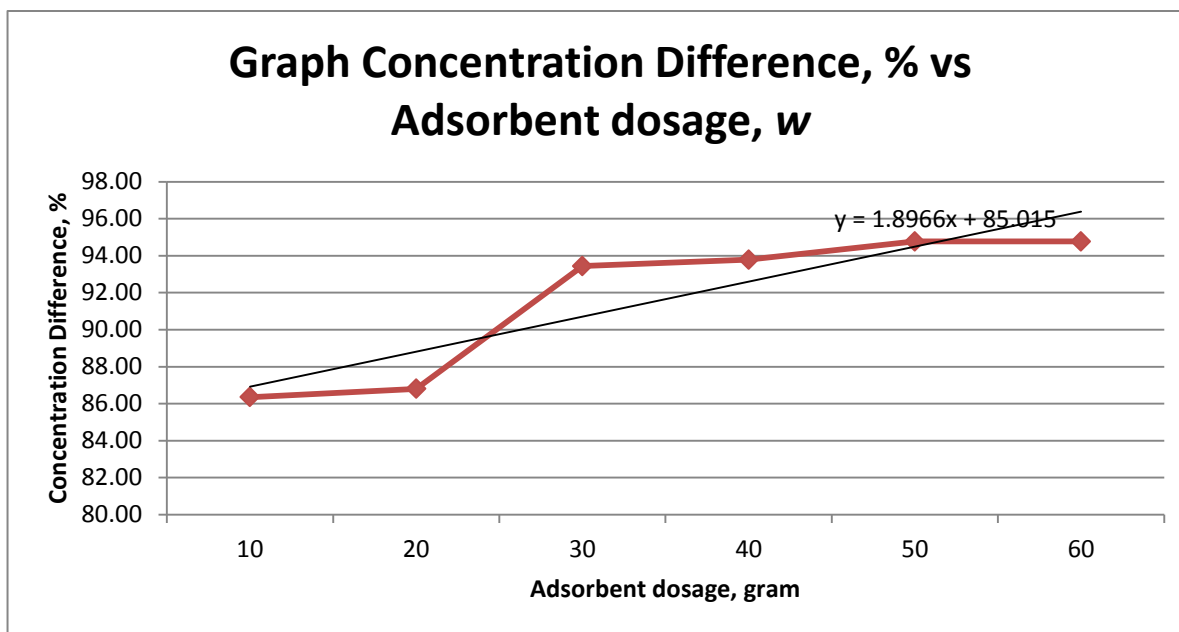


Figure 4.9: Graph Concentration Difference, % vs Adsorbent Dosage, w

Based on Figure 4.5, the straight line was created and this shows that when the adsorbent dosage, w increased, the absorbance, A will also be increased. The lowest Absorbance, A which is 0.355, occurred at weight 10 g while the highest absorbance which is 2.921 occurred at highest adsorbent dosage, w at 60 g. For graph 4.6, the highest value of concentration difference is calculated getting 96.03 % is occurred at highest amount of adsorbent dosage which is 60 gram. While the lowest concentration difference percentage calculated and get 86.35 % is at 10 gram. The hypothesis can be made here, as the adsorbent dosage increased, the absorbance value will also increase and this will cause the highest percentage of concentration difference.

4.5 Study the Effect of Contact Time

For the last experiment is to study the time versus concentration. The volume of synthetic batik dye is maintained at 100 ml and the mass of GAC is held constant at 20 gram. Only time will change in this experiment. There are about four samples need to be prepared and the time are; 4, 6, 10, and 20 minutes. Below are the results and the graph plotted of the study the effect of contact time:

Contact time, t (min)	Absorbance, A	Initial Concentration, Co (g/L)	Final Concentration, C (g/L)	Concentration Difference, %
4	0.503	133.33	7.88	94.09
6	0.504	133.33	7.85	94.11
10	0.507	133.33	7.83	94.13
20	0.508	133.33	6.58	95.06

Table 4.4: Adsorbance vs Contact Time Table

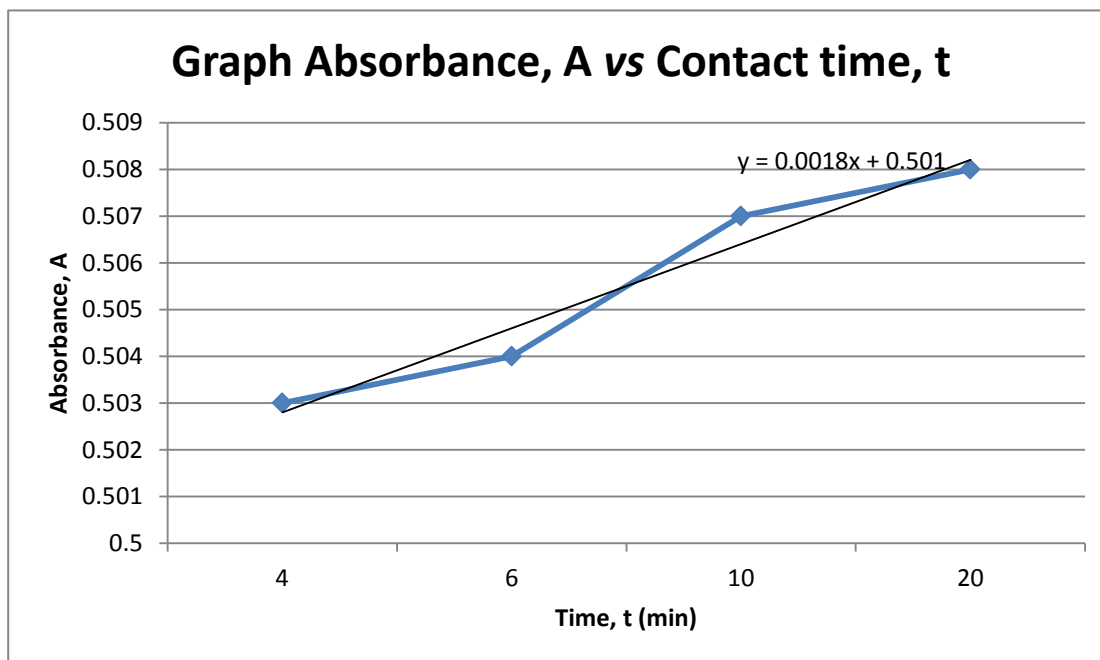


Figure 4.10: Graph Absorbance, A vs Contact time, t

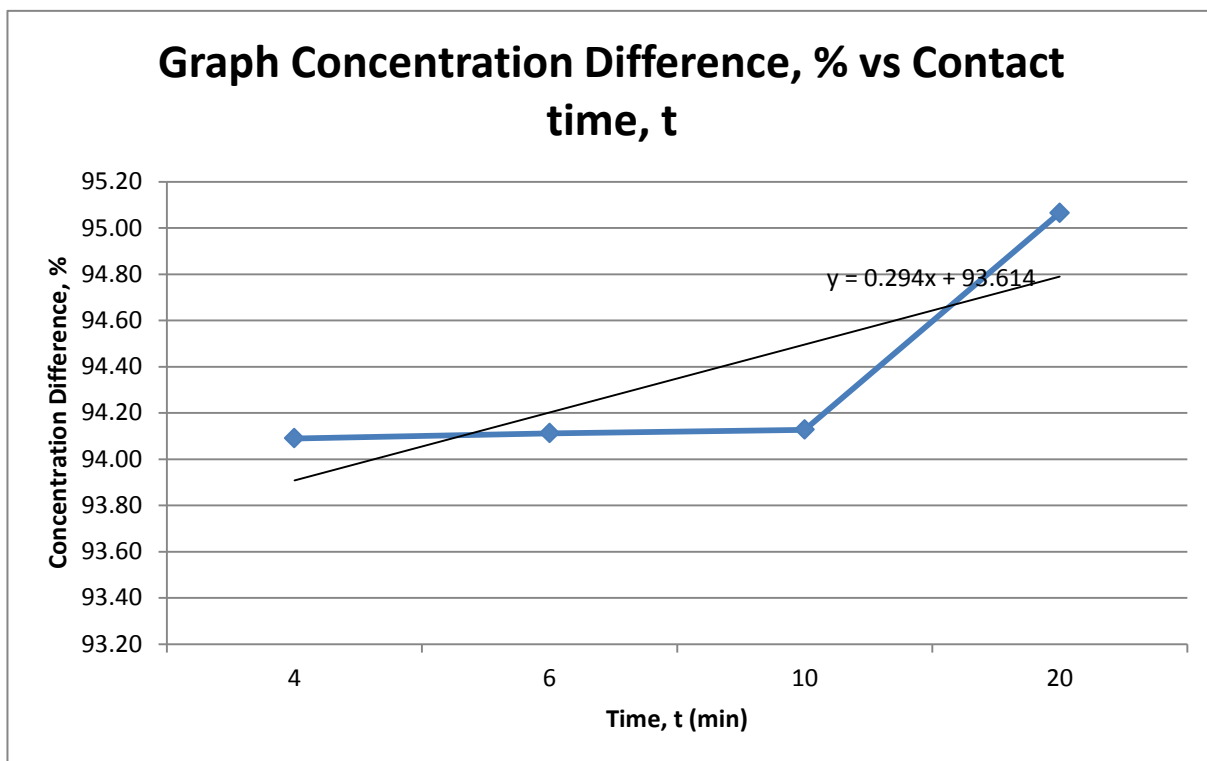


Figure 4.11: Graph Concentration Difference, % vs Contact time, t

From figure 4.7, it shows a slightly increasing straight line where the absorbance value is increased a bit when the contact time more longer. The lowest absorbance was recorded at contact time is 4 minutes where the absorbance value is 0.503. While the highest absorbance that can get is 0.508 at time 20 minutes. For figure 4.8, at time 4 minutes, the concentration difference is the lowest where the percentage is 94.09 %. And the highest percentage is at 20 minutes and the percentage is 95.06 %. This means the longer the contact time between synthetic batik dye (SBD) and granular activated carbon (GAC) to adsorb, the higher the value of absorbance will be.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

As a conclusion from the research, the findings and experiment that have been done and the result obtain will help researcher to improve kinetics and equilibrium studies on the adsorption of synthetic batik dye using granular activated carbon (GAC). The results obtained from these experiments really help the researcher to find the concentration or any other kinetics studies. The batch process of this study is a lab scale where this research can be continued by using continuous process. Lastly, these experiments are considered successful. The synthetic batik dye (SBD) can be treated by using granular activated carbon.

5.2 Recommendations

Some recommendations have been made to improve the result for future references:

1. The color must also be tested using turbidity meter and compare it with the discharge permits which were outlined by the Department of Environment Malaysia (DOE).
2. The turbidity testing must be done before and after the mixing process of SBD and GAC in order to measure the color appears.
3. The color changes must be mentioned clearly with the picture proof.
4. The UV-Visible Spectrophotometer must always be calibrated so that accurate result can be obtained.
5. Make the same research but change the adsorbent with powdered activated carbon (PAC)
6. Change the adsorbent with other agricultural waste adsorbent.

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APPENDIX A



Figure A.1: Synthetic Batik Dye in powdered form before dilution



Figure A.2: Synthetic Batik Dye solution after dilution

APPENDIX B

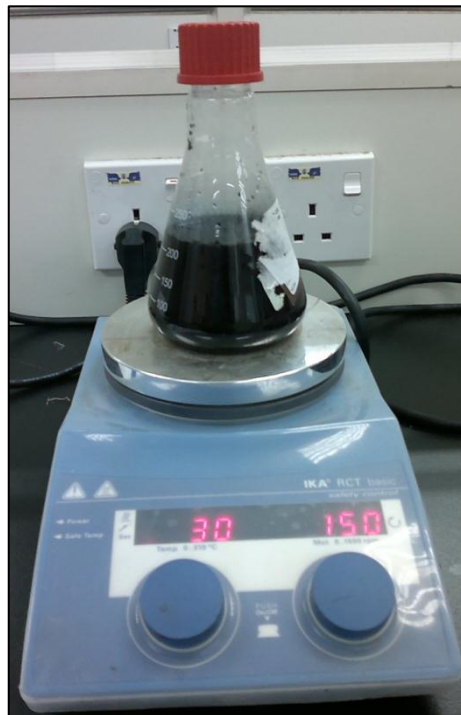


Figure B.1: Mixed solution is stirred on the magnetic stirrer



Figure B.2: Mixed solution with different amount of GAC is in the stackable incubator shaker

APPENDIX C

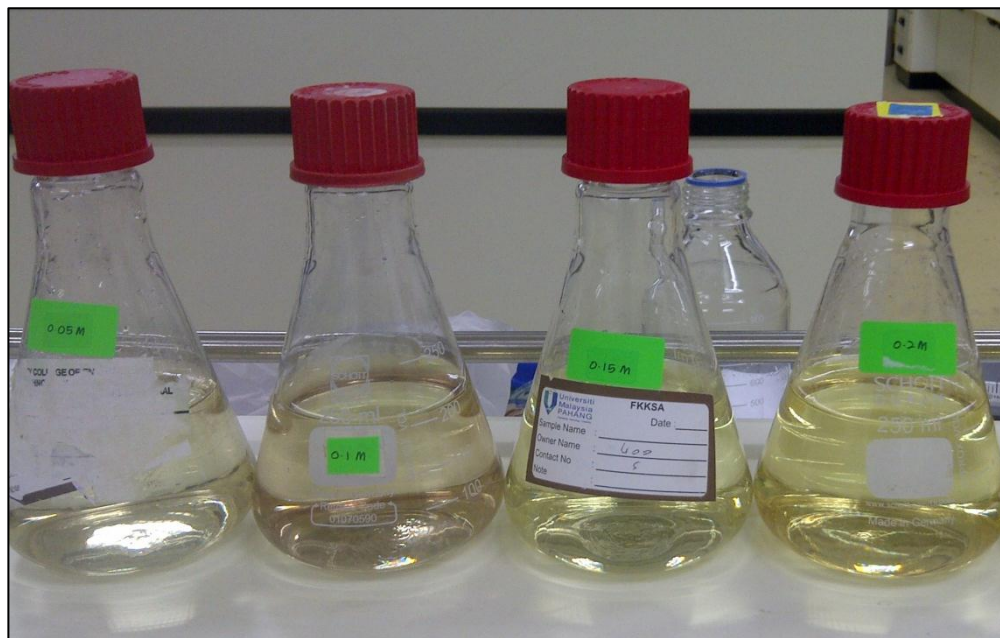


Figure C.1: SBD solution after dilution with different initial concentration

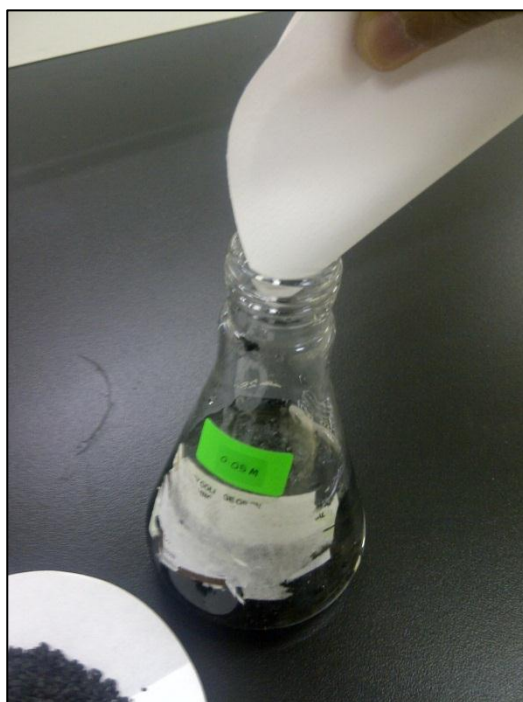


Figure C.2: GAC is inserted in the flask to contact with different initial concentration



Figure C.3: After mixing the GAC and SBD, the solution is put into stackable incubator shaker

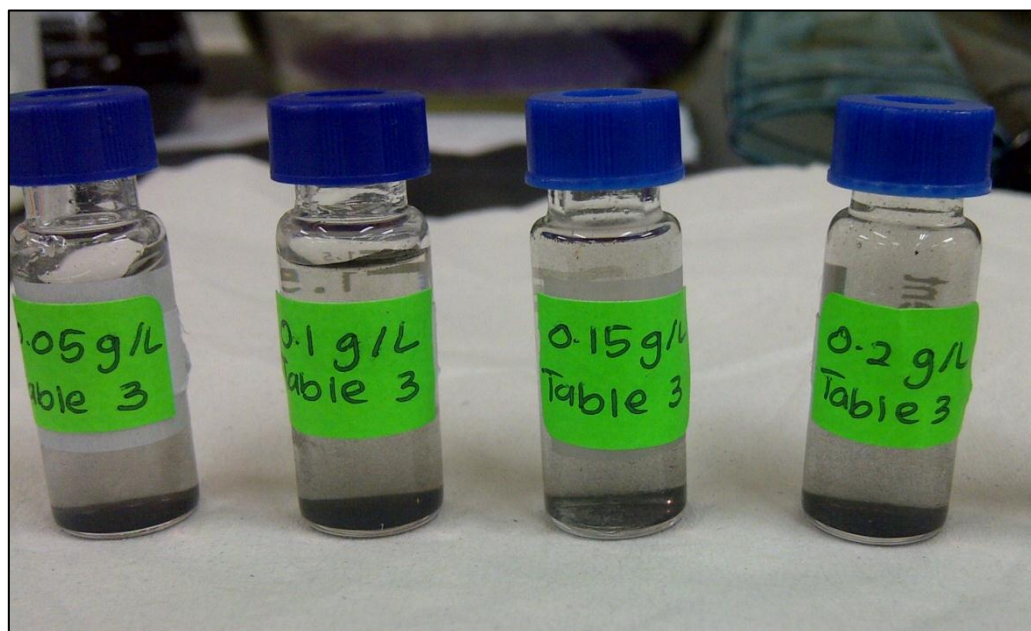


Figure C.4: The sample is taken and put it in the vial

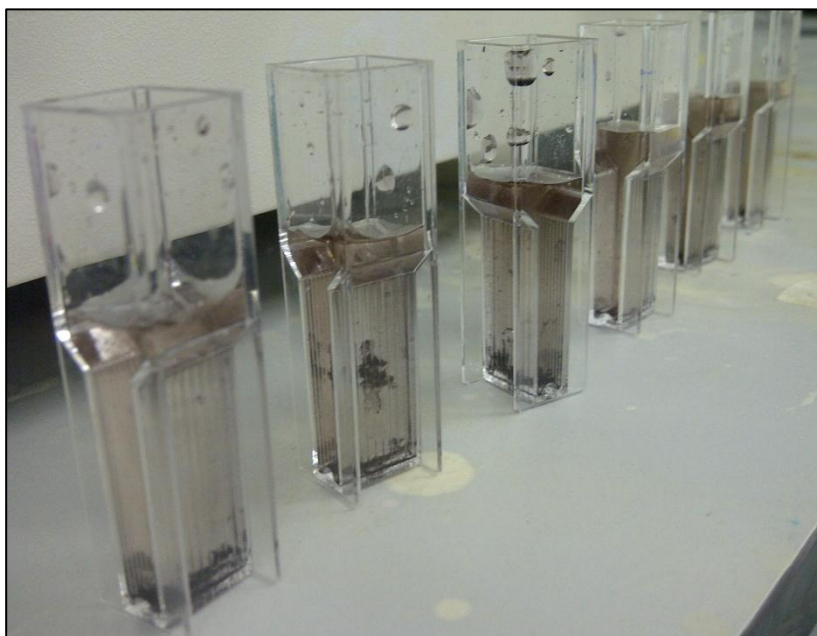
APPENDIX D

Figure D.1: The sample collected from the experiment is put in the cuvette for analysis

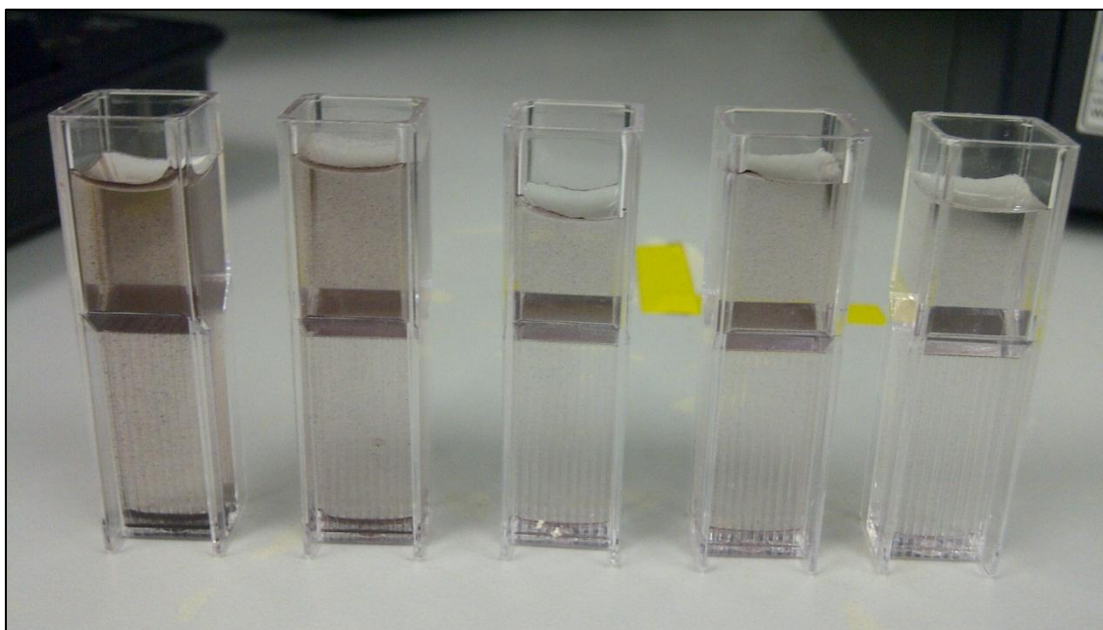


Figure D.2: Different color at different SBD initial concentration

APPENDIX E**Equation E.1: Equation and Calculation for the Experiment**

$$M_1 V_1 = M_2 V_2$$

$$V_1 = \frac{M_2 V_2}{M_1}$$

Where:

M_1 = Concentration of SBD solution (g/L)

M_2 = Concentration of dilute solution needed (g/L)

V_1 = Volume of SBD needed to be diluted (mL)

V_2 = Volume of Water to dilute the SBD solution (mL)

For standard curve, the concentration need to be diluted with 1, 2, and 3 g/L since the concentration of SBD solution is 4.0483 g/L.

For 1 g/L,

$$M_1 V_1 = M_2 V_2$$

$$4.0483 (V_1) = 1 (10)$$

$$V_1 = \frac{10}{4.0483} = 2.47 \text{ mL}$$

For 2 g/L,

$$M_1 V_1 = M_2 V_2$$

$$4.0483 (V_1) = 2 (25)$$

$$V_1 = \frac{50}{4.0483} = 12.35 \text{ mL}$$

For 3 g/L,

$$M_1 V_1 = M_2 V_2$$

$$4.0483 (V_1) = 3 (100)$$

$$V_1 = \frac{300}{4.0483} = 74.105 \text{ mL}$$

Equation E.2: Calculation for Percentage of Concentration Difference, %

$$\text{Concentration Difference \%} = \frac{C_0 - C}{C_0} \times 100\%$$

The calculation is used to get the concentration difference between final concentration and initial concentration of the sample. All three parameters used this equation to get the percentage.

time, t (min)	Absorbance, A	Initial Concentration, C ₀ (g/L)	Final Concentration, C (g/L)	Concentration Difference, %
4	0.503	133.33	7.88	94.09
6	0.504	133.33	7.85	94.11
10	0.507	133.33	7.83	94.13
20	0.508	133.33	6.58	95.06

$$\text{Concentration Difference, \% at time, } t = 4 \text{ minutes} = \frac{133.33 - 7.88}{133.33} \times 100 \% = 94.09 \%$$

$$\text{Concentration Difference, \% at time, } t = 6 \text{ minutes} = \frac{133.33 - 7.85}{133.33} \times 100 \% = 94.11 \%$$

$$\text{Concentration Difference, \% at time, } t = 10 \text{ minutes} = \frac{133.33 - 7.83}{133.33} \times 100 \% = 94.13 \%$$

$$\text{Concentration Difference, \% at time, } t = 20 \text{ minutes} = \frac{133.33 - 6.58}{133.33} \times 100 \% = 95.06 \%$$