Adsorptive Removal of Reactive Yellow 15 from Aqueous Solution by Banana Peel

by

Muhammad Amin Bin Azhari

Dissertation submitted in partial fulfilment of
the requirements for the
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CERTIFICATION OF APPROVAL

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Civil Engineering Programme
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Approved by,

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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

MUHAMMAD AMIN BIN AZHARI

ABSTRACT

The adsorbent was prepared from banana peel and adsorption of Reactive Yellow 15 (RY15) dye by the banana peel adsorbent was examined. Batch adsorption tests showed that extent of dye adsorption was dependent on pH, dye concentration, contact time and adsorbent dose. Equilibrium adsorption was attained in 3 h, with maximum adsorption occurring at a pH value 2 and adsorbent dose of 6 g/L. Equilibrium adsorption data were well described by Langmuir and Freundlich adsorption isotherm models. The adsorption of Reactive Yellow 15 followed pseudo-first-order kinetics. The results indicate that adsorbent prepared from banana peel is effective in adsorptive removal of Reactive Yellow 15. Hence, banana peel would be suitable low cost substitute for commercial activated carbon in the removal of reactive dyes from aqueous solution.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Dyes are chemicals which on binding with a material to produce colour to the material, such as fabric. Dyes that form a covalent bond with the substrate during the dyeing process are known as reactive dyes. There is a rapid growth rate in the used of reactive dyes due to the increasing use of cellulosic fibres and the technical and economic limitations of other dyes used for these fibres (Allen & Koumanova, 2005). Coloured effluent arises as a direct result of the dye production and it is estimated that 10-15% of the dye is lost in the effluent during the dyeing process (Garg et al., 2003; Young & Yu, 1997).

Adsorption techniques for wastewater treatment have become more popular in recent years owing to their efficiency in the removal of pollutants too stable for biological methods, hence producing high quality water (Allen & Koumanova, 2005). Adsorption has been found to be superior to other techniques for wastewater treatment in terms of initial cost, simplicity of design, ease of operation and insensitivity to toxic substances. McKay (1982) specifies that main adsorbent that has been widely used in industry as activated carbon which comes in the form of granular (GAC) or powder (PAC).

1.2 Problem Statement

1.2.1 Problem Identification

Carr (1995) indicates 40% of the colour is discharged in the effluent from reactive dyeing operation resulting in a highly coloured effluent. An additional problem is the reactive dyes in both ordinary and hydrolysed forms are not easily biodegradable, hence even after extensive treatment; colour may still remain in the effluent. The conventional processes such as coagulation, flocculation and biological methods adopted for decolourising effluent containing reactive dyes are no longer effective to achieve an adequate colour removal.

Strickland & Perkins (1995) testified the greatest environmental concern with dyes are their absorption and reflection of sunlight entering the water which interferes with the growth of bacteria to levels insufficient to biologically degrade impurities the water. Colour in effluents can cause problems in several ways. Dyes can have acute and chronic effects on exposed organisms depending on the exposure time and dye concentration. Dyes are inherently highly visible, meaning that the concentrations as low as 0.005 ppm capture the attention of both public and the authorities (Pierce, 1994). Dyes also absorb and reflect sunlight entering water and so can interfere with the growth of bacteria and hinder photosynthesis in aquatic plants (Slokar & Marechal, 1998).

Kharaisheh et al. (2002) reported that reactive dye is known to have low absorbability on a wide range of adsorbents. Reactive dye wastewater has limited biodegradability in an aerobic environment. Many reactive azo dyes, constituting the largest dye groups decompose to potential carcinogenic aromatic amines under anaerobic conditions (Lazearidis et al., 2003; Netpradit et al., 2003). Adsorption has proved to be more versatile and efficient compared to conventional physic-chemical methods of dye removal (Senthilkumar et al., 2006). Garg et al. (2004) agree adsorption methods using activated carbon have been invariably successful to decolourise dye effluents due to its large surface area, micro porous structure and high adsorption capacity. However, this application is limited by the high cost of adsorbents.

1.2.2 Significance of the Project

Many industries especially textile industry often use dyes and pigments to colour their products. Most dyes are inert and non-toxic at the concentration discharged into the receiving water, however, they impart colour undesirable to the water user. Colour removal from textile effluents is a major environmental problem because of the difficulty to treating such streams by conventional physicochemical and biological treatment methods (McKay, 1982). Activated carbon is the most widely used adsorbent for this purpose because it has a high capacity for adsorption of organic matter, but its use is limited because of its high cost (Battacharya & Venkobachar, 1984; Singh & Rawat, 1994). This has led to search for cheaper substitutes. Coal, fly ash, wood, silica gel, clay materials and agricultural wastes have been tried with varying success for colour removal. Low-cost and effective technologies are needed to solve the problem. From the criteria mentioned above, banana peel appears to be a suitable potential adsorbent due to the fact that it is a food waste and has been reported to adsorb heavy metals (Annadurai et al., 2003; Okro & Ejike, 2007; Anwar et al., 2010; Castro et al., 2011). Moreover, banana peel is easily available and cheap in nature.

1.3 Objectives of the Study

- To prepare and characterize the banana peel adsorbent.
- To assess the potential of banana peel in adsorptive removal of C.I Reactive Yellow 15.
- To compare the reactive dye adsorption capacity onto banana peel adsorbent with that onto other plant-based adsorbents.

1.4 Scope of the Study

- Preparations of banana peel adsorbent.
- Characterizations of banana peel adsorbent by scanning electron microscope, infra-red spectra, surface area and pore size analysis.
- Feasibility of using banana peel as a low-cost adsorbent for the removal of reactive dye (C.I Reactive Yellow 15).
- Parameters that affect the adsorption process of the reactive dye.
- Comparison between reactive dye adsorption capacities onto banana peel against other plant-based adsorbents.

1.5 Relevancy of the Project

This project is relevant to human health, safety and environment. The world-wide high level of production and use of dyes generates colored wastewaters, which give cause of environmental concern. Textile companies, dye manufacturing industries, paper and pulp mills, tanneries, electroplating factories, distilleries, food companies and a host of other industries discharge colored wastewater. As a matter of fact, the discharge of such effluents in the environment is worrying for both toxicological and esthetical reasons. In most countries including Malaysia, banana peel is one of the organic materials produced from agricultural industries. Banana is one of the largest consumed fruits in the world and it's useless peels, therefore, creates one of the major agro-waste problems. Study has showed that that several tons of banana peels are produced daily in market places and household garbage that create environmental nuisance (Anwar et al., 2010). Therefore, this study will be able to contribute to the betterment of global populace as it will produce a removal technique that is effective yet economical.

1.6 Feasibility of the Project within the Scope and Time Frame

This project is an extended study over two semesters for fulfilment of FYP I and FYP II courses. The followings are the aim of the project for the first four months (FYP I):

- Literature review on the topic.
- Preparation of the banana peels adsorbent

After the completion of FYP I, adsorption experiments and data analysis was conducted within the remaining four months (FYP II). Among the experiments that were conducted for FYP II are:

- Optimum pH test
- Optimum adsorbent concentration
- Optimum contact time
- Adsorption isotherm experiment

The estimated project cost is within the budget allowed by the university. Besides that, all the materials and equipment for this project are available at the Environmental Engineering Laboratory.

CHAPTER 2

LITERATURE REVIEW

2.1 Reactive Dye

Reactive dye is a class of highly coloured organic substances, which attach to the substrates by chemical reaction and forms a covalent bond between the molecule of dye and the fibre. The dyestuff therefore becomes part of the fibre and much less likely to be removed by washing.

The reactive dyes are originally designed for cellulose fibres but also can be used for polyamide, cotton, linen, wool and nylon. In theory, when the dyes are applied to a fibre in a weakly alkaline dyebath, the reactive group will form a chemical bond with the fibre. Thus, the most important characteristics of reactive dyes are the formation of covalent bond with the substrate to be coloured.

Today, reactive dyeing in textile industries is currently the most widespread process in the world. Reactive dyes are superior compared to all other dyes because it binds chemically to the textile fibre, hence significantly improving the product's colour stability and wash ability.

2.2 Environmental Problems Associated With Dyes

Dyeing and finishing operations produce large quantities of wastewater that contains organic dyestuff, surfactants, chelating agents, and other contaminants that can be characterized as high level of total organic content and color (Guendy, 2010). Synthetic dyes are used extensively by several industries, mainly in textile dyeing process. It is estimated that up to 15% of the dye is lost during this operation and disposed out in the textile effluents with a remarkable spent of water (Flox et al., 2006; Robinson, 2001). Thus, the treatment of the effluent containing these compounds is important for the protection of natural waters. The impact and toxicity of these pollutants in the environment have been extensively studied and have shown that dyes have caused negative impact to the environment (Mala & Babu, 2005; Sharma et al., 2007).

2.3 Removal of Dyes from Wastewater

There are more than 100,000 commercially available dye exist and more than 7x105 tonnes per year are produced annually (Pearce et al., 2003, McMullan et al., 2001). Wastewater containing dyes is very difficult to treat, since the dyes are recalcitrant organic molecules, resistant to aerobic digestion, and are stable to light. A synthetic dye in wastewater cannot be efficiently decolorized by traditional methods. This is because of the high cost and disposal problems for treating dye wastewater at large scale in the textile and paper industries (Ghoreishi & Haghighi, 2003). The technologies for colour removal can be divided into three categories: biological, chemical and physical (Robinson et al., 2001). All of them have advantages and drawbacks.

2.3.1 Biological Methods

Biological treatment is the often the most economical alternatives when compared with other physical and chemical processes. Biodegradation methods such as fungal decolourization, microbial degradation, adsorption by (living or dead) microbial biomass and bioremediation systems are commonly applied to the treatment of industrial effluents because many microorganisms such as bacteria, yeasts, algaes and fungi are able to accumulate and degrade different pollutants (McMullan et al., 2001; Fu & Viraraghavan, 2001). However, their application is often restricted because of technical constraint. According to Bhattacharyya and Sharma, (2003), biological treatment requires a large land area and is constrained by sensitivity toward diurnal variation as well as toxicity of some chemicals, and less flexibility in design and operation. Further, biological treatment is incapable of obtaining satisfactory colour elimination with current conventional biodegradation processes (Robinson et al., 2001). Moreover, although many organic molecules are degraded, many others are recalcitrant due to their complex chemical structure and synthetic organic origin (Kumar et al., 1998). In particular, due to their xenobiotic nature, azo dyes are not totally degraded

2.3.2 Chemical Methods

Chemical methods include coagulation or flocculation combined with flotation and filtration, precipitation-flocculation with Fe(II)/Ca(OH)2, electroflotation, electrokinetic coagulation, conventional oxidation methods by oxidizing agents (ozone), irradiation or electrochemical processes. These chemical techniques are often expensive, and although the dyes are removed, accumulation of concentrated sludge creates a disposal problem. There is also the possibility that a secondary pollution problem will arise because of excessive chemical use. Recently, other emerging techniques, known as advanced oxidation processes, which are based on the generation of very powerful oxidizing agents such as hydroxyl radicals, have been applied with success for the pollutant degradation. Although these methods are efficient for the treatment of waters contaminated with pollutants, they are very costly and commercially unattractive. The high electrical energy demand and the consumption of chemical reagents are common problems.

2.3.3 Physical Methods

Different physical methods are also widely used, such as membrane – filtration processes (nanofiltration, reverse osmosis, electrodialysis) and adsorption techniques. In accordance with the very abundant literature data, liquid-phase adsorption is one of the most popular methods for the removal of pollutants from wastewater since proper design of the adsorption process will produce a high-quality treated effluent. This process provides an attractive alternative for the treatment of contaminated waters, especially if the sorbent is inexpensive and does not require an additional pre-treatment step before its application. Adsorption is a well-known equilibrium separation process and an effective method for water decontamination applications (Dabrowski, 2001). Adsorption has been found to be superior to other techniques for water re-use in terms of initial cost, flexibility and simplicity of design, ease of operation and insensitivity to toxic pollutants. Decolourisation is a result of two mechanisms: adsorption and ion exchange (Slokar & Le Marechal, 1998), and is influenced by many physio-chemical factors, such as, dye/sorbent interaction, sorbent surface area, particle size, temperature, pH, and contact time (Kumar et al., 1998). Adsorption also does not result in the formation of harmful substance.

2.4 Adsorptive Processes

Adsorption is the process of transferring material from a fluid phase to a solid phase. Based on Mohan & Pittman (2007), adsorbents are broadly divided into two classes which are commercial and synthetic activated carbons and also low-cost adsorbent.

2.4.1 Commercial and Synthetic Activated Carbon

Activated carbon is perhaps the most widely used adsorbent for the removal of many organic contaminants which are biologically resistant, but activated carbon is prohibitively expensive. The technology to manufacture activated carbon of good quality is not fully developed in developing countries. Moreover, there are many problems connected with the regeneration used activated carbon. Consequently, the high cost of the activated carbon, coupled with the problems associated with regeneration, has necessitated the search for alternate adsorbents (Amin, 2008).

2.4.2 Low-Cost Adsorbents

As mentioned earlier, there are many methods of removing dye from wastewater. Most of these methods are expensive and also are not affordable for developing countries. This has therefore led to the use of agricultural wastes in removal of dyes. Agricultural wastes are characterized by ready availability, affordability, eco-friendliness and high uptake capacity for heavy metals due to the presence of functional groups which can bind metals to effect the removal of heavy metal (Kehinde et al., 2007). Agricultural and food wastes are high volume, low value materials that are highly prone to microbial spoilage with limits their potential. Due to these issues, these materials are currently only have been used as animal feed or disposed to packed landfills causing environmental problems. Therefore, a greater use of these wastes is needed not just for the sake of environment but also for the benefits of people especially in rural areas. Thus, using these wastes as low-cost adsorbents seem to solve both of the problems stated. Moreover, the effectiveness of chemically modified or native biomass for dye removal has been evaluated and proved by various authors (McKay et al., 1998; Garg et al., 2003).

2.5 Adsorption

Adsorption, first observed by C.W. Scheele in 1773 for gases and subsequently for solutions by Lowitz in 1785, now is recognized as a significant phenomenon in most natural physical, biological, and chemical processes. Sorption on solids, particularly active carbon, has become a widely used operation for purification of waters and wastewaters (Weber, 1972).

Adsorption involves the interphase accumulation or concentration of substances at a surface interface. The process can occur at an interface between any two phases, such as, liquid-liquid, gas-liquid, gas-solid, or liquid-solid interfaces. The material being concentrated or adsorbed is the adsorbate, and the adsorbing phase is termed the adsorbent (Weber, 1972).

Adsorption, conversely, is a process in which the, molecules or atoms of one phase interpenetrate nearly uniformly among those of another phase to form a "solution" with the second phase. The term sorption, which includes both adsorption and absorption, is a general expression for a process in which a component moves from one phase to be accumulated in another, particularly for cases in which in which the second phase is solid (Weber, 1972).

Adsorption from solution onto a solid occurs as the results of one of two characteristics properties for a given solvent-solute-solid system, or a combination thereof. The primary driving force for adsorption maybe a consequence lyophobic (solvent disliking) character of the solute relative to the particular solvent or high affinity of the solute for the solid. For the majority of systems encountered in water and wastewater treatment practice, adsorption results from the combined action of the two forces. Each of the two primary causes for adsorption can be affected to varying degrees by a number of factors. The degree of solubility of a dissolved substance is by far the most significant factor in determining the intensity of the first of the two driving forces. In the present context, "degree" of solubility can be thought of as the extent of chemical compatibility between a solvent system and a solute (Weber, 19720).

2.5.1 Adsorption Isotherm

According to Weber (1972), in adsorption in a solid liquid system, the distribution ration of the solute between the liquid and the solid phases are the measurement of the position of equilibrium. The preferred form of depicting this distribution is to express the quantity, q_e as a function of C_e at a fixed temperature, the quantity, q_e being the amount of solute adsorbed per unit weight of the solid adsorbent, and C_e the concentration of the solute remaining in solution at equilibrium. An expression of this type is termed as adsorption isotherm. The Langmuir adsorption isotherm is given as:

$$q_e = \frac{(Q_0 b C_e)}{(1 + bC_e)} \tag{1}$$

The other two convenient forms of Langmuir Equations are;

$$\frac{C_e}{q_e} = \frac{1}{bQ_0} + \frac{C_e}{Q_0} \tag{2}$$

or

$$\frac{1}{q_e} = \frac{1}{Q_0} + \left(\frac{1}{bQ_0}\right)\left(\frac{1}{C_e}\right) \tag{3}$$

Either of these forms may be used for linearization of data that accord with the Langmuir equation. The form chosen usually depends on the range and spread of the data and on the particular data to be emphasized. Although the basic assumptions explicit in the development of the Langmuir isotherm are not met in most adsorption system concerning water and wastewater treatment, the Langmuir isotherm has been found particularly useful for description of equilibrium data for such systems providing parameters (Q_0 and b) with which to quantitatively compare adsorption behaviour in different adsorbate-adsorbent systems.

One other equation for isothermal adsorption, the Freundlich or van Bemmelen equation, has been widely used for many years. This equation is a special case for heterogeneous surface energies in which the energy term, b, in the Langmuir equation (Equation 1) varies as a function of surface coverage, q_e, due to variations in heat of adsorption.

The Freundlich equation has the general form of:

$$q_e = K_F C^{\frac{1}{n}} \tag{4}$$

where K_F and n are constants, and $n > 1$.

The Freundlich equation is basically empirical; but is often useful as a means of data description. Data are usually fitted to the logarithmic form of equation

$$\log q_e = \log K_F + \frac{1}{n} \log C \tag{5}$$

Which gives a straight line with a slope of 1/n and intercept equal to the value of the log K_F for $C_e = 1$. The intercept is roughly an indicator of sorption capacity and the slope, 1/n, of adsorption intensity. The Freundlich equation generally agrees quite well with the Langmuir equation and experimental data over moderate ranges of the concentration, C_e. Unlike the Langmuir equation, however, it does not reduce to a linear adsorption expression at very low concentrations. Nor does it agree well with the Langmuir equation at high temperatures, since n must reach some limit when the surface is fully covered.

2.5.2 Adsorption Kinetics

Several kinetic models are in use to explain the mechanism of the adsorption process. A simple pseudo first order equation due to Lagergen was used by Ho & Mckay (1998, 1999):

$$\frac{d_{qt}}{dt} = k_{ad} \left(q_e - q_t\right) \tag{6}$$

Where: q_e, q_t = the amount of adsorption at equilibrium and at time t respectively

k_{ad} = rate constant of the pseudo first-order adsorption process

The integrated rate law after application of the initial condition of $q_t = 0$ at $t = 0$, becomes:

$$\log(q_e - q_t) = \log q_e - \frac{k_{ad} t}{2.303} \tag{7}$$

Plot of log ($q_e - q_t$) versus t gives a straight line for first order adsorption kinetics, which allow computation of the adsorption rate constant, k_{ad}.

This equation differs from a true first order equation in two ways: (i) the parameter kad ($q_e - q_t$) does not represent the number of available sites, and (ii) the parameter loq (q_e) is an adjustable parameter and often it is found to be not equal to the intercept of the plot of log ($q_e - q_t$) versus t, whereas in a true first order log (q_e) should be equal to the intercept. In such cases, applicability of the second order kinetics has to be tested with the rate equation (Sharma & Bhattacharyya, 2004; Ozturk & Kavak, 2005):

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \tag{8}$$

Where k_2 = the second order rate constant in g/(mg)(min)

From the boundary conditions, t = 0 to t = t and qt = 0 to qt = qt, the integrated form of the equation becomes:

$$\frac{1}{(q_e - q_t)} = \frac{1}{q_e} + k_2 t \tag{9}$$

which can be written in the linear form,

$$\frac{t}{q_t} = \frac{1}{h} + \left(\frac{1}{q_e}\right) t \tag{10}$$

Where $h = k_2qe^2$ can be regarded as the initial sorption rate as t → 0. Under such circumstances, the plot of $\frac{t}{q_t}$ vs. t should give a linear relationship, which allows computation of q_e, k and h.

CHAPTER 3

METHODOLOGY

3.1 Research Methodology

This chapter describes the preparation of experimental materials as well as their characterization, standard-curve, experimental procedure for adsorption and adsorption isotherms.

3.1.1 Preparation of Banana Peel Adsorbent

The preparation of the banana peel adsorbent was based on the method used by Gupta et al., (2009). The banana peel was sourced from the local market and restaurants. First, the peels were separated from the fruit gently and were washed thoroughly. This was done in order to remove dust, soil and other materials adhering to the peels. After that, the clean banana peels was dried in sun light for 5 days and then subsequently in a hot air oven at 80°C overnight. The dried banana peels was then cut into small pieces and ground in a mixer. It was followed by sieving to get the biomass particles of 0.2-0.4 mm diameter.

3.1.2 Dye Concentration and Absorbance

One reactive dye which is Reactive Yellow 15 was used in this study. Dye concentration was determined by finding the maximum wavelength of absorbance for dye in concentration of 20 mg/L. To obtain 20 mg/L dye concentration, first, one stock solution of 100 mg/L was composed by adding 0.1000 g of reactive dye in powder form into 1 Litre of distilled water. It is a necessity to ensure that the reactive dye was exactly 0.100 g in amount using digital analytical balance to avoid any errors later in adsorption studies.

Then, the stock solution of 100 mg/L was diluted by mixing 100 mL of the stock solution with 400 mL of distilled water in 500 mL volumetric flask to obtain 20 mg/L dye concentration. After that, the 20 mg/L dye concentration was analysed by spectrophotometer to obtain dye absorbance and wavelength by setting manually the numerous values of wavelength. Distilled water was used as a blank sample for each dye absorbance determination of new wavelength. At the end of the test, wavelength at highest dye absorbance was chosen for further studies used.

By using wavelength at highest dye absorbance of 20 mg/L dye concentration, dye absorbances for other concentrations were measured. Standard curve was plotted from the results obtained and can be referred in further studies.

3.1.3 Preliminary Test

A preliminary adsorption test was conducted to show the potential of banana peel to adsorb dye from water. The test was conducted by taking an aqueous solution of reactive dye yellow placed on the orbital shaker (140 rpm) for 24 h with 0.5 g sieved banana peel adsorbent. After 24 h, the solution was filtered through 0.45 μm membrane filter and analysed to check the amount of dye adsorbed.

3.1.4 Batch Adsorption Test

Batch adsorption test was conducted at room temperature (22°C) on an orbital shaker at a speed of 150 rpm. A test of percentage adsorption was conducted against pH, with time and adsorbent dosage being set as constant. A second test was conducted to obtain the optimum contact time with optimum pH and adsorbent dosage being set as constant. After that, the effect of adsorbent dosage or concentration on adsorption was determined with optimum pH and optimum contact time.

Equilibrium adsorption test was conducted under optimum pH and contact time by adding amounts of banana peel adsorbent to 100 mL of dye–bearing water and agitated on the orbital shaker. At the end of the equilibration, the reaction mixture was filtered through 0.45 μm membrane filter and analysed for.

3.1.4.1 Effect of pH

One of the important factors affecting adsorption process is pH of the solution. Various studies have been conducted to prove this statement (Annadurai et al., 2002; Wong et al., 2009; Guendy, 2010). The effect of pH was investigated in the range of 1-10. The experiment was conducted at standard room temperature of 22°C, using a fixed dosage of adsorbent and contact time of 24 h.

3.1.4.2 Effect of Contact Time

Contact time is an important factor affecting adsorption efficiency (Wong et al., 2009; Amin, 2008). In order to determine the optimum contact time, experiments was conducted by adding a fixed amount of adsorbent dosage into flasks with dye–bearing water at optimum pH and agitated for 30 min to 4 h. The data was used to study the adsorption kinetics — pseudo-first-order or pseudo-second-order, indicating the nature of adsorption

3.1.4.3 Effect of Adsorbent Dose

The effect of adsorbent dose on adsorption of dye was studied using different dosage of the adsorbent. 100 mL of dye–bearing water was added in each conical flask containing the adsorbent in the range of 0.2 to1.2 g.

3.1.5 Adsorption Isotherm

The adsorption isotherm was conducted soon after the batch studies were completed. It was carried out by using ten different concentrations of reactive dye varying from 10 to 100 mg/L. Contact time, pH and carbon dosage for dye were determined from the optimum condition in batch studies. The final solution was then measured by using spectrophotometer for dye absorbance.

3.1.6 Adsorption Kinetics

Adsorption kinetics study was carried out by varying the contact time of the adsorption from 15 to 240 min. pH and carbon dosage for dye were determined from the optimum condition in batch studies. The final solution was then measured by using spectrophotometer for dye absorbance.

3.1.7 Field Emission Scanning Electron Microscope (FESEM)

Field Emission Scanning Electron Microscope (FESEM) is a microscope that uses electrons rather than light to form an image and it has a large depth of field, which allows a large amount of sample to be in focus at one time. It also produces images of high resolution, which means that closely spaced features can be examined at a high magnification. Since most FESEMs only require the sample to be conductive, preparation of samples is relatively easy.

3.1.8 Fourier Transform Infrared (FT-IR) Spectroscopic Analysis

Fourier Transform Infrared Spectroscopy was used to characterize the functional group and the constituents of the banana peel adsorbent. In addition, FT-IR also is frequently capable of distinguishing adsorbed molecules on different sites and the amount of adsorption on respective sites is separately obtainable from the IR peak intensities.

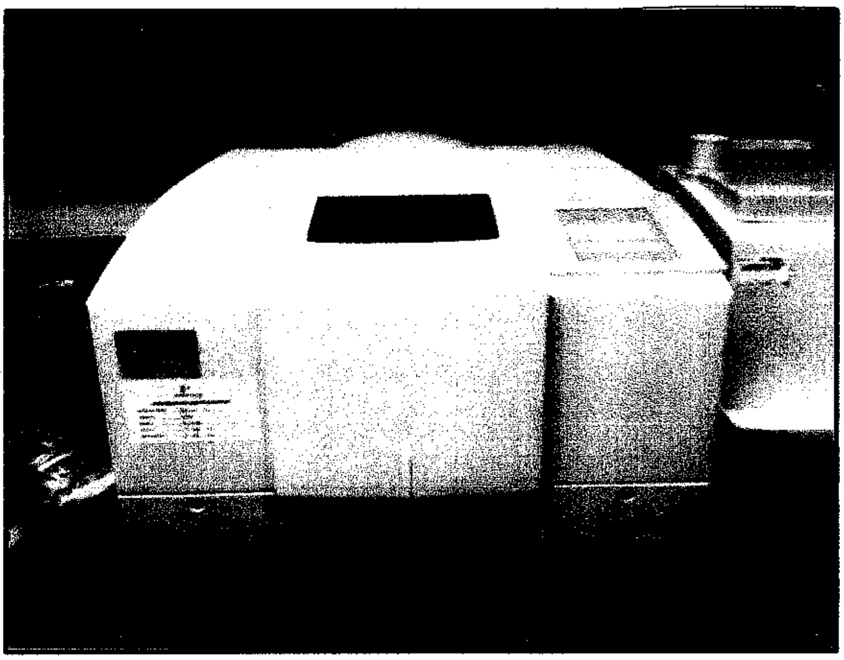

Figure 3.1: Fourier Transform Infrared Spectroscopy

3.2 Apparatus and Chemicals

3.2.1 Apparatus

1. Pipettes
2. Volumetric flasks
3. Orbital shaker
4. Oven
5. Sieve
6. Reagent bottles
7. 0.45 μm membrane filter and filtration apparatus
8. Analytical balance
9. 250 mL Erlenmeyer flasks
10. Spectrophotometer 5000
11. 50 mm sample cells
12. Field Emission Scanning Electron Microscope
13. Fourier Transform Infrared Spectroscopy

3.2.2 Chemicals and Materials

1. C.I Reactive Yellow 15

3. Sodium hydroxide, NaOH

4. Hydrochloric acid, HCl

6. Distilled water

7. Banana peel

3. 3 Project Activities and Key Milestones

Several targets have been set for the FYP I and FYP II. Table 2.0 and Table 3.0 show the project activities and key milestones for FYP I and FYP II respectively.

Table 3.1: Project Activities and Key Milestones for FYP I

No.	Detail/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Selection of Project Topic	▨							MID SEM BREAK							
2	Literatures Review		▨	▨	▨	▨										
3	Submission of Extended Proposal						○									
4	Preparation Banana Peel Adsorbent									▨	▨	▨				
5	Proposal Defence											○				
6	Preliminary Experiment												▨	▨		
7	Submission of Interim Draft Report														○	
8	Submission of Interim Report															○

Legends:

▨ Project Activity

○ Key Milestone

Table 3.2: Project Activities and Key Milestones for FYP II

No.	Detail/ Week	1	2	3	4	5	6	7	MID SEM BREAK	8	9	10	11	12	13	14	15
1	Experiment Execution	▨	▨	▨	▨	▨	▨	▨		▨	▨	▨	▨				
2	Submission of Progress Report									○							
3	Compare the Efficiency with other Adsorbents												▨				
4	Pre-EDX												○				
5	Submission of Draft Report													○			
6	Submission of Dissertation (soft bound)														○		
7	Submission of Technical Paper														○		
8	Oral Presentation															○	
9	Submission of Project Dissertation (Hard Bound)																○

Legends:

▨ Project Activity

○ Key Milestone

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Preparation of Banana Peel Adsorbent

The banana peel adsorbent was prepared. First, the banana peel was dried under sunlight for 5 days. Then, the banana peel was dried in 80°C for 24 hours. After that, the banana peel was ground in a mixer followed by sieving to get the particles of 0.2-0.4 mm diameter.

Step 1: Select, wash and dry banana peel

Step 2: Dry in oven (80°C) for 24 hours

Step 3: Grinding of banana peel

Step 4: Sieve banana peel

4.2 Dye Concentration and Absorbance

One type of reactive dye namely Reactive Yellow 15 was investigated in this study. After testing the dye solution spectrophotometrically, the results of absorbance for the reactive dye were plotted in Figure 4.1.

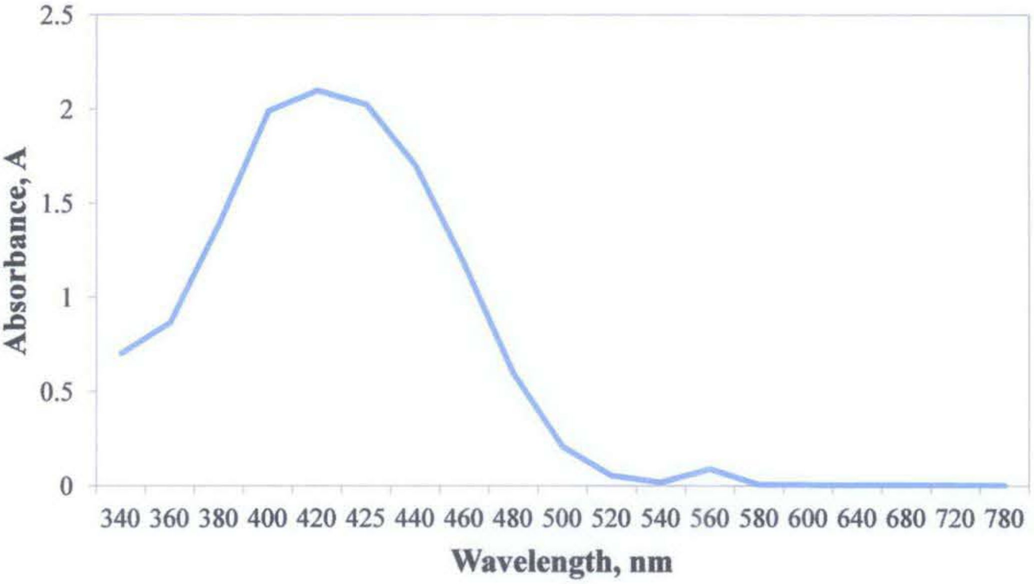

Figure 4.1: Wavelength for Reactive Yellow 15

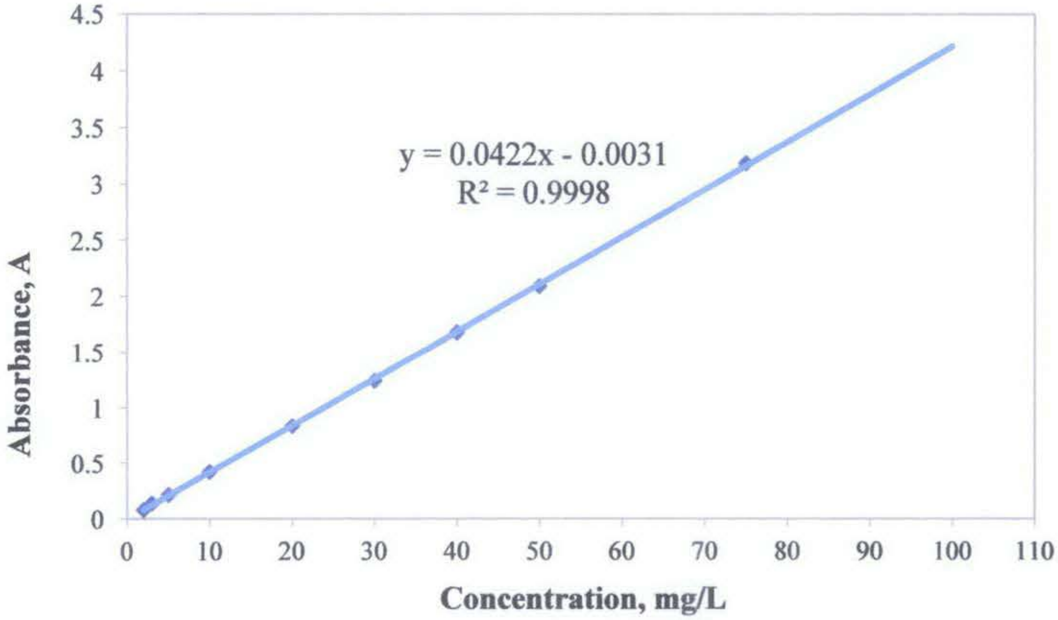

Figure 4.2: Standard Curve for Reactive Yellow 15 at Wavelength of 420 nm

Maximum dye absorbance for Reactive Yellow 15 was found at wavelength of 420 nm. By plotting the standard curve (Figure 4.2) for other dye concentrations based on 420 nm wavelength, unknown dye concentration based on absorbance measured can be examined easily. For further studies, the final concentration of dye can be determined by referring to the standard curve.

4.3 pH Measurement

Test to determine the pH of banana peel adsorbent was conducted three times using pH meter. The pH was quantified by immersing 1.0 g of the banana peel powder in 100 mL of distilled water. The solution was then stirred for about 1 hour before the pH of the slurry was measured. The data for each test were recorded as followed:

Table 4.1 pH of banana peel adsorbent

Sample	pH
1	5.64
2	5.64
3	5.62

From the data, mean value of pH for banana peel adsorbent was at pH 5.63.

4.4 Characterization of Banana Peel Adsorbent

The adsorbent were subjected to SEM (Scanning Electron Microscope) analysis. The SEM was carried out on a Carl Zeiss AG SUPRA 55VP, field emission scanning electron microscope. The Fourier Transform infrared (FT-IR) spectra were obtained on a Perkin Elmer, Spectrum One/BX model, Fourier Transform infrared spectroscope.

4.4.1 Fourier Transform Infrared (FT-IR) Spectroscopy

The prepared banana peel adsorbent was sent for Fourier Transform Infrared Spectroscopy (FT-IR) analysis. The purpose of this is to determine the chemical contents and composition of the sample. FT-IR spectra were recorded over the wavenumber range 400–4000 cm^{-1}. The major constituents of banana peel are cellulose and protein. The FT-IR spectral data of native and surface modified fruit peels are given in Table 1. The FT-IR spectra of banana peel indicated that this adsorbent contains carboxyl and hydroxyl groups. A broad peak at 3200 to 3400 cm^{-1} in Figure 4.3 is due to the hydroxyl (OH) functioning group. A slight peak at 2800 – 3100 cm^{-1} is due to compounds containing CH group. Besides that, it can be clearly seen that the banana peel contains protein as there is a broad peak at 1640 to 1760 cm^{-1}.

Table 4.2: The FT-IR spectral data of native and surface modified fruit peels (Thirumavalan., 2011)

Modes	Frequency (cm^{-1})
OH Stretching	3200 - 3400
CH Stretching	2800 - 3100
COO Stretching	1745
C=O Stretching	1640 - 1760
CH Bending	1320
CO Stretching	1020 - 1300
COC Stretching	1150

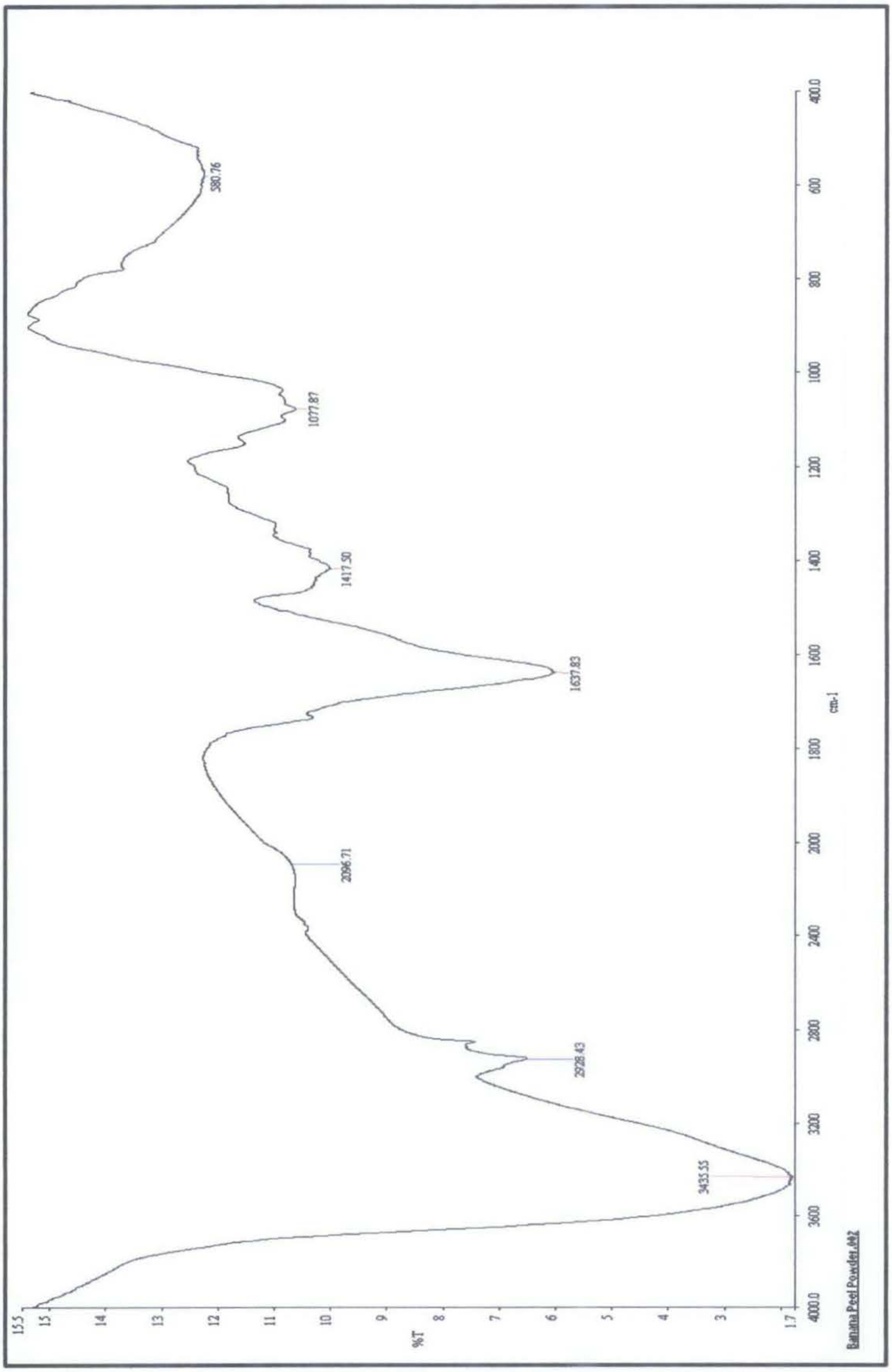

Figure 4.3: FT-IR spectra of banana peel

4.4.2 Field Emission Scanning Electron Microscope (FESEM)

The prepared banana peel adsorbent was sent for scanning in the FESEM. The purpose of this is to determine the sample's surface topography and composition. Scanning electron micrographs of banana peel for 50x magnification and 1000x magnification are shown in Figure 4.5. The composition of banana peel are shown in Table 4.4

The elements of C and O are found to be the primary components in banana peel sample, each accounting 54.52% and 42.58% of the weight of the sample respectively.

Table 4.3: Composition of banana peel adsorbent

Element	Weight (%)	Atomic (%)
C K	54.52	62.59
O K	42.58	36.27
K K	2.9	1.01
Fe K	0.55	0.14
Total	100	

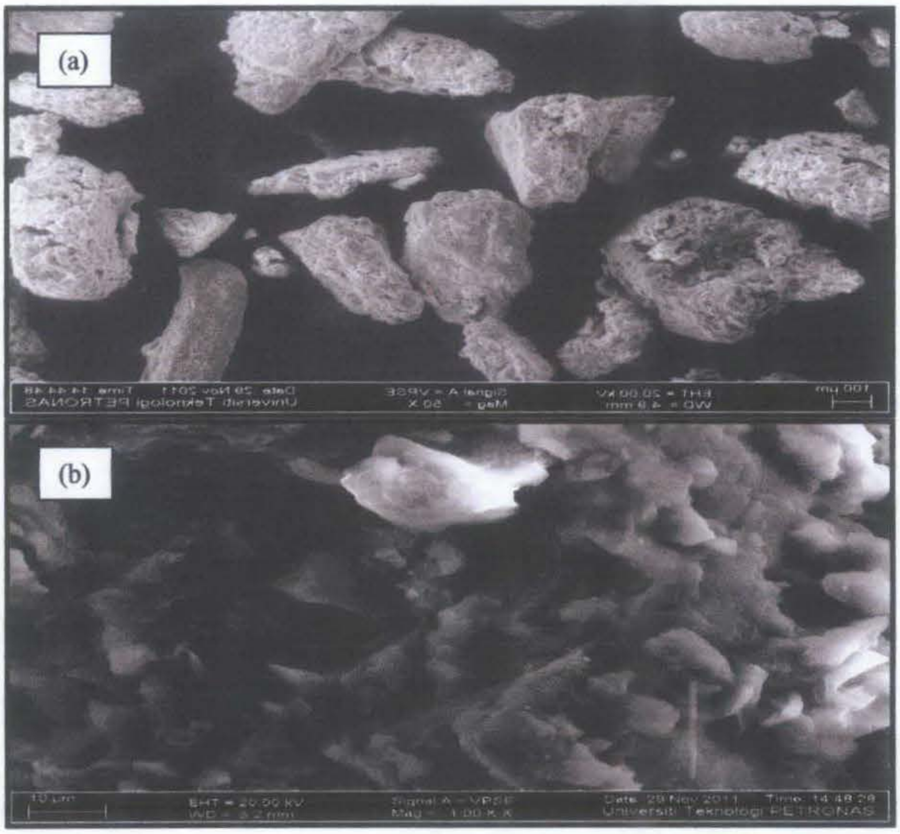

Figure 4.4: SEM of banana peel (a) 50x (b) 1000x

4.5 Adsorption Study

Batch studies were carried out to examine the removal efficiency of prepared banana peel adsorbent. During the analysis, effect pH was investigated.

4.5.1 Effect of pH

In order to obtain the desirable pH for the dye solution, 0.1 N NaOH and 0.1 N HCl were used. The concentration of dye used was 20 mg/L with pH varied from 2 to 10 for each sample. The results were summarized in Table 4.4.

Table 4.4: Effect of pH on removal of Reactive Yellow 15

pH	Initial Concentration (mg/L)	Final Concentration (mg/L)	% Removal
1	20	4.54	77.30
2	20	3.45	82.75
3	20	9.52	52.40
4	20	14.61	26.95
5	20	14.3	28.50
6	20	14.1	29.50
7	20	13.21	33.95
8	20	13.81	30.95
9	20	13.15	34.25
10	20	12.2	39.00

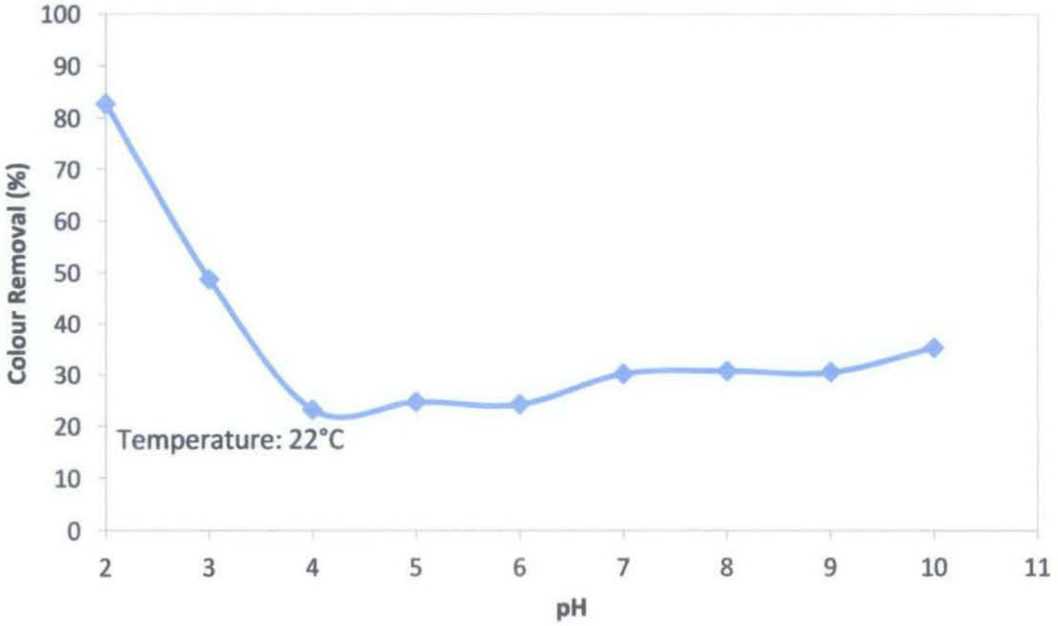

Figure 4.5: Effect of pH on adsorption of Reactive Yellow 15

This experiment was conducted with standard room temperature of 22°C, using adsorbent dosage of 0.5 g/L and using maximum contact time of 24 h. Results in Figure 4.3 clearly indicate that the maximum adsorption of Reactive Yellow 15 on banana peel were found in the acidic range of 1-3. Amin (2008) also indicates similar observations. The positive charges on the surface increased as the pH of the adsorption solution was lowered. This behaviour would attract the negatively charged functional group located in the reactive dye. The optimum pH for further studies was decided at pH 2.0 at which the removal was optimum.

4.5.2 Effect of Contact Time and Dye Concentration

Effects of contact time and dye concentration on adsorption of Reactive Yellow 15 by banana peel adsorbent are shown in Figure 4.5. The extent of dye adsorption increased both as the initial dye concentration decreased and also the contact time increased. Equilibrium adsorption was attained in 3 h. Similar results for adsorption of reactive dyes on adsorbent prepared from peach stones (Gercel et al. 2009) and coconut tree flower and jute fibre (Senthilkumar et al. 2006) have been reported. A contact time of 3 h was used in all subsequent tests. The data recorded from the test can be referred in Table 4.5.

Table 4.5: Effect of initial concentration and contact time on removal of Reactive Yellow 15

Contact Time (m)	Initial Concentration = 40 mg/L		Initial Concentration = 20 mg/L	
	Final Concentration (mg/L)	% Removal	Final Concentration (mg/L)	% Removal
0.25	8.03	59.83	19.22	51.95
0.50	5.90	70.50	13.96	65.10
0.75	5.83	70.85	11.31	71.74
1.00	5.36	73.22	9.86	75.35
1.50	5.07	74.64	8.25	79.38
2.00	4.93	75.36	7.99	80.03
3.00	4.48	77.61	7.23	81.93
4.00	4.41	77.96	7.16	82.10

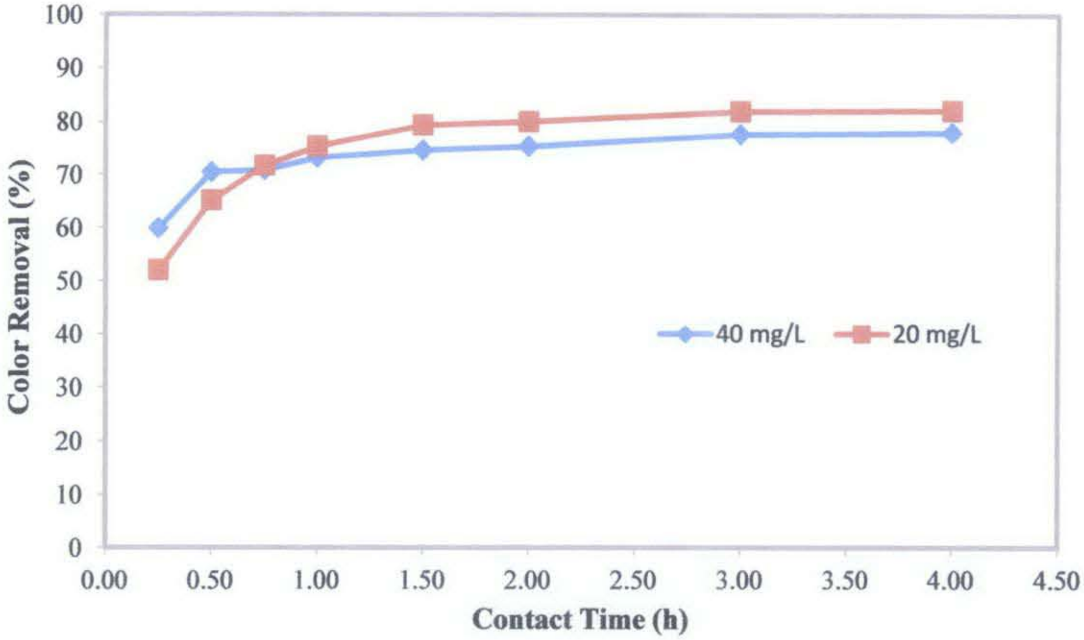

Figure 4.6: Effect of contact time and dye concentration on adsorption of Reactive Yellow 15

4.5.3 Effect of Adsorbent Dose

Dyes in the concentration of 20 mg/L were used to investigate the effect of carbon dose where the amount of banana peel adsorbent added in each container were varied from 0.2 g to 1.2 g. The pH value for all solutions was maintained at pH 2.0. The data recorded from the test can be referred as follow.

Table 4.6: Effect of adsorbent dose on removal of Reactive Yellow 15

Adsorbent Dose (g)	Reactive Yellow 15 (20 mg/L)		Reactive Yellow 15 (40 mg/L)	
	Final Concentration (mg/L)	% Removal	Final Concentration (mg/L)	% Removal
2	5.78	71.09	12.16	69.60
4	4.91	75.47	10.14	74.64
5	4.23	78.85	8.96	77.60
6	3.66	81.71	8.34	79.14
8	3.65	81.73	8.25	79.38
10	3.64	81.78	8.18	79.56
12	3.64	81.81	8.11	79.73

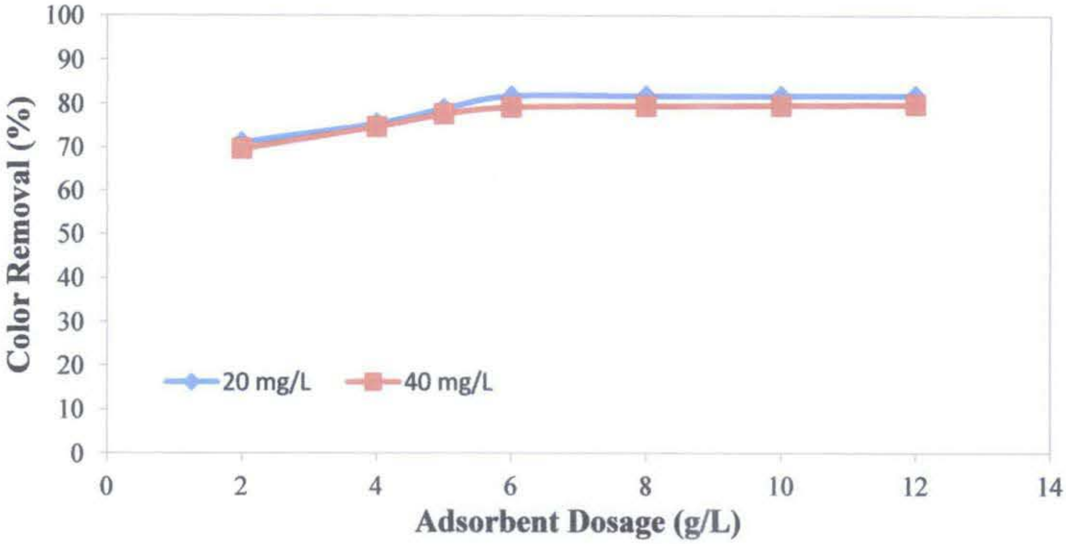

Figure 4.7: Effect of adsorbent dose on adsorption of Reactive Yellow 15

It was observed from the graph that the dye uptake increased with increase amount of banana peel adsorbent added to the flask. After 3 hours, colour removal of about 82% and 79% was achieved at 6 g/L for 20 mg/L and 40 mg/L respectively.

4.6 Adsorption Isotherm

The adsorption isotherm was carried out by using ten different initial concentrations of both reactive dyes varied from 10 to 100 mg/L. The pH of each solution was adjusted at pH 2.0 with adsorbent dosage of 0.6 g was added into each solution. The 100 mL dye solution was then rotated for 3 hours contact time and the final solution was measured spectrophotometrically for dye absorbance. The data obtained from both experiments was fitted and compared with Langmuir and Freundlich models.

4.6.1 The Langmuir Adsorption Isotherm

Figure 4.7 indicate the Langmuir adsorption isotherm for Reactive Yellow 15. It was found out that the adsorption of Reactive Yellow 15 onto banana peel was best fitted using Langmuir adsorption isotherm.

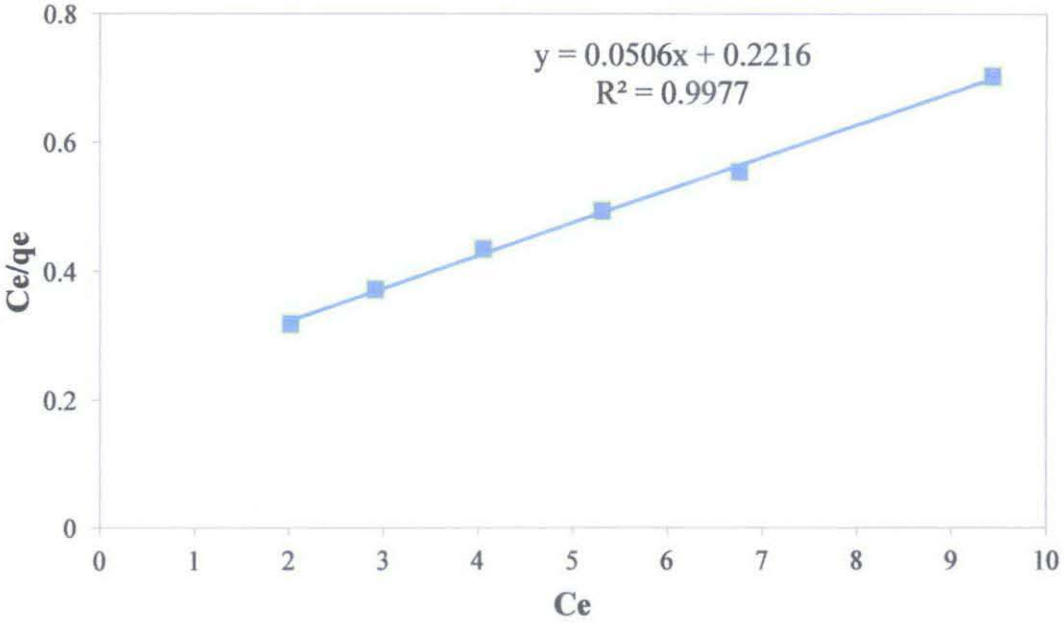

Figure 4.8: Langmuir adsorption isotherm for Reactive Yellow 15 adsorption

By referring to Equation 2 and comparing with the equation on Figure 4.7, values of constants were calculated. The value of Q_0 indicated the number of moles of solute adsorbed per unit weight of adsorbent in forming a complete monolayer on the surface and b was a constant related to energy of adsorption.

Table 4.7: Empirical constant of Langmuir adsorption isotherm

Adsorbent	Langmuir Constant	
	Q_0	b
Banana Peel	10.90	0.88

4.6.2 The Freundlich Adsorption Isotherm

From the same adsorption data, Freundlich adsorption was plotted to best fitted regression line as in Figure 4.8.

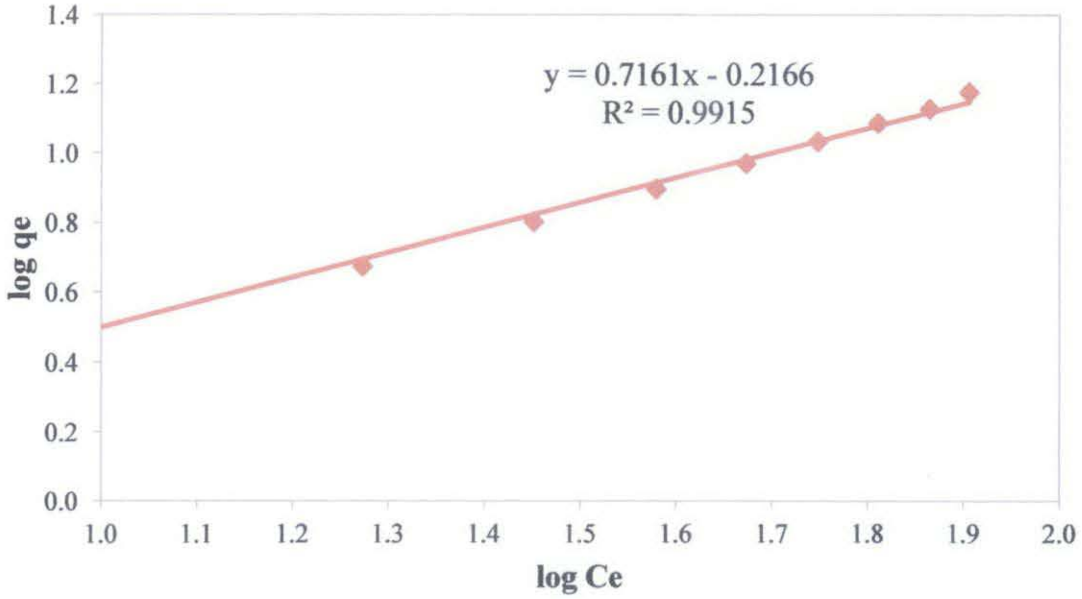

Figure 4.9: Freundlich adsorption isotherm for Reactive Yellow 15 adsorption

The value of K_F (intercept) and 1/n (slope) are roughly an indicator of adsorption capacity and adsorption energy of the adsorbent. By referring to Equation 5 and comparing with equation in Figure 4.8, value of K_F and 1/n were calculated and recoded in Table 4.8.

Table 4.8: Empirical constant of Freundlich adsorption isotherm

Adsorbent	Freundlich Constant	
	1/n	K_F
Banana Peel	0.08	3.3

4.7 Adsorption Kinetics

Pseudo-first-order and pseudo-second-order are presented in Figure 4.9 and Figure 4.10 respectively. As can be seen from both figures, the values of R^2 for the pseudo-second-order model were comparatively higher than those for the pseudo-first-order model indicating that the pseudo-second-order model gave a better fit to the experimental data.

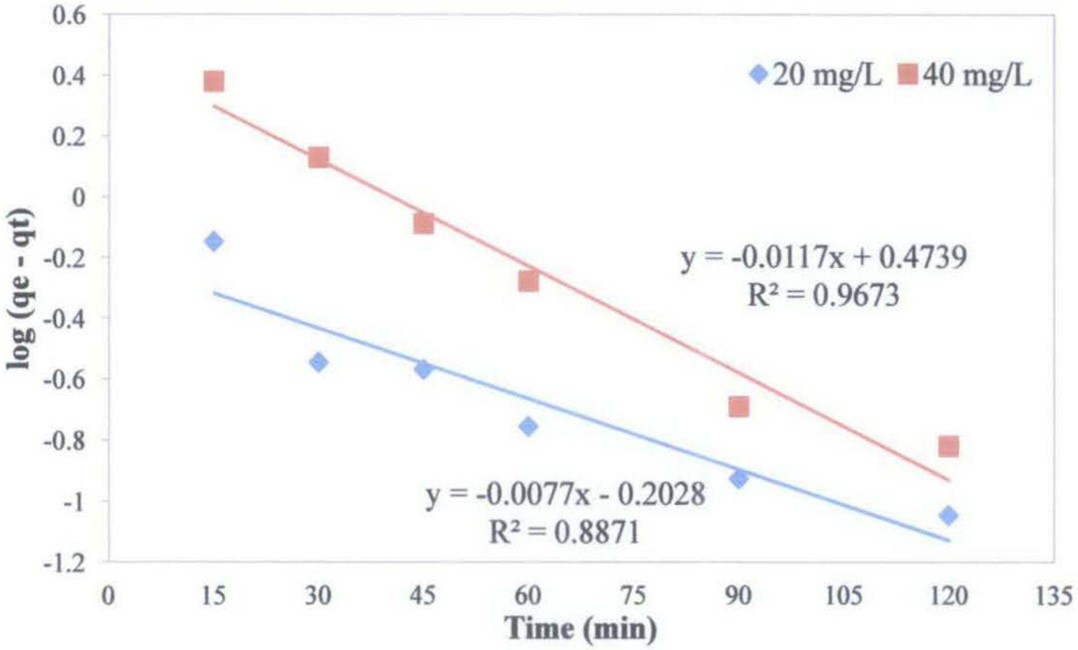

Figure 4.10: Application of the pseudo-first-order kinetics model to the adsorption of Reactive Yellow 15 onto banana peel

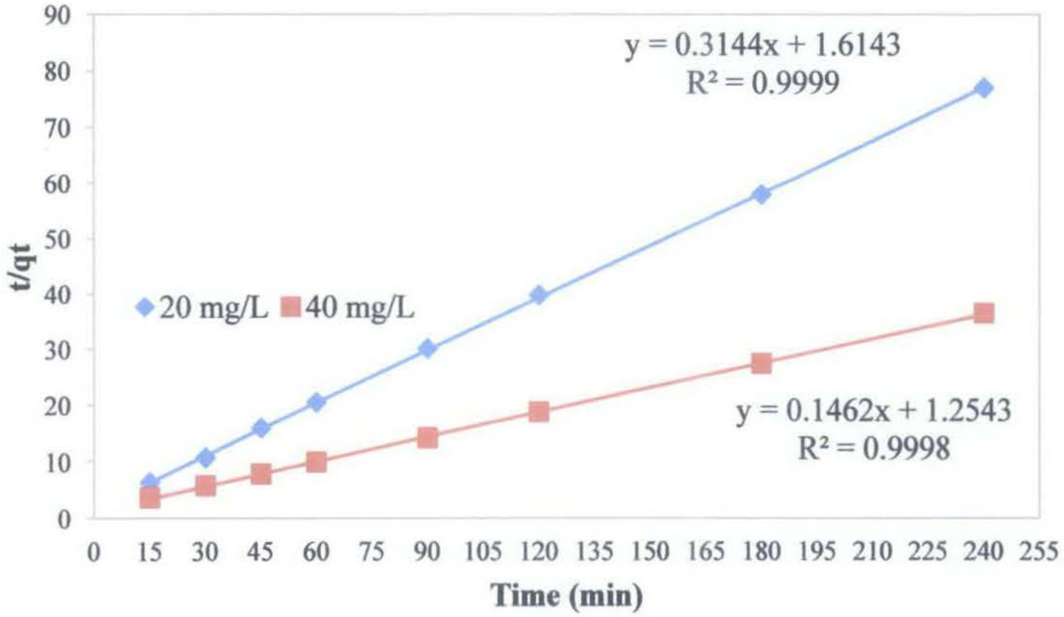

Figure 4.11: Application of the pseudo-second-order kinetics model to the adsorption of Reactive Yellow 15 onto banana peel

4.8 Comparison between Reactive Dye Adsorption of Banana Peel against Other Plant Based Adsorbents

Table 4.9 lists and compares the maximum monolayer adsorption capacity of some dyes on various adsorbents. Compared with some data in the literature, Table 2 shows that the banana peel studied in this work has a comparable adsorption capacity.

Table 4.9: Comparison between reactive dye adsorption of banana peel against other plant based adsorbents

Dyes	Adsorbent	Adsorption Capacity (mg/g)	Reference
Reactive Yellow 15	Banana peel	10.90	This study
Acid Brilliant Blue	Banana pith	4.3	(Aksu & Tezer, 2000)
Acid Violet	Coir pith	1.6	(Namasivayam et al., 2001)
Acid Brilliant Blue	Coir pith	16.6	(Namasivayam et al., 2001)
Rhodamine B	Sugar cane dust	4.26	(Khattri & Singh, 1999)
Rhodamine B	Orange peel	3.23	(Namasivayam et al., 1996)
Procion Orange	Orange peel	1.33	(Namasivayam et al., 1996)

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The present research indicates that adsorbent prepared from banana peel is a suitable for removal of reactive dye from aqueous solution. High cellulose contents of banana peel is suitable factor which can enhance the rate of adsorption process. In adsorption batch studies, it was shown that dye uptake increased with an increase in contact time and adsorbent dosage and decrease in dye concentration. The banana peel optimum time for adsorption is 3 hours, optimum pH is 2 and optimum dose is 6 g/L. Equilibrium adsorption data were well described by Langmuir and Freundlich adsorption isotherm models. Reactive Yellow 15 adsorption by banana peel is better expressed by pseudo-second-order kinetic model. From this research, it can be concluded that adsorbent prepared from banana peel proved as one of successful low-cost alternative to remove reactive dyes from aqueous solution.

5.2 Recommendations

Few other experiments should be conducted by using real effluent so that the effectiveness of applying banana peel to treat textile wastewater can be observed.

Desorption studies should be carried out to determine the maximum number of time the banana peel can be used.

In addition, comparison studies against commercial activated carbon should be carried out to determine the efficiency in replacing commercial activated carbon in adsorptive removal of reactive dyes.

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APPENDICES

APPENDIX 1
DILUTION FACTOR FOR DYE SOLUTION OF 100 mg/L

APPENDIX 1: DILUTION FACTOR

Table A1: Dilution factor for 100 mg/L dye stock solution

Dye concentration to be obtained (mg/L)	Volume of stock solution to be added (mL)	Volume of distilled water to be added (mL)	Total volume of dye concentration (mL)
2.5	6.25	243.75	250
5	12.5	237.5	250
10	25	225	250
12.5	31.25	218.75	250
15	37.5	212.5	250
20	50	200	250
25	62.5	187.5	250
30	75	175	250
40	100	150	250
50	125	125	250
60	150	100	250
70	175	75	250
80	200	50	250
90	225	25	250

APPENDIX 2

DYE CONCENTRATION AND ABSORBANCE DATA SHEET

APPENDIX 2: DYE CONCENTRATION AND ABSORBANCE

C.I. REACTIVE YELLOW 15

Table A2: Wavelength and dye absorbance

Wavelength (nm)	Dye absorbance
340	0.701
360	0.866
380	1.391
400	1.991
420	**2.098**
425	2.024
440	1.703
460	1.173
480	0.596
500	0.208
520	0.054
540	0.018
560	0.09
580	0.007
600	0.006
640	0.005
680	0.005
720	0.004
780	0.003

APPENDIX 3
ADSORPTION ISOTHERM

APPENDIX 3: DYE CONCENTRATION AND ABSORBANCE

LANGMUIR ADSORPTION ISOTHERM

Table A3: Langmuir calculation for C. I. Reactive Yellow 15 (Figure 4.8)

C_o	C_e	$C_o - C_e$	$(C_o - C_e) / C_o$	V	$(C_o - C_e)V$	m	x/m	$C_e/(x/m)$
40	2.02	37.98	0.95	0.10	3.798341232	0.6	6.3306	0.318547632
50	2.92	47.08	0.94	0.10	4.708293839	0.6	7.8472	0.371734863
60	4.05	55.95	0.93	0.10	5.594549763	0.6	9.3242	0.434834173
70	5.31	64.69	0.92	0.10	6.468957346	0.6	10.782	0.492545514
80	6.76	73.24	0.92	0.10	7.324407583	0.6	12.207	0.55343104
90	9.43	80.57	0.90	0.10	8.056635071	0.6	13.428	0.702550075

FREUNDLICH ADSORPTION ISOTHERM

Table A4: Freundlich calculation for C. I. Reactive Yellow 15 (Figure 4.9)

Co	Ce	Co - Ce	(Co - Ce) / Co	V	(Co - Ce)V	m	x/m	Log Ce	Log (x/m)
20	1.23	18.77	0.94	0.10	1.876540284	0.6	3.12757	1.273357892	0.495206642
30	1.68	28.32	0.94	0.10	2.831516588	0.6	4.71919	1.45201911	0.67386786
40	2.02	37.98	0.95	0.10	3.798341232	0.6	6.33057	1.579593978	0.801442728
50	2.92	47.08	0.94	0.10	4.708293839	0.6	7.84716	1.672863559	0.894712308
60	4.05	55.95	0.93	0.10	5.594549763	0.6	9.32425	1.747765141	0.969613891
70	5.31	64.69	0.92	0.10	6.468957346	0.6	10.7816	1.810834288	1.032683037
80	6.76	73.24	0.92	0.10	7.324407583	0.6	12.2073	1.864772504	1.086621253
90	9.43	80.57	0.90	0.10	8.056635071	0.6	13.4277	1.906153693	1.128002442
100	9.95	90.05	0.90	0.10	9.00450237	0.6	15.0075	1.954459717	1.176308466