ADSORPTIVE REMOVAL OF TEXTILE DYE DIRECT BLUE 86 FROM AQUEOUS SOLUTION BY RICE HUSK-BASED ADSORBENT

By

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CERTIFICATION OF APPROVAL

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TRONOH, PERAK
MAY 2013

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.

MOHAMAD ZULBAHARI BIN MOHAMAD ZU

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ABSTRACT

Adsorption by activated carbon has proven to be most effective method of dye removal. However due to high production and regeneration cost of activated carbon, various studies on low-cost adsorbent have been carried out for alternative. Agricultural waste such as rice husk is seen to be a good adsorbent for dye removal. Moreover, rice husk can be easily obtained in any part of the country. In this study, rice husk-based adsorbents were prepared by chemical and thermal treatment. Standard curve of DB 86 at pH 1-10 were prepared to determine the concentration of unadsorbed dye in adsorption test at different pH. The adsorptive potential of the adsorbent for textile dye DB 86 was evaluated by batch adsorption screening test. The most effective adsorbent, RH6 was selected and the potential of RH6 was evaluated and compared to commercial PAC. The optimum pH for DB 86 removal is pH 2 for both adsorbent. The optimum contact time were observed to be 3 hours with 20 mg/L of dye concentration for RH6 and PAC. The optimum adsorbent dosage is 4 g/L for RH6 and 3 g/L for PAC. Both Langmuir and Freundlich provide high correlation coefficients R^2 (>0.97) but Langmuir isotherm is the best to describe the process with correlation coefficients R^2 >0.99. Adsorption capacity obtained was 34.4828 mg/g for RH6 and 47.6190 mg/g for PAC. Pseudo second order kinetic model yielded high R^2 values (>0.99) to prove that the model is best fit for the adsorption mechanism of RH6 and PAC compared to pseudo first order.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Historical record of the use of natural dyes extracted from vegetables, fruits, flowers, certain inserts and fish dating back to 3500BC have been found (Kant, 2012). The synthetic dyes discovered by W. H. Perkins in 1856 have provided a wide range of dyes that are colour fast and come in wider colour range and brighter shades (Whitaker and Willock, 1949). Dye is one of the major pollutants that can be found in wastewater of most of textile producing countries like Indonesia, China, India and Malaysia. There are about 10,000 different commercial textile dyes with estimated annual production of 7×10^5 metric tonnes (Baban et al., 2010; Robinson et al., 2001; Solomon et al., 2009). 10-25% of textile dyes are lost during the dyeing process and 2-20% is directly discharged as aqueous effluent in different environmental components (Zaharia and Suteu, 2012).

Effluent from the textile industry are highly coloured and their discharge into water channel makes water unsuitable for domestic, agricultural and industrial purposes (O’Mahony et al., 2002). The effluent containing dyes are not only toxic to aquatic life but also carcinogenic, which can cause cancer and other mutagenic diseases to living organism. Therefore, adequate treatment must be conducted to ensure the final discharge or disposal will not cause any disadvantage to society as well as the environment. The treatment of dye waste using conventional physical, chemical and biological method is costly (Robinson et al., 2002; Gong et al., 2005). Therefore, researchers currently focus on exploiting the use of low cost materials and waste biomass as potential absorbents for removal of dyes from dye waste. Rice husk is one of the high potential materials to be used as absorbent for dye waste treatment.

1.2 Problem Statement

1.2.1 Problem Identification

The treatment of dye waste is one of the major concerns due to difficulties faced with conventional methods (Safa and Bhatti, 2011). Moreover, presence of even small amounts of dyes in water is highly visible and undesirable (Crini, 2006). The discharge of industrial effluent containing dyes not only pollutes the rivers but also disturbs the growth of aquatic life by interfering in the transmission of sunlight and reducing the action for photosynthesis. Since dyes and their degradation products are carcinogenic and toxic, their removal process must not be left upon bio-degradation alone.

Adsorption technique is popular because the process is simple. Besides that, the effectiveness for removal of non-biodegradable pollutant including dyes from wastewater is proven (Aksu, 2005). According to Malik (2003), adsorption is the most effective method for dye removal since its sludge-free clean operation and complete removal of dyes even from a dilute solution. An activated carbon absorbent has good capacity for removal of organic pollutant, but it will cost more for the wastewater treatment. Therefore, the potential demand for absorbent made of low-cost materials and without other unnecessary pre-treatment is very high.

Rice husk (rice hull) is an agricultural waste consist that of cellulose (32.23%), hemicelluloses (21.34%), lignin (21.22%) and mineral ash (15.05%) (Rahman et al., 1997) with high percentage of silica, (96.34%) in the mineral ash (Rahman and Ismail, 1993). Therefore, rice husk is expected to be an effective absorbent for dye removal. However, the rice husk needs to be modified or treated before being applied for absorption of dyes (Chakraborty et al., 2011). Moreover, according to Daffala et al. (2010), chemical and thermal treatment would reduce cellulose, hemicelluloses and lignin crystalinity, leading to an increase of specific area for adsorption.

1.2.2 Relevancy of the Project

The coloured wastewater from industry (textile) released into ecosystem without proper treatment is the source of aesthetic pollution and disturbance to the aquatic life (Mane et al., 2007). Thus, dye removal is crucial in order to protect the marine/aquatic ecosystem. The dyes also cause allergy, skin irritation, cancer and mutagenic diseases to living organism (Anouzla et al., 2009). Even though there are various methods of colour removal, difficulties for textile dye waste treatment is unquestionable since other conventional method like physicochemical and biological treatment are costly (McKay, 1982).

Activated carbon adsorption is proven to be more effective than conventional methods due to high capacity adsorption of organic matter and micro-porous structures that increase the contact surface areas, but limited use due to high initial and regeneration cost. Therefore, rise husk-based adsorbent is a potentially low-cost replacement for the adsorbent used in the textile waste treatment system. Furthermore, this study should be able to contribute solution of dyes waste treatment which is not only effective but also economical. Hence, textile producing countries like India, China, Indonesia and Malaysia will surely gain the benefits of the new dye removal method. Since rice husk is an agricultural waste, it can be easily found in any part of the world accounting for about one-fifth of the annual gross rice production (545 million metric tonnes) of the world (Sharma et al., 2010).

This project should benefit the society since dye waste is a cause of significant amount of environmental degradation and human illness. About 40 percent globally used colorants contain organically bound, chlorine a known carcinogen. All the organic materials present in wastewater from a textile industry are at great concern in water treatment because they react with many disinfectants especially chlorine. Those chemicals in water will be evaporated and mix with the air we inhale or being absorbed through our skin then show up as allergic reactions and may cause harm to children even before birth (Kant, 2012). Thus, this project is relevant to human health, safety and environment.

1.3 Objectives and Scope of the Study

1.3.1 Objectives

- To prepare six rice husk-based adsorbents by chemical and thermal treatment and assess their potential in adsorbing the textile dyes Direct Blue 86 from aqueous solution.
- To identify and characterise the most effective adsorbent.
- To examine in detail adsorption of the textile dye Direct Blue 86 by the best rice husk-based adsorbent and a commercial activated carbon under varying environmental conditions.

1.3.2 Scope of the Study

- Preparation of six rice husk-based absorbent.
- Potential of the rice husk-based absorbent as low-cost absorbent for dye removal (Direct Blue 86).
- Parameters that affect the adsorption of the dye.
- The mechanism of adsorption of textile dye by the adsorbents.
- Comparison between the adsorbent and a commercial activated carbon adsorbent.

CHAPTER 2

LITERATURE REVIEW

2.1 Textile Dye

The dyes are natural and synthetic compounds that make the world more beautiful through various colourful products (Zaharia and Suteu, 2012). Colours are the main attraction of any fabric. Fabric was earlier being dyed with natural dyes but it gives limited and dull range of colours. Besides, they show low colour fastness when exposed to washing and sunlight (Kant, 2012). The synthetic dyes was discovered by W. H. Perkins in 1856 has provided a wide range of dyes that are colour fast and come in wider colour range and brighter shades (Whitaker and Willock, 1949). Since then, the synthetic dyes are widely used in textile industries.

These synthetic dyes are aromatic compounds produced by chemical synthesis and having into their structure aromatic rings that contain delocated electrons and also different functional groups (Zaharia and Suteu, 2012). Their colour is due to the chromogene-chromophore structure (acceptor of electrons) and the dyeing capacity is due to auxochrome groups (donor of electrons). The chromogene is constituted from an aromatic structure normally based on rings of benzene, naphthaline or antracene, from which are binding chromofores that contain double conjugated links with delocated electrons (Suteu et al., 2011; Welham, 2000).

The textile dyes are mainly classified in two different ways: (1) based on its application characteristics (i.e. CI Generic Name such as acid, basic, direct, disperse, mordant, reactive, sulphur dye, pigment, vat, azo insoluble), and (2) based on its chemical structure respectively (i.e. CI Constitution Number such as nitro, azo, carotenoid, diphenylmethane, xanthene, acridine, quinoline, indamine, sulphur, amino- and hydroxyl ketone, anthraquinone, indigoid, phthalocyanine, inorganic pigment, etc.) (Zaharia and Suteu, 2012).

2.2 Health, Safety and Environmental Concerns Associated with Dyes

Highly coloured wastes are not only aesthetically displeasing but also hinder the light penetration and may in consequence disturb biological processes in water bodies. The dye wastes are toxic and even carcinogenic, thus poses a serious threat to aquatic life. It also can cause allergy, skin irritation, cancer and other mutagenic diseases to living organism (Anouzla et al., 2009). The treatment of dye wastes is one of the major environmental concerns due to difficulties during conventional treatment methods. Various physical, chemical and biological treatments have been used for the treatment of dye wastes but these techniques are costly.

Furthermore, the colour in watercourse is accepted as an aesthetic problem rather than eco-toxic hazard. Thus, the public seems to accept blue, green or brown colour of river but the non-natural colour as red and purple usually cause most concern. The polluting effects of dyes against aquatic environment can be also the result of toxic effect due to their long time presence in environment (i.e. half-life time of several years), accumulation in sediments especially in fishes or other aquatic life forms and decomposition of pollutant in carcinogenic or mutagenic compound. (Zaharia and Suteu, 2012).

Several azo dyes cause damage of DNA that can lead to genesis of malignant tumours. Electron-donating substituent in ortho and para position can increase the carcinogenic potential. Toxicity diminished essentially with the protonation of aminic groups. Some of the best known azo dyes and their breakdown derivatives inducing cancer in humans and animals. In different toxicological studies indicated that 98% of dyes have a lethal concentration value LC_{50} for fishes higher than 1mg/L, and 59% have an LC_{50} value higher than 100 mg/L (Zaharia and Suteu, 2012). Other ecotoxicological studies indicated that over 18% of 200 dyes tested in England showed significant inhabitation of respiration rate of the biomass from sewerage and these were all basic dyes (Cooper, 1995).

Considering only the general structure, textile dyes are classified in anionic, non-ionic and cationic dyes. The major anionic dyes are direct, acid and reactive dyes (Robinson et al., 2001) and the most problematic ones are the brightly coloured, water soluble reactive and acid dyes since they cannot be removed through conventional treatment systems (Zaharia and Suteu, 2012). The major non-ionic dyes are disperse dyes that does not ionised in the aqueous environmental and major cationic dyes are the azo basic, anthraquinone disperse and reactive dyes, etc.

The most problematic dyes are those which are made from known carcinogens such as benzidine and other aromatic compounds (i.e. anthraquinone-based dyes are resistant to degradation due to their fused aromatic ring structure). Some disperse dyes have good ability to bioaccumulation and the azo and nitro compounds are reduced in sediments, other dyes-accumulating substrates to toxic amines (e.g. $R_1\text{-N} = \text{N-}R_2 + 4H^+ + 4e^- \rightarrow R_1\text{-NH}_2 + R_2\text{-NH}_2$). The organic dyes used in the textile dyeing process must have a high chemical and photolytic stability and the conventional textile effluent treatment in aerobic conditions does not degrade these textile dyes and are presented in high quantities into the natural water resources in absence of some tertiary treatments (Zaharia and Suteu, 2012).

2.3 Methods of Dye Removal

Since the introduction of dyes, not much environmental concern has been taken into consideration until this few years. Environmentalist suggest that textile dyes must be separated and eliminated if necessary from water but especially from industrial wastewater by effective and viable treatment at sewerage treatment works or on site following two different treatment concepts as: (1) separation of organic pollutants from water environment or (2) the partial or complete mineralization or decomposition of organic pollutant.

Separation process is based on fluid mechanics (sedimentation, centrifugation, filtration and floatation) or on synthetic membranes (micro-, ultra- and nanofiltration, reverse osmosis). Additionally, physicochemical processes (i.e. adsorption, chemical precipitation, coagulation-flocculation and ionic exchange) can be used to separate dissolve, emulsified and solid-separating compound from water environment (Anjaneyulu et al., 2005; Babu et al., 2007; Robinson et al., 2001; Suteu et al., 2009a; Suteu et al., 2011a; Zaharia, 2006; Zaharia et al., 2009; Zaharia et al., 2011; Zaharia and Suteu, 2012).

The partial and complete mineralization or decomposition of pollutants can be achieved by biological and chemical processes. **Table 2.1** show the dye waste treatment methodologies and their advantages as well as their limitations.

Table 2.1: *Dye waste treatment methodologies and their advantages and limitations. (Anjaneyulu et al., 2005; Babu et al., 2007 et al., 2001; Zaharia and Suteu, 2012)*

Treatment methodology	**Treatment stage**	**Advantages**	**Limitations**
Physicochemical treatment			
Precipitation, coagulation-flocculation	Pre/main treatment	Short detention time and low capital costs. Relatively good removal efficiencies.	Agglomerates separation and treatment. Selected operating condition.
Electro kinetic coagulation	Pre/main treatment	Economically feasible	High sludge production
Fenton process	Pre/main treatment	Effective for both soluble and insoluble coloured contaminants. No alternation in volume.	Sludge generation; problem with sludge disposal. Prohibitively expensive.
Ozonation	Main treatment	Effective for azo dye removal. Applied in gaseous state: no alteration of volume	Not suitable for dispersed dyes. Releases aromatic dyes. Short half-life of ozone (20 min)
Oxidation with NaOCl	Post treatment	Low temperature requirement. Initiates and accelerates azo- bond cleavage	Cost intensive process. Release of aromatic amines
Adsorption with solid adsorbent			
Activated carbon	Pre/post treatment	Economically attractive. Good removal efficiency of wide variety of dyes.	Very expensive; cost intensive regeneration process
Peat	Pre treatment	Effective adsorbent due to cellular structure. No activation required.	Surface area is lower than activated carbon
Coal ashes	Pre treatment	Economically attractive. Good removal efficiency.	Larger contact times and huge quantities are required. Specific surface area for adsorption are lower than activated carbon

Treatment methodology	**Treatment stage**	**Advantages**	**Limitations**
Wood chips/ Wood sawdust	Pre treatment	Effective adsorbent due to cellular structure. Economically attractive. Good adsorption capacity for acid dyes	Long retention times and huge quantities are required.
Silica gels	Pre treatment	Effective for basic dyes	Side reactions prevent commercial application
Irradiation	Post treatment	Effective oxidation at lab scale	Requires a lot of dissolved oxygen (O2)
Photochemical process	Post treatment	No sludge production	Formation of by-products
Electrochemical oxidation	Pre treatment	No additional chemicals required and the end products are non-dangerous/hazardous.	Cost intensive process; mainly high cost of electricity
Ion exchange	Main treatment	Regeneration with low loss of adsorbents	Specific application; not effective for all dyes
Biological treatments			
Aerobic process	Post treatment	Partial or complete decolourization for all classes of dyes	Expensive treatment
Anaerobic process	Main treatment	Resistant to wide variety of complex coloured compounds. Bio gas produced is used for stream generation.	Longer acclimatization phase
Single cell (Fungal, algal & bacterial)	Post treatment	Good removal efficiency for low volumes and concentrations. Very effective for specific colour removal.	Culture maintenance is cost intensive. Cannot cope up with large volumes of WW.
Emerging treatments			
Other advanced oxidation process	Main treatment	Complete mineralization ensured. Growing number of commercial applications. Effective pre-treatment methodology in integrated systems and enhances biodegradability.	Cost intensive process

Treatment methodology	**Treatment stage**	**Advantages**	**Limitations**
Membrane filtration	Main treatment	Removes all dye types; recovery and reuse of chemicals and water.	High running cost. Concentrated sludge production. Dissolved solids are not separated in this process
Photocatalysis	Post treatment	Process carried out at ambient conditions. Inputs are no toxic and inexpensive. Complete mineralization with shorter detention times.	Effective for small amount of coloured compounds. Expensive process.
Sonication	Pre treatment	Simplicity in use. Very effective in integrated systems.	Relatively new method and awaiting full scale application.
Enzymatic treatment	Post treatment	Effective for specifically selected compounds. Unaffected by shock loadings and shorter contact times required.	Enzyme isolation and purification is tedious. Efficiency curtailed due to the presence of interferences.
Redox mediators	Pre/ supportive treatment	Easily available and enhances the process by increasing electron transfer efficiency	Concentration of redox mediator may give antagonistic effect. Also depends on biological activity of the system.
Engineered wetland systems	Pre/post treatment	Cost effective technology and can be operated with huge volumes of wastewater	High initial installation cost. Requires expertise and managing during monsoon becomes difficult

2.4 Activated Carbon

According to Jassim et al. (2012), activated carbon is a microcrystalline form of carbon with very high porosity and surface area. Activated carbon has the highest volume adsorbing porosity and the strongest physical adsorption forces. The surface area of activated carbon can be greater than 1000 m^2/g. Activated carbons have become one of the most effective adsorbent due to its chemical structure that allows preferential adsorption of toxic substances like metal ions, organic compounds and dyes (Saha et al., 2003; Tseng et al., 2003; Ozdemir et al., 2011).

Activated carbon is usually made from carbonaceous materials such as nutshells, coconut shells, coals, woods and peat. The characteristics and performance of the activated carbon highly depends on the raw material used. There are three main forms of activated carbons which are Granular Activated Carbon (GAC), Powder Activated Carbon (PAC) and Extruded Activated Carbon (EAC). GAC is in irregular shape with size ranging from 0.2 to 5 mm and suitable for liquid and gas phase application. PAC is pulverised carbon with size less than 0.18 mm. PAC is commonly used in liquid phase application and for flue gas treatment. EAC is extruded and cylindrical shaped with diameter from 0.18 to 5 mm. EAC is mainly being used for gas phase application because of their low pressure drop, high mechanical strength and low dust content.

Activated carbon is commonly used in air treatment, drinking water treatment, wastewater treatment, industrial process as well as medication. The cost for initial and regeneration for commercial activated carbon is considered high (Chakraborty et al., 2005). Therefore, the research now focused of research to find cheap substitutes which are inexpensive as well as endowed with reasonable adsorption capacities (Ahmad et. Al., 2007). The studies include the utilization of agricultural waste such as rice husk, palm-fruit bunch, walnut shells and orange peels.

2.5 Low-cost Adsorbent

Sharma et al. (2010) in their review paper reveals the compilation list of low-cost adsorbents made up from various types of materials. These adsorbent have been classified into five different categories on basis of their availability:

a. Agriculture and industry waste
b. Fruit waste
c. Plant waste
d. Natural inorganic materials
e. Bioadsorbents

Rice is one of the major crops grown throughout the world, sharing equal importance with wheat as principal staple food and a provider of nourishment for the world’s population. Covering 1% of the earth’s surface, rice is being grown on every continent and deeply embedded in cultures, rituals and myths (Bronzeoak, 2003). In Asia alone, rice constitutes as much as 60-70% of the total calorie uptake on average for more than 2000 million people (Foo and Hameed, 2009).

Concomitant with the rigorous development of rice milling industries, rice husks an abundantly available by product. Rice husk is an agricultural waste, contains about 20% of silica and has been reported as a good adsorbent of many metals and basic dyes. According to the statistics compiled by the Malaysian Ministry of Agriculture, there are about 408000 tonnes of rice husk produce in Malaysia annually (Wong et al., 2003; Chuah et al., 2005).

The chemical components of rice husks are found to be SiO_2, H_2O_2, Al_2O_3, Fe_2O_3, Na_2O, CaO and MgO (Feng et al., 2004), fluctuating with the varieties of paddy sown, proportion of irrigated area, geographical conditions, fertilizer used, climatic variation, soil chemistry, timeliness crop production operation and agronomic practices in the paddy growth process. (Foo and Hameed, 2009). Traditionally, the rice hull or rice husks have been disposed in landfills thereby resulting aesthetic pollution and disturbances in the aquatic life (Mane et al., 2007). **Table 2.2** and **Table 2.3** shows reported physicochemical characteristic and typical compositions of rice husk respectively.

Table 2.2: *Reported physicochemical characteristic of rice husk by (Chuah et al., 2005; Malik, 2003)*

Characteristic	**Value**
Bulk density (g/ml)	0.73
Solid density (g/ml)	1.5
Moisture content (%)	6.62
Ash content (%)	45.97
Particle size (mesh)	200-16
Surface area (m^2/g)	272.5
Surface acidity (meq/gm)	0.1
Surface basicity (meq/gm)	0.45

Table 2.3: *Typical compositions of rice husk (Chuah et al., 2005; Rahman et al., 1997; Rahman et al., 1993; Damel, 1976)*

Composition	**Percentage**
Cellulose	32.24
Hemicelluloses	21.34
Lignin	21.44
Extractives	1.82
Water	8.11
Mineral Ash	15.05
Chemical composition in mineral ash:	
SiO_2	96.34
K_2O	2.31
MgO	0.45
Fe_2O_3	0.2
Al_2O_3	0.41
CaO	0.41
K_2O	0.08

Rice husk can be made into absorbent for use in water purification or industrial wastewater treatment. It would add value to these agricultural commodities by reducing the cost of waste disposal as well as providing a potentially cheap alternative to existing commercial activated carbons (Chuah et al., 2005).

Table 2.5 shows the reported adsorption capacity of other low-cost adsorbent in dye removal process. Additionally **Table 2.4** shows the reported adsorption capacity of rice husk in dye removal process.

Table 2.4: *Reported adsorption capacity of other low-cost adsorbent in dye removal process*

Adsorbent	**Dye**	**Adsorption Capacity**	**References**
Palm ash	Direct blue 71	400.01 mg/g	Ahmad et al. (2007)
Banana pith	Acid brilliant blue	4.3 mg/g	Namasivayam et al. (1998)
Coir pith	Acid violet	1.6 mg/g	Namasivayam et al. (2001)
	Acid brilliant blue	16.6 mg/g	
	Rhodamine B	203.2 mg/g	
Peat	Acid blue 29	13.95 mg/g	Ramakrishna and Viraraghavan (1997)
Sugarcane bagasse	Methylene blue	99.60 mg/g	Raghuvanshi et al. (2004)
Orange peel	Direct blue 86	33.78 mg/g	Nerm et al. (2008)

Table 2.5: *Reported adsorption capacity of rice husk in dye removal process*

Dye	**Adsorption Capacity**	**References**
Indigo carmine	65.90 mg/g	Lakshmi et al. (2009)
Acid yellow 36	86.90 mg/g	Malik (2003)
α-picoline	15.46 mg/g	Lataye et al. (2006)
Cargo red	14.00 mg/g	Han et al. ((2008)
Safranine	178.10 mg/g	Kumar and Sivanesan (2007)
Brilliant green	26.20 mg/g	Mane et al. (2007a)

2.6 Adsorption Isotherm

The relationship between the amounts of a substance absorbed at constant temperature and its concentration in the equilibrium solution is called the absorption isotherm. The equilibrium absorption density, q_e increase with the increase in dye concentration (Ahmad et al., 2007). Waber (1972) stated that 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. Several models have been published in the literature to describe experimental data of absorption isotherms. The Langmuir and Freundlich are the most frequently employed models.

2.6.1 Langmuir Isotherm

The preferred form of depicting this distribution is to express the quantity, q_e as a function of C_e at fixed temperature, the quantity; q_e being the amount of solute absorbed per unit weight of solid adsorbent and C_e is the concentration of the solute remaining in solution at equilibrium.

The following equation can represents the Langmuir isotherm model:

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

The linear forms of the Langmuir equation:

$$\frac{C_e}{q_e} = \frac{1}{bQ_0} + \frac{C_e}{Q_0} \qquad (2)$$

or,

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

where, q_e is the amount adsorbed at equilibrium (mg/g), C_e the equilibrium concentration of the adsorbent (mg/L), and Q_0 (mg/g) and b (L/mg) are the Langmuir constants related to the maximum adsorption capacity and the energy of adsorption, respectively. Either of these forms can be used depending on range and spread of data and on the particular data to be emphasized.

Although the basic assumptions explicit in the development of the Langmuir Isotherm are not met the most adsorption system concerning water and wastewater treatment, the Langmuir Isotherm has been found particularly useful for description of equilibrium data for such system providing parameters (Q_0 and b) with which to quantitatively compare adsorption behaviour.

2.6.2 Freundlich Isotherm

Freundlich or van Bemmelen equation has been useful for special case that heterogeneous surface energies in which the energy term, b in the Langmuir Isotherm (equation 1) varies ad function of surface coverage, q_e due to variations in heat adsorption.

General form of the Freundlich isotherm:

$$\boldsymbol{q_e = K_f C_e^{\frac{1}{n}}} \qquad (\mathbf{4})$$

Linear form of the Freundlich equation:

$$\boldsymbol{log q_e = log K_f + \frac{1}{n} log C_e} \qquad (\mathbf{5})$$

where, K_f (mg/g) $(L/mg)^{1/n}$ and $1/n$ are Freundlich constants related to adsorption capacity and adsorption intensity of the absorbent, respectively which give 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 Freundlich equation generally agrees quite well with the Langmuir equation but does not reduce to liner adsorption expression at very low concentration nor does it agree well at high temperature, since n must reach some limit when the surface is fully covered.

2.7 Adsorption Kinetics

A simple pseudo first-order equation is commonly used to explain the mechanism of the adsorption process.

$$\frac{d_{qt}}{d_t} = k_1(q_e - q_t) \tag{6}$$

where, q_e and q_t are the amount of adsorption at equilibrium and at time t, respectively and k_1 is the rate constant of the pseudo first-order adsorption process. The integrated rate law after application of the initial condition $q_t = 0$ at $t = 0$:

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \tag{7}$$

The graph of $\log (q_e - q_t)$ versus t gives a straight line for first-order adsorption kinetics, which allows computation of the adsorption rate constant, k_1. This equation differs from a true first-order equation in two ways:

i. The parameters $k_1 (q_e - q_t)$ does not represent the number of available sites.
ii. The parameters $\log (q_e)$ is an adjustable parameter and often it is founded to be not equal to intercept of the plot of $\log (q_e - q_t)$ versus t, whereas in true first order $\log (q_e)$ should be equal to the intercept.

In such cases, applicability of the pseudo second-order kinetics has to be tested with the rate of equation (Sharma and Bhattacharya, 2004; Oztuk and Kavak, 2005):

$$\frac{d_{qt}}{d_t} = k_2(q_e - q_t) \tag{8}$$

where, k_2 is the second order rate constant in g/(mg)(min)

From the boundary conditions, $t = 0$ to $t = 1$ and $q_t = 0$ to $q_t = q_t$, the integrated form of the equation become:

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

This can be written in linear form;

$$\boldsymbol{\frac{t}{q_t} = \frac{1}{h} + \left(\frac{1}{q_t}\right) t} \qquad \textbf{(10)}$$

where, $h = k_2 q_e^2$ can be regarded as initial sorption rate as $t \rightarrow 0$. Under such circumstances, the plot of t/q_t versus t should give a linear relationship which allows computation of q_e, k and h.

CHAPTER 3

MATERIALS AND METHODS

3.1 Materials

Figure 3.1 below shows the molecular structure of Direct Blue 86 and **Table 3.1** shows the chemical properties of Direct Blue 86 (DB 86). The dye was readily available in Environmental laboratory. The raw rice husk was obtained from a local rice processing factory in Malaysia.

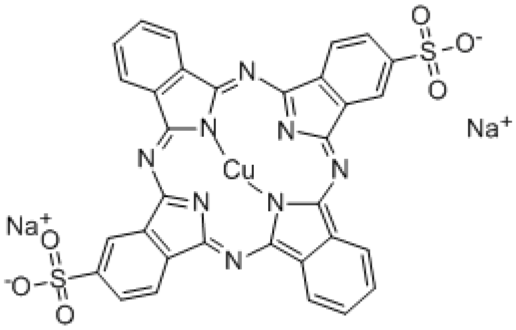


Figure 3.1: *Molecular structure of Direct Blue 86*

Table 3.1: *Molecular properties of Direct Blue 86*

Molecular formula	:	$C_{32}H_{14}CuN_8Na_2O_6S_2$
Molecular weight	:	780.16
Colour index	:	74180
Stability	:	Stable. Incompatible with strong oxidizing agents
Synonyms	:	dihydrogenphthalocyaninedisulfonato(2-))-coppedisodiumsalt [29h,31h-phthalocyanine-c,c-disulfonato(4-)-n29,n39,n31,n32]-cuprate(2-dis; abcolturquoiseblue; acidblue87; aizenprimulaturquoisebluegl; amafastturquoise8ggl; amanilfastturquoise; amanilfastturquoiselb

Source:

http://www.chemicalbook.com/ProductChemicalPropertiesCB8336918_EN.htm

3.2 Preparation of Direct Blue 86 Standard Curve

Dye concentration was determined by finding the wavelength of maximum absorbance for dye in solution of 50 mg/L. A stock solution of 1000 mg/L was prepared by adding 1.000 g of DB 86 into 1 L of distilled water. From the stock solution, 5 ml was diluted into 100 ml in a volumetric flask to obtain 50 mg/L of dye solution. Then, by using spectrophotometer and varying the wavelength, the wavelength with the highest value of dye absorbance was determined. By using the same wavelength another test was conducted by varying the dye concentration to plot the standard curve and the results obtained must plot a straight line with $R^2>0.99$ so that it can be referred for next stage of study. Standard curve at pH 1 to pH 10 was prepared to overcome the colour changes during pH adjustment.

3.3 Preparation of Rice Husk-Based Adsorbents

Rice husk-based (RH) adsorbents were prepared by two types of preparation which are chemical and thermal treatment.

3.3.1 Chemical treatment

3.3.1.1 Sulphuric acid

Rice husk was washed with a stream of distilled water through a sieve of 16 mesh (Tyler Standard Screen Scale) to remove dirt, dust and any superficial impurities. The rice husk was put in trays and left to dry in open air at room temperature to constant weight. The absorbent was prepared by using the clean air-dried rice husk (20 g) was weighed in a clean dry beaker of capacity 500 ml. One hundred millilitres of 13 M sulphuric acid was added to the rice husk and the mixture was heated to 175–180°C in 20 minutes with occasional stirring. The temperature was kept in this range (175–180°C) for 20 minutes. The resulting black mixture was allowed to cool, and then filtered using a Buchner funnel under vacuum. The black spent sulphuric acid (black liquor) was filtered off and the carbonized material was washed several times with distilled water before being used (El-Shafey, 2007). Acid treated rice husk labelled as RH1.

3.3.1.2 Sodium hydroxide

The rice husk was washed thoroughly with distilled water. It was dried at 105°C to remove moisture and then was grounded to pass through a 1-mm sieve. The rice husk was treated with 0.5 M NaOH solution at room temperature for 4 h. Excess of NaOH will be removed with water and the material was dried at room temperature (Low et al., 2000). The alkali treated adsorbent labelled as RH2.

3.3.2 Thermal treatment

The rice husk was washed thoroughly with tap water and then rinsed 2-3 times with distilled water. It was dried at 105°C to remove moisture. The rice husk was burnt in a muffle furnace at 300°C for 1 hour (RH3), at 300°C for 4 hours (RH4), at 400°C for 4 hours (RH5) and at 600°C for 4 hours (RH6). According to Daffala et al. (2010), at 300°C considerable amount of carbon would be produced, however at 400°C the amount of carbon decrease which caused an increase in the amount of silica. On the other hand, at 600°C high amorphous silica would be produced (Nair et al., 2006).

3.4 Batch Adsorption Test

3.4.1 Batch Adsorption Screening Test

Batch adsorption screening test with six rice husk-based (RH) absorbents and a commercial powder activated carbon (PAC) was conducted at standard room temperature (22°C) by using a fixed dosage (2 g/L) of adsorbents and contact time of 24 hours. The dye solution was prepared at the concentration of 20 mg/L and pH varied from 1 to 10. The desired pH was obtained by HCl and NaOH solution (Isa et al., 2008). Based on the result, the best adsorbent and pH was selected.

3.4.2 Effect of Contact Time and Dye Concentration

The experiment was conducted with the selected adsorbent at a fixed pH (optimum pH) of dye solution and contact time of 0, 10, 20, 30, 45, 60, 90, 120, 180 and 240 minutes while the concentration of the dye is varied from 20 mg/L to 80 mg/L. The RH and PAC adsorbent dosage will be fixed at 2 g/L. The optimum contact time was determined by the plot of time versus adsorption.

3.4.3 Effect of Adsorbent Dosage

The experiment was conducted by using a fix dye concentration. The adsorbent dosage added into the 50 mL of DB 86 dye solution was varied from 0.2 g to 1.2 g. The pH of all the solution was maintained base on the optimum pH obtained from the previous experiment. The contact time also made as constant based on the optimum contact time.

3.5 Equilibrium Adsorption Test

Equilibrium adsorption test was conducted under optimum pH and contact time by adding constant dosage of RH and PAC adsorbent into different concentration for dye (20 to 160 mg/L). The dye solution after equilibrium adsorption will be measured by the standard curve. From the test results, the adsorbent isotherm and adsorption capacity will be evaluated.

3.6 Kinetic Study

Kinetic study was conducted using the same procedure as effect of contact time and dye concentration. The similar data obtained were used to analyze the kinetic mechanism of adsorption process.

3.7 Study Plan

Table 3.2 and **Table 3.3** below show the study plan for the project.

Table 3.2: *Study Plan for FYP I*

Detail/week	**1**	**2**	**3**	**4**	**5**	**6**	**7**		**8**	**9**	**10**	**11**	**12**	**13**	**14**
Selection of project topic and FYP briefing	🟦							**Mid-Sem Break**							
Literature review		🟦	🟦	🟦	🟦	🟦									
Preparation of Extended Proposal				🟦	🟦	🟦	🟦								
Submission of extended proposal							🟨								
Proposal defence										🟨					
Preparation of Interim Report											🟦	🟦	🟦	🟦	
Project work continues: preparation of absorbent and preliminary experiment									🟦	🟦	🟦	🟦	🟦	🟦	🟦
Submission of Interim Draft Report														🟨	
Submission of Interim Report															🟨

🟦 Project activity (progress) 🟨 Key Milestone

Table 3.3: *Study Plan for FYP II*

Detail/week	**1**	**2**	**3**	**4**	**5**	**6**	**7**		**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
Project work continues and preparing Progress Report	🟩	🟩	🟩	🟩	🟩	🟩	🟩	**Mid-Sem Break**								
Submission of Progress Report									🟥							
Preparing Dissertation Report						🟩	🟩		🟩	🟩	🟩	🟩	🟩			
Project work continues									🟩	🟩	🟩	🟩	🟩			
Pre-SEDEX												🟥				
Submission of draft Dissertation Report													🟥			
Submission of Dissertation Report (soft bound)														🟥		
Submission of Technical Paper														🟥		
Oral Presentation															🟥	
Submission of Dissertation Report (hard bound)																🟥

🟩 Project activity (progress) 🟥 Key Mileston

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Optimum Wavelength (λ) for Direct Blue 86 Absorption.

Figure 4.1 shows the optimum wavelength (λ) for DB 86 solution for unadjusted pH which is 9.83 for solution 1000 mg/L of DB 86. The wavelength of maximum absorbance is 620 *n*m. This result was obtained by manipulating the wavelength value of spectrophotometer with a constant concentration of DB 86.

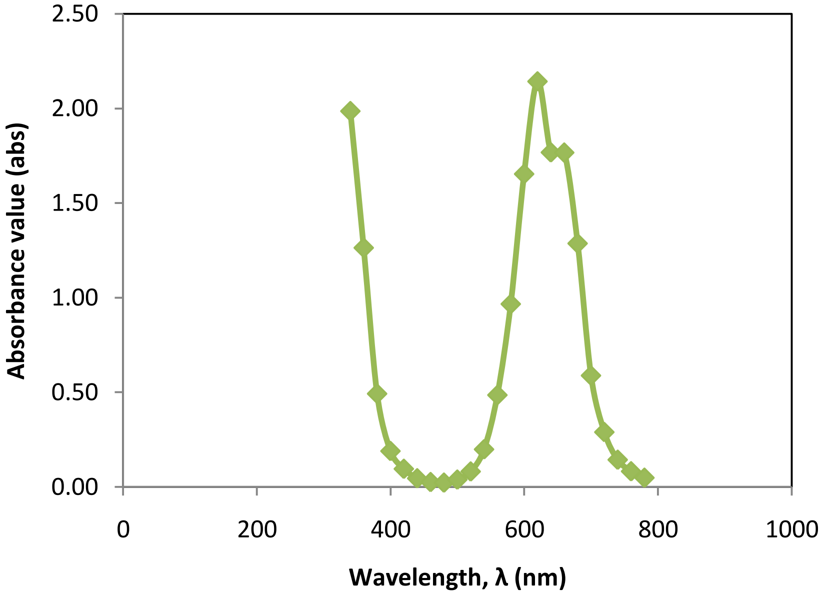


Figure 4.1: *The optimum wavelength (λ) for DB 86 absorption.*

4.2 Standard Curve for Direct Blue 86

Figure 4.2 shows the standard curve for DB 86 at λ = 620 *n*m at unadjusted pH, 9.83 at 1000 mg/L of dye solution. This standard curve was plotted by manipulating the concentration of DB 86 to obtain their absorbance value at constant λ. The purpose of the standard curve is to obtain the concentration of the unabsorbed dye in an adsorption test. The value displayed by spectrophotometer in absorbance (abs) and by using that value, referring to standard curve plotted, the concentration of unabsorbed dye will be known. Then, the adsorption percentage can be evaluated.

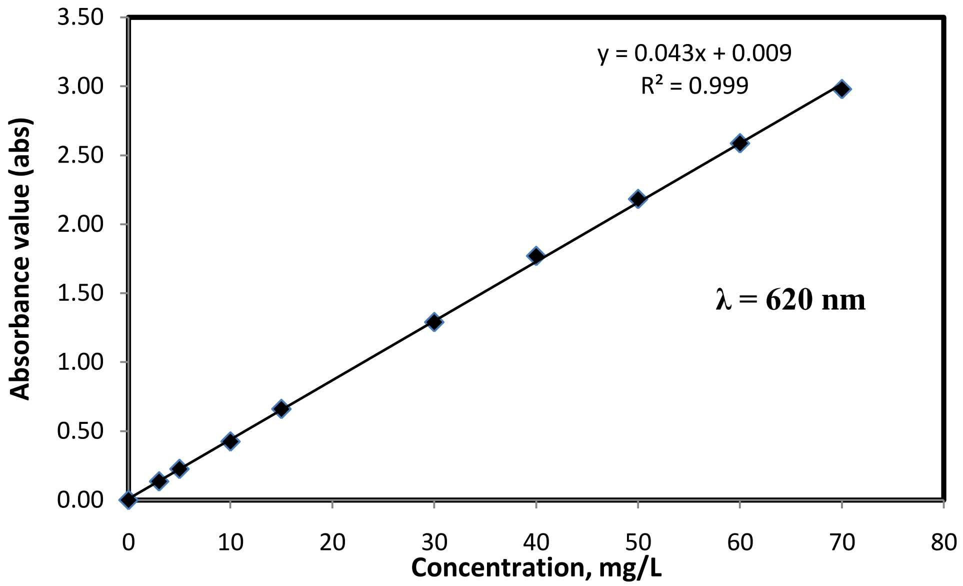


Figure 4.2: *Standard curve of DB 86*

4.2.1 Standard Curve for Every pH

The colour concentration of dye and absorbance changed due to pH adjustment during experiment by adding HCl and NaOH. **Figure 4.3** shows the standard curves plotted at different pH. This standard curve was used to determine the concentration of unadsorbed dye in adsorption test at different pH. **Table 4.1** indicate the details for each standard curve model. The value of R^2 for each pH more than 0.99 indicating the standard curve is suitable to determine the concentration of dye based on the absorbance value. For individual plot see **Appendix A**.

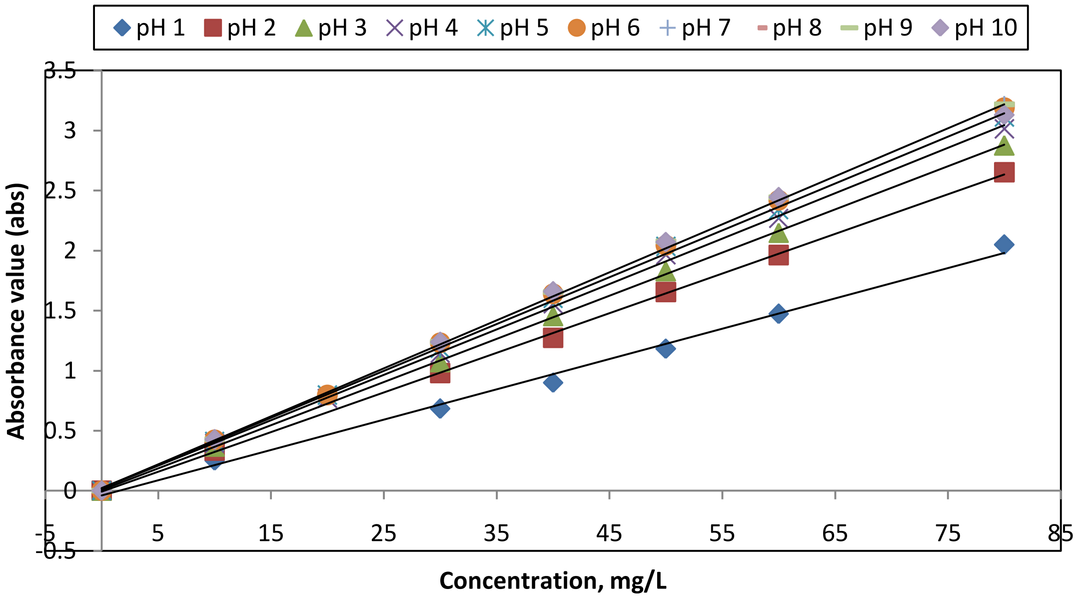


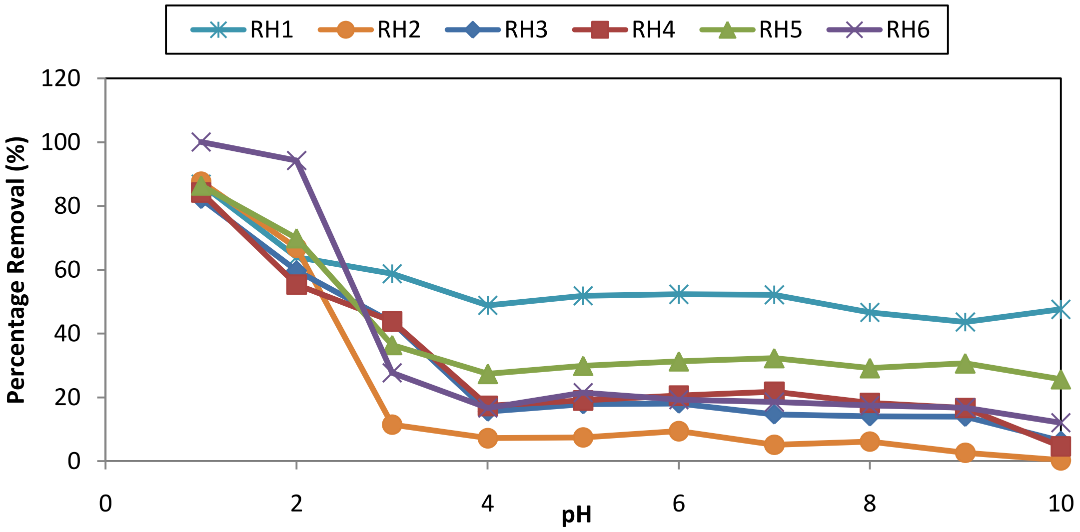
Figure 4.3: *Standard curves of DB 86 at different pH*

Table 4.1: *The detail of standard curve*

pH	Linear equation	R^2
1	y = 0.025x – 0.039	0.994
2	y = 0.033x – 0.007	0.999
3	y = 0.036x + 0.005	0.999
4	y = 0.037x + 0.019	0.999
5	y = 0.039x + 0.020	0.999
6	y = 0.040x + 0.018	0.999
7	y = 0.040x + 0.024	0.999
8	y = 0.040x + 0.023	0.999
9	y = 0.040x + 0.024	0.999
10	y = 0.039x + 0.041	0.998

4.3 Batch Adsorption Screening Test

The effect of pH and adsorbent type were investigated by batch adsorption screening test for RH adsorbent. **Figure 4.4** shows the best adsorbent for dye removal was RH6 which is RH adsorbent burnt at 600°C for 4 hours. The optimum pH for dye removal was found at pH 2. Nemr et al (2009) reported that optimum adsorption of DB 86 using orange peel was observed at pH 2 which indicated similar observation to this test. Lower pH value tends to increase the H^+ ions concentration in the system and strong electrostatic attraction appears between the positively charge adsorbent surface and anionic dye molecule lead to maximum adsorption of DB 86. On the other hand, electrostatic repulsion happens at higher pH since the increase of negative charges and decreases the positive charges. The potential for each adsorbent rapidly decrease at pH > 3.



. ***Figure 4.4:*** *Effect of pH (RH adsorbent)*

The decreasing in adsorption of DB 86 was due to competition anionic dye and OH^- in the solution. Acid treated adsorbent give the highest percentage removal of Direct Blue after pH 3. This is due to acidic nature of the adsorbent; provide extra H^+ to the system. Likewise for alkali treated adsorbent give the lowest percentage removal due to adsorbent's nature. Moreover, the dye concentration also increases resulting negative value in percentage removal.

Meanwhile, a PAC was used to compare with the rice husk-based adsorbents. **Figure 4.5** below shows the effect of pH for PAC. The optimum pH for dye removal also observed at pH 2.

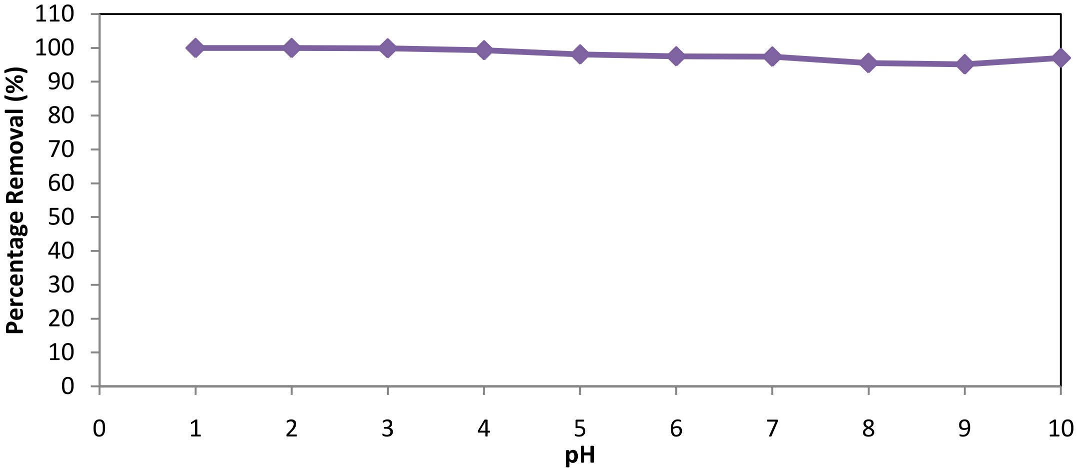


Figure 4.5: *Effect of pH (Commercial PAC)*

4.4 Effect of Contact Time and Dye Concentration

The result for effect of time and dye concentration of adsorption for DB 86 by the best RH adsorbent (RH6) and PAC were shown in **Figure 4.6** and **Figure 4.7** respectively. This test was conducted at optimum pH which is pH 2 while varying the contact time (3, 5, 10, 20, 30, 45, 60, 90, 120, 180, 240) minutes and different initial concentration of dye (20, 40, 60, 80) mg/L. More than 45% removal of DB 86 concentration occurred in the first 5 minutes of the contact time and the rate of adsorption slowly increase until reach equilibrium. The equilibrium state found to be nearly 180 minutes of contact time when the maximum adsorption onto adsorbent was reached. The rapid adsorption is due to the availability of positively charged surface of rice husk-based adsorbent for adsorption of anionic Direct Blue 86 in the solution at pH 2 (Nemr et al., 2009).

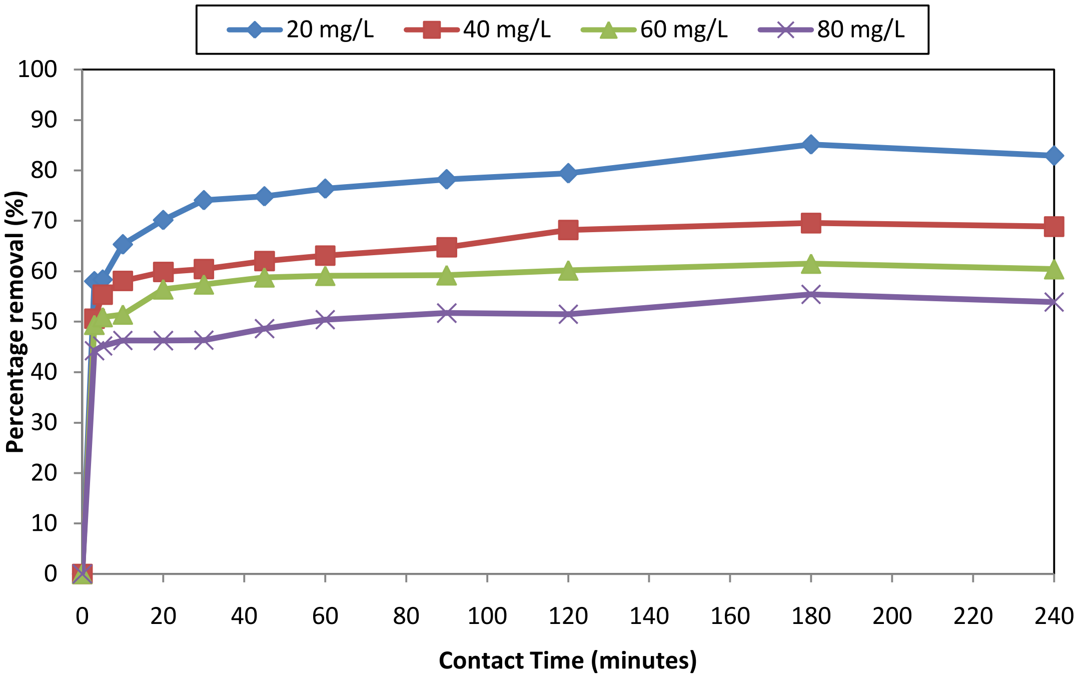


Figure 4.6: *Effect of contact time and dye concentration (RH6)*

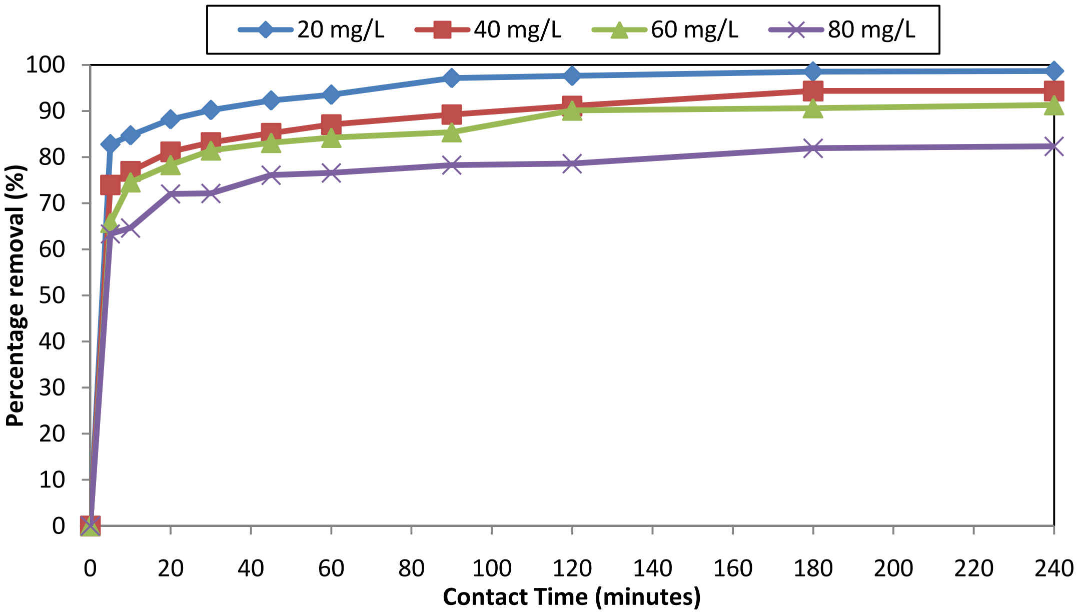


Figure 4.7: *Effect of contact time and dye concentration (PAC)*

Figure 4.6 and **Figure 4.7** also showed that the percentage removal of dye decreased with increasing initial concentration of dye because the porous structure of the adsorbent fully occupied by dye molecules. Furthermore according to Nemr et. Al. (2009), initially dye molecules have to encounter the boundary layer effect and diffused from boundary layer film onto adsorbent surface and finally diffuse into porous structure of the adsorbent and the phenomenon will take longer contact time.

4.5 Effect of Adsorbent Dosage

This study was done by using different dosage of RH6 and PAC into optimum pH 2, optimum contact time (180 minutes) and using constant dye concentration (20 mg/L). The dose varied from 2 g/L to 18 g/L. **Figure 4.8** shows the effective dosage is 4 g/L of RH6 to completely remove the dye. In the other hand, **Figure 4.9** shows the optimum PAC in DB 86 removal. The result shows that only 3 g/L of PAC needed to completely remove DB 86. The quantity of adsorbent dosage was indirectly increasing the porous surface area of the system. Therefore, the result shows that RH6 has less porous surface area than PAC.

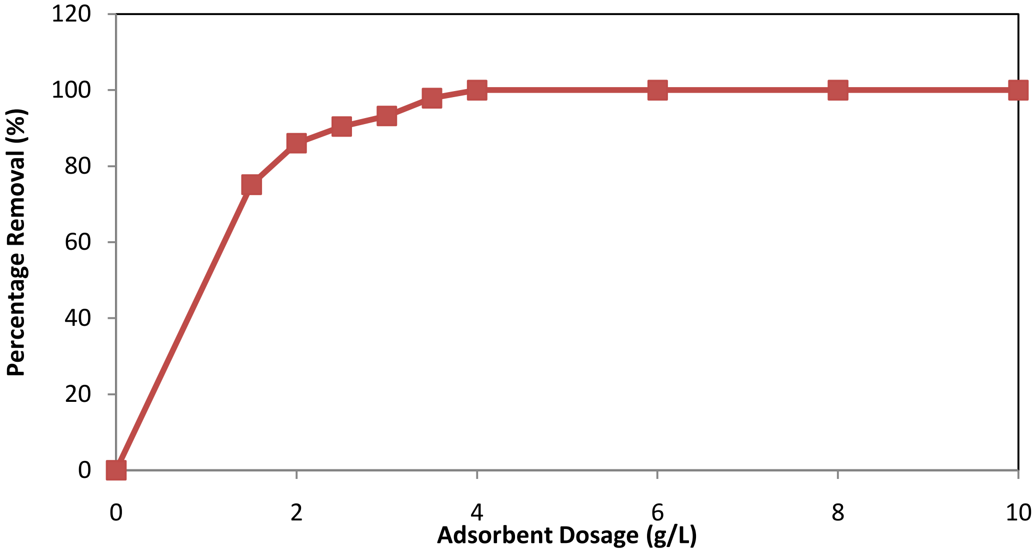


Figure 4.8: *Effect of Adsorbent Dosage (RH6)*

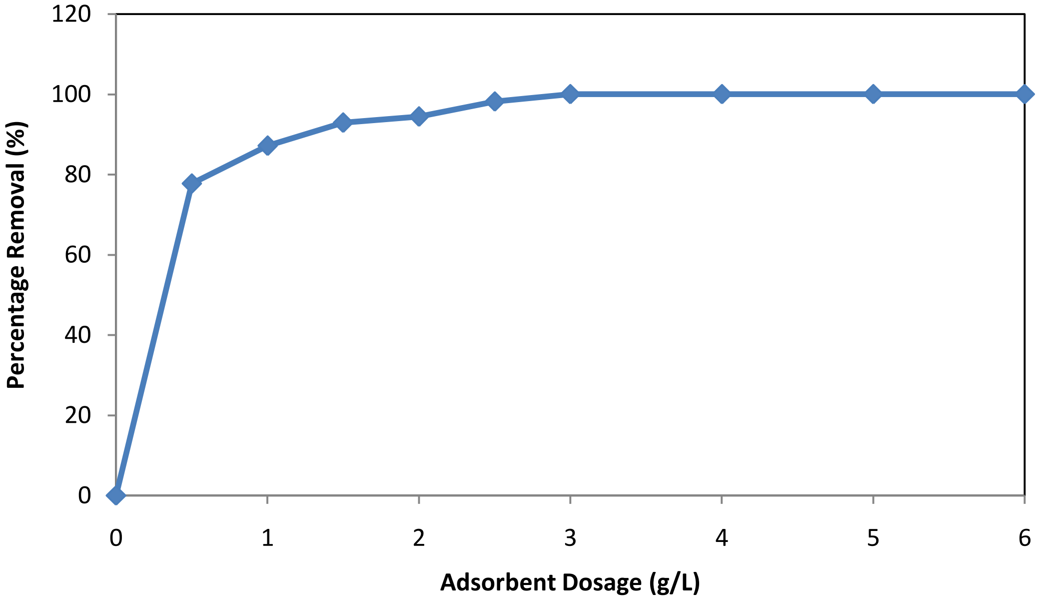


Figure 4.9: *Effect of Adsorbent Dosage (PAC)*

4.6 Equilibrium Adsorption Test

In equilibrium adsorption study, Langmuir and Freundlich isotherms were employed based on equation **(2)** and **(5)**. **Figure 4.10** shows linear plot of Langmuir isotherm while **Figure 4.11** shows the Freundlich isotherm linear plot. Both Langmuir and Freundlich constants for RH6 and PAC obtained graphically from the figures. Their values are shown in **Table 4.2**.

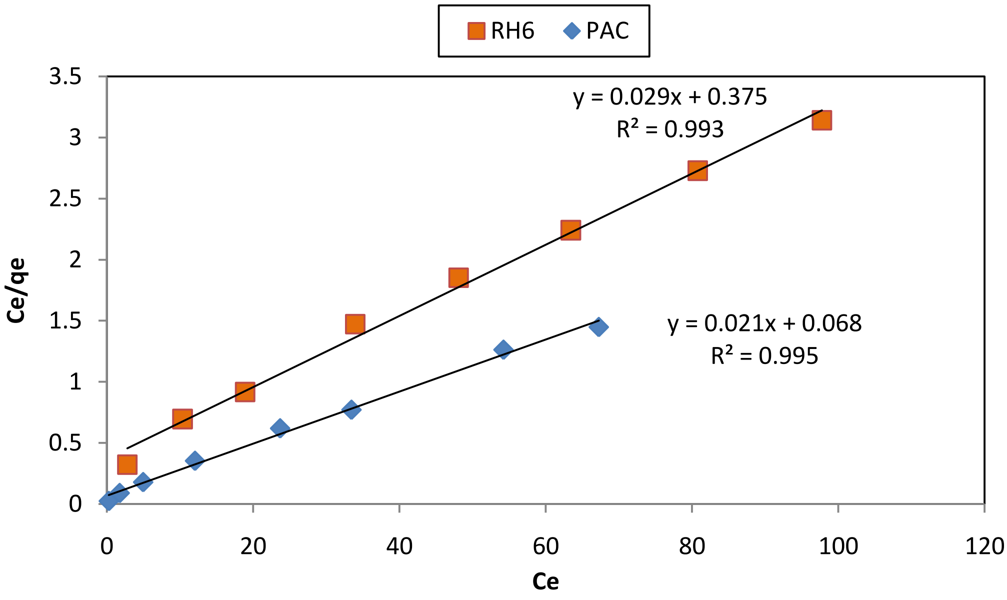


Figure 4.10: *Langmuir isotherm for RH6 and PAC*

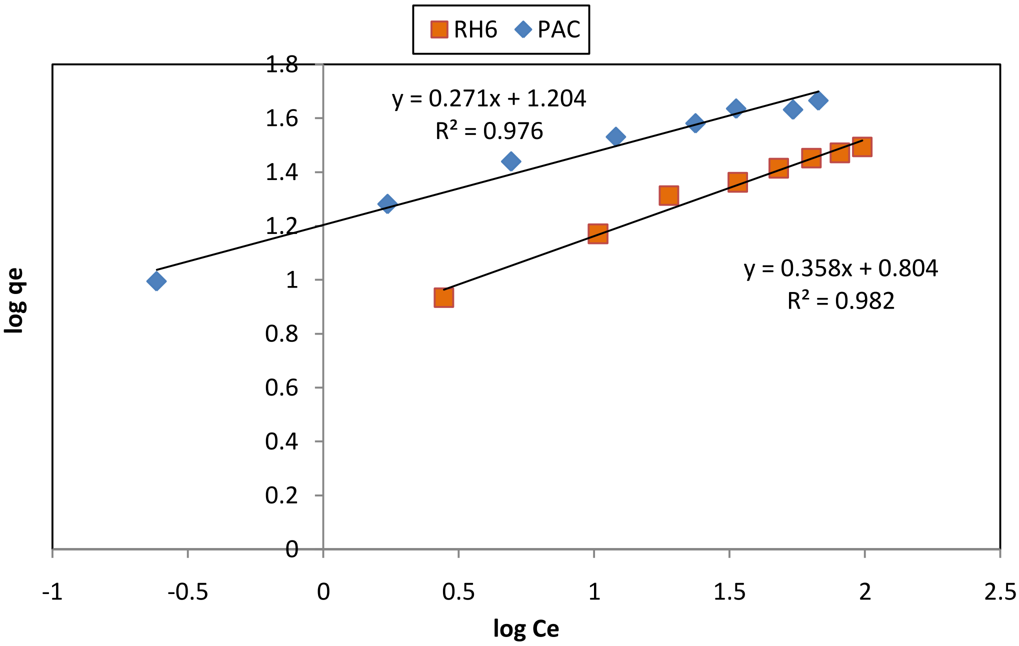


Figure 4.11: *Freundlich isotherm for RH6 and PAC*

Table 4.2: *Isotherm constants and correlation coefficients*

Adsorbent	Langmuir isotherm coefficients			Freundlich isotherm coefficients		
	Q_o	b	R^2	K_f	1/n	R^2
RH6	34.4828	0.07733	0.993	6.3680	0.358	0.982
PAC	47.6190	0.30882	0.995	15.9956	0.271	0.976

The high correlative coefficient, (R^2>0.99) show strong evidence that adsorption of DB 86 onto RH and PAC follow the Langmuir model. Nevertheless, the R^2 value for Freundlich model is comparable to Langmuir model for both RH6 and PAC. Langmuir model assumes monolayer adsorption and uniform adsorption energies independent of surface coverage, meanwhile the Freundlich model encompasses the heterogeneity of the adsorbent surface, exponential distribution sites and their energies (Isa et al., 2008). The 1/n value for RH6 and PAC are 0.358 and 0.271 respectively. Both values are favourably smaller than 1 which indicating good adsorption of DB 86 (Nemr et al., 2009). Q_o represent the adsorption capacity of the adsorbent. The Q_o of PAC more than RH6 proves that PAC is better than RH6 in adsorption of DB 86. However, RH6 give better value compare to the study conducted by Nemr et al., (2009) by using carbon orange peel (COP) in DB 86 removal reported in **Table 2.4**.

4.7 Adsorption Kinetic

Two kinetic models i.e. pseudo first and pseudo second order were used in this study to identify the mechanism responsible for DB 86 adsorption. **Figure 4.12** and **Figure 4.13** show the linear plot of the pseudo first order for RH6 and PAC respectively. **Table 4.3** shows that the constants calculated from the plot. $q_{e,exp}$ represent the amount of adsorption at equilibrium obtained from experiment and the $q_{e,cal}$ is the calculated amount of adsorption at equilibrium based on the pseudo first order plot.

Both **Figure 4.12** and **Figure 4.13** shows that the pseudo first order equation fit well for the first 60 minutes and thereafter the data deviates from theory. Thus, the pseudo first order cannot be applied for entire adsorption process. Furthermore, the R^2 values are relatively low for RH6 compared to PAC. Calculated amount of adsorption at equilibrium, $q_{e,cal}$ based on the pseudo first order plot also very low compared to the $q_{e,exp}$ obtained from the experiment.

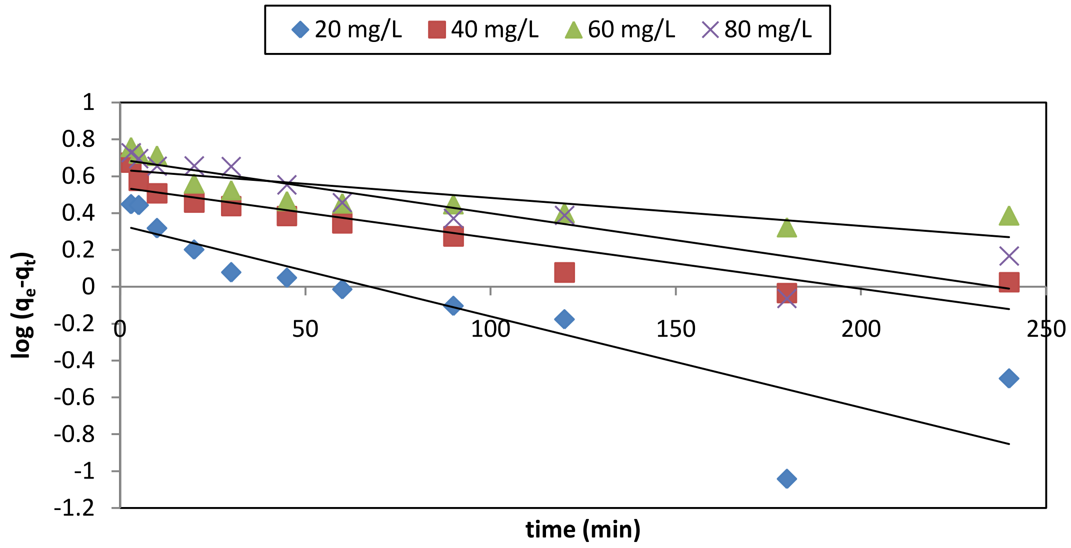


Figure 4.12: *Pseudo first order kinetic at different initial concentration (RH6)*

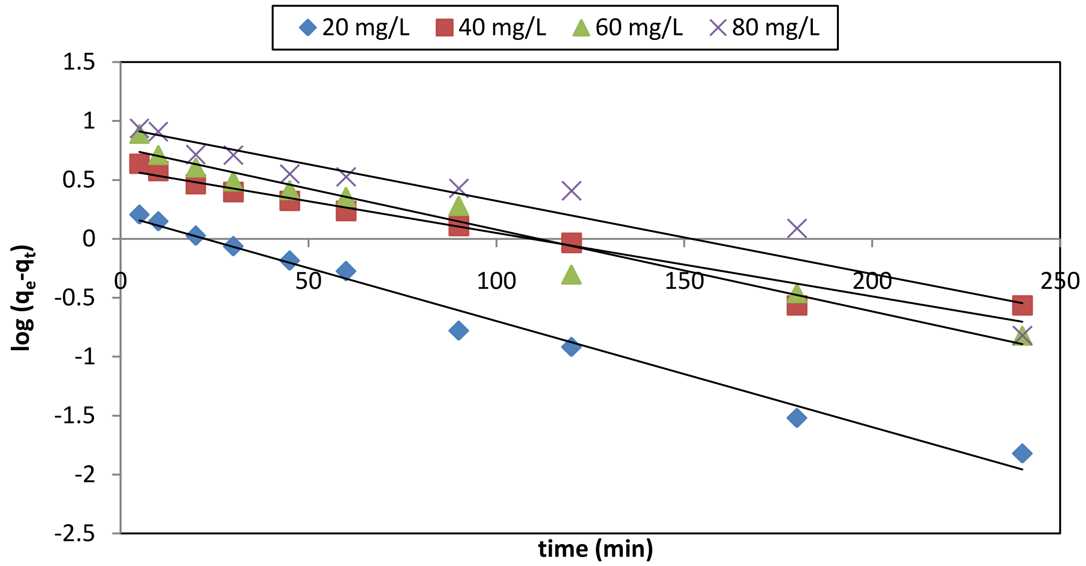


Figure 4.13: *Pseudo first order kinetic at different initial concentration (PAC)*

Table 4.3: *Pseudo first order reaction rate constants for DB 86 adsorption*

Adsorbent	Initial conc. (mg/L)	$q_{e,exp}$	$q_{e,cal}$	k_1	R^2	Equation
RH6	20.0	8.60606	2.1578	0.009212	0.778	y = -0.004x + 0.334
	40.0	14.8333	3.4594	0.004606	0.866	y = -0.002x + 0.539
	60.0	20.5606	4.9091	0.004606	0.841	y = -0.002x + 0.691
	80.0	23.0303	4.3053	0.002303	0.640	y = -0.001x + 0.634
PAC	20.0	9.87878	1.5886	0.020727	0.985	y = -0.009x + 0.201
	40.0	19.1363	3.8726	0.011515	0.961	y = -0.005x + 0.588
	60.0	27.5303	5.9293	0.013818	0.959	y = -0.006x + 0.773
	80.0	33.9849	8.7097	0.013818	0.906	y = -0.006x + 0.940

Meanwhile **Figure 4.14** and **Figure 4.15** show the pseudo second order kinetics for RH6 and PAC respectively whereas **Table 4.4** show the constants calculated from the plot of pseudo second order. The linear plot for both RH6 and PAC show good agreement with greater correlation coefficient $R^2 > 0.998$ for every initial concentration. Thus, the pseudo second order proven to be better model to describe the mechanism of adsorption kinetic of DB 86 compared to pseudo first order model. Isa et al. (2009) reported that the compliance to second order kinetic model strongly suggest chemical or chemisorptions between the adsorbent and adsorbate.

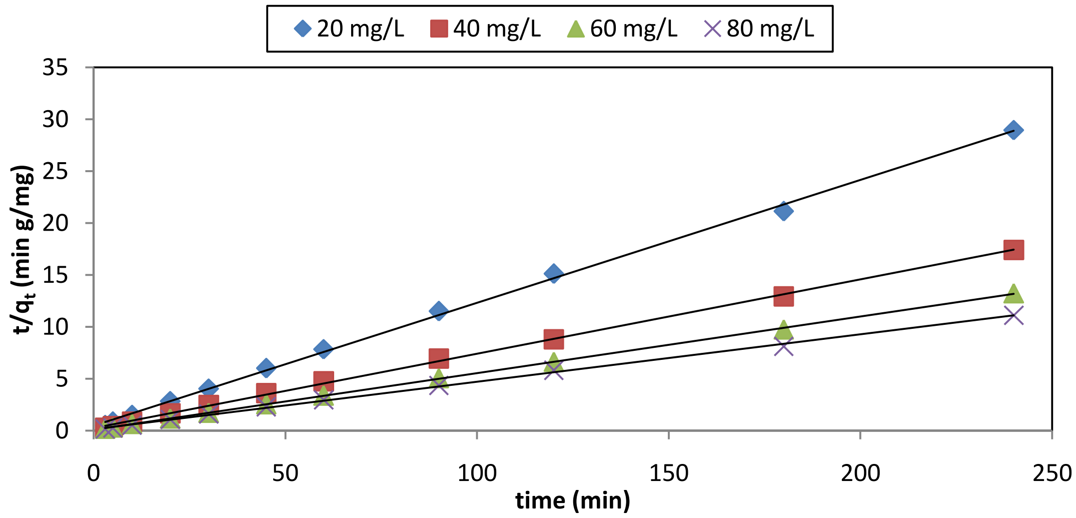


Figure 4.14: *Pseudo second order kinetic at different initial concentration (RH6)*

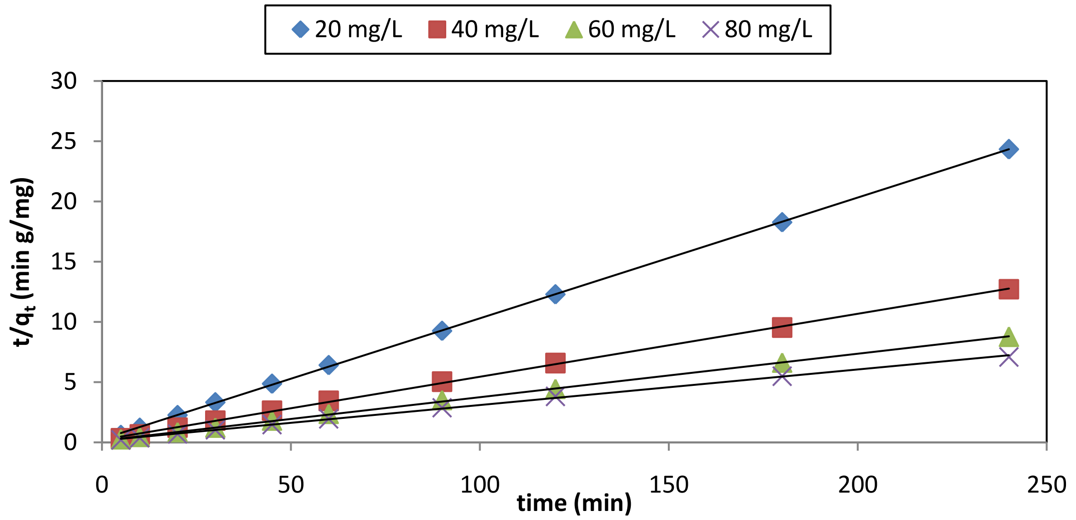


Figure 4.15: *Pseudo second order kinetic at different initial concentration (PAC)*

Table 4.4: *Pseudo second order reaction rate constants for DB 86 adsorption*

Adsorbent	Initial conc. (mg/L)	$q_{e,exp}$	$q_{e,cal}$	k_2	R^2	Equation
RH6	20.0	8.60606	8.4746	0.028948	0.998	$y = 0.118x + 0.481$
	40.0	14.8333	14.085	0.020004	0.999	$y = 0.071x + 0.252$
	60.0	20.5606	18.519	0.033517	0.999	$y = 0.054x + 0.087$
	80.0	23.0303	22.222	0.012656	0.998	$y = 0.045x + 0.160$
PAC	20.0	9.87878	10.000	0.039370	0.999	$y = 0.100x + 0.254$
	40.0	19.1363	19.231	0.012695	0.999	$y = 0.052x + 0.213$
	60.0	27.5303	27.778	0.009000	0.999	$y = 0.036x + 0.144$
	80.0	33.9849	34.483	0.005923	0.998	$y = 0.029x + 0.142$

CHAPTER 5

SUMMARY, CONCLUSIONS AND FUTURE WORK

5.1 Summary and Conclusions

The potential of the RH6 was evaluated and compared to commercial PAC. The optimum pH for DB 86 removal is pH 2 for both adsorbent. Both adsorbent give similar optimum contact time and dye concentration are 180 minutes and 20 mg/L respectively. The optimum adsorbent dosage is 4 g/L for RH6 and 3 g/L for PAC. Both Langmuir and Freundlich provide high correlation coefficients R^2 (>0.97) but Langmuir isotherm is the best to describe the process with correlation coefficients R^2 >0.99. Adsorption capacity obtained was 34.4828 mg/g for RH6 and 47.6190 mg/g for PAC. Pseudo second order kinetic model yielded high R^2 values (>0.99) to prove that the model is best fit for the adsorption mechanism compared to pseudo first order.

The dye removal process by using low-cost adsorbent from materials such as industrial and agriculture waste, fruit waste, plant waste and bioadsorbent are an interesting alternative to the conventional method such as coagulation-flocculation, ozonation and photo degradation. Despite of its cost, the preparation also very simple and most importantly, the raw material is readily available in any part of the world. This study will provide an attractive technology if the low-cost adsorbent ready for use.

5.2 Future work and Recommendations

Due to time constraint, this study only applicable for an individual dye which is DB 86. Future study for improvement of this research can be done by using the real wastewater from textile industry. A similar study can be conducted to use rice husk-based adsorbent to treat the toxicity and heavy metal removal. Besides that, the study should include desorption test to determine their reuse potential.

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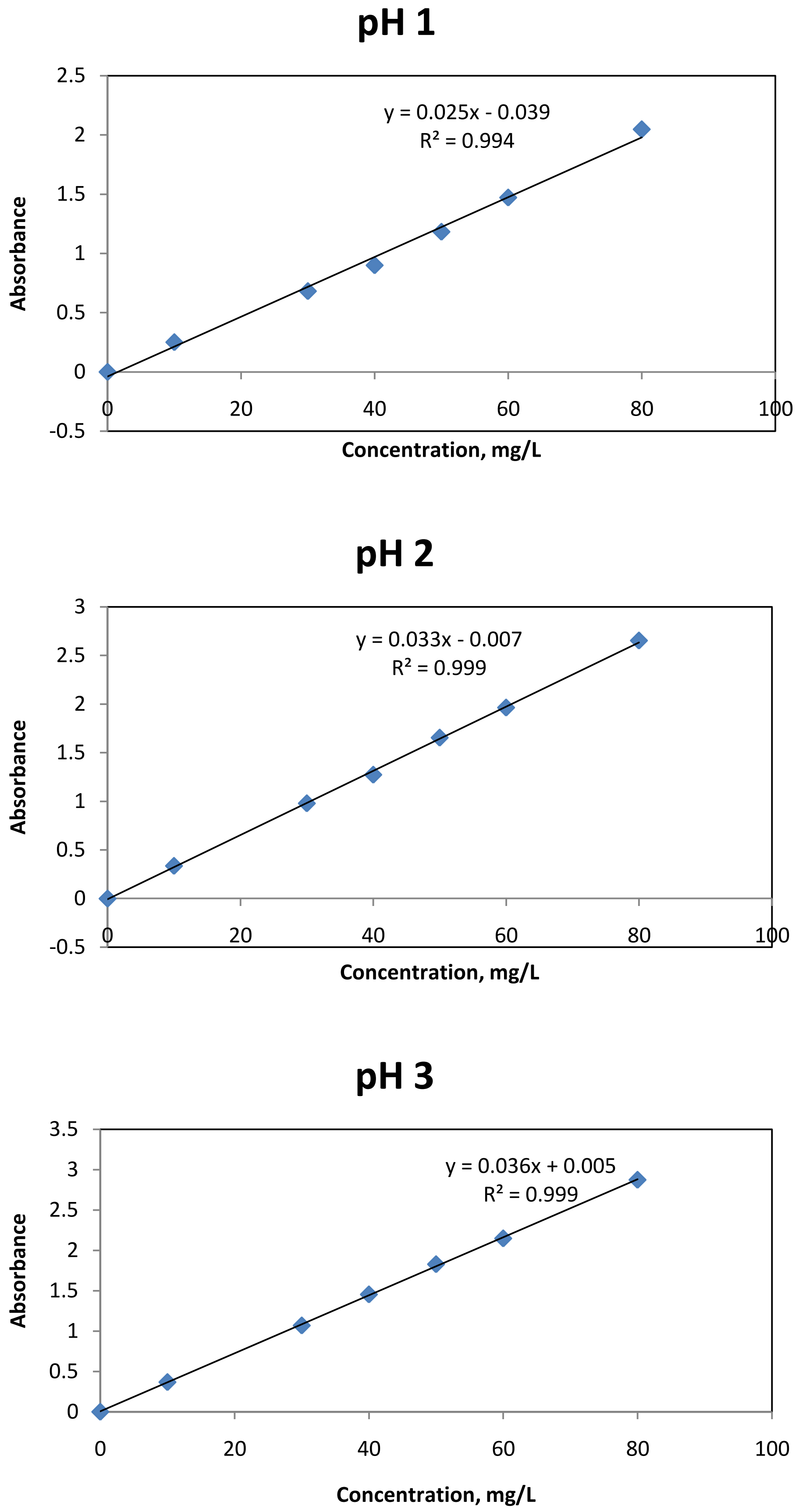
APPENDIX A

APPENDIX B

APPENDIX C


pH 1
y = 0.025x - 0.039
R² = 0.994
Absorbance
Concentration, mg/L
2.5
2
1.5
1
0.5
0
-0.5
0
20
40
60
80
100
pH 2
y = 0.033x - 0.007
R² = 0.999
Absorbance
Concentration, mg/L
3
2.5
2
1.5
1
0.5
0
-0.5
0
20
40
60
80
100
pH 3
y = 0.036x + 0.005
R² = 0.999
Absorbance
Concentration, mg/L
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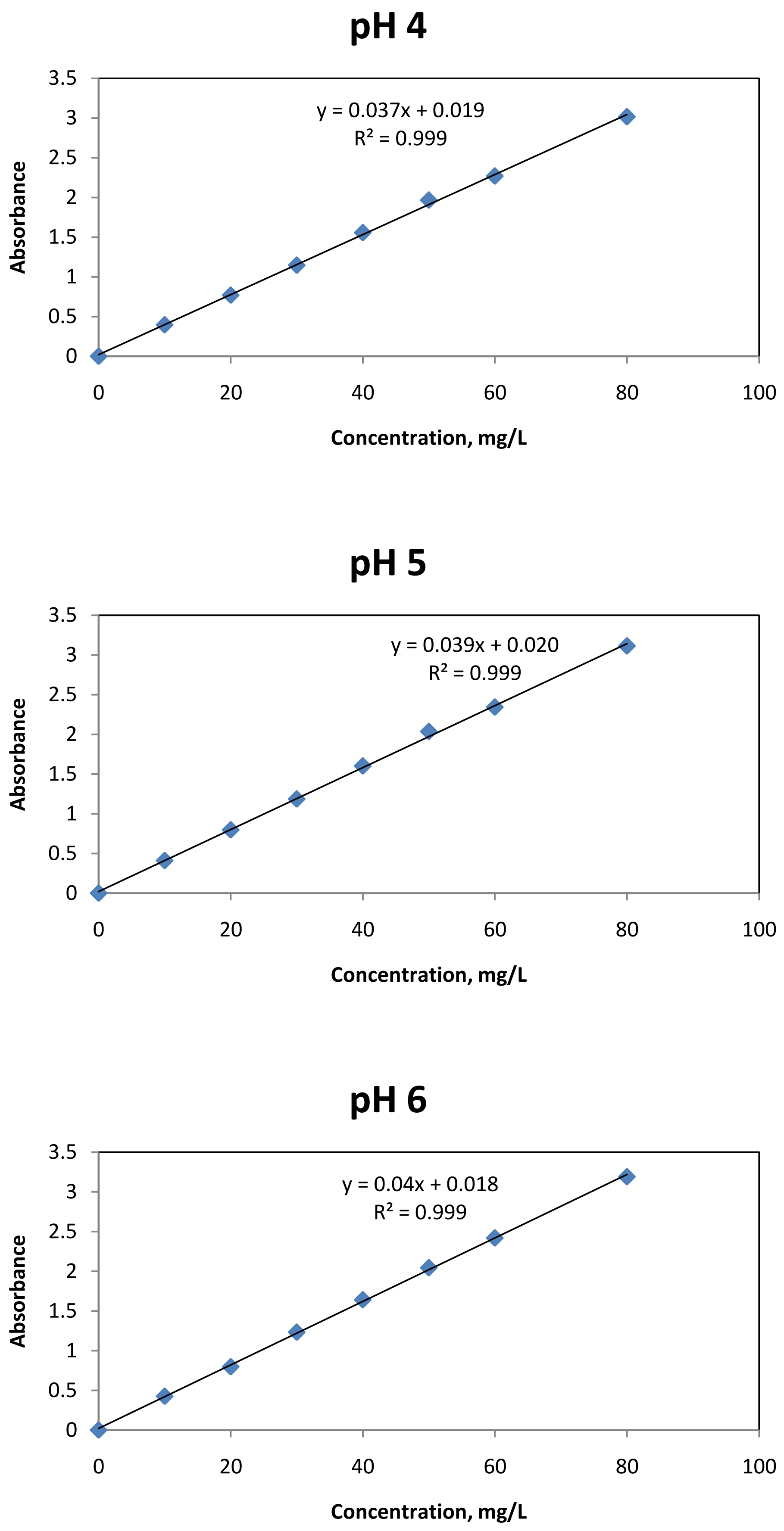
3
2.5
2
1.5
1
0.5
0
0
20
40
60
80
100

a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y = 0.037x + 0.019
R² = 0.999
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Concentration, mg/L
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0.5
1
1.5
2
2.5
3
3.5
0
20
40
60
80
100
pH 5
y = 0.039x + 0.020
R² = 0.999
Absorbance
Concentration, mg/L
0
0.5
1
1.5
2
2.5
3
3.5
0
20
40
60
80
100
pH 6
y = 0.04x + 0.018
R² = 0.999
Absorbance
Concentration, mg/L
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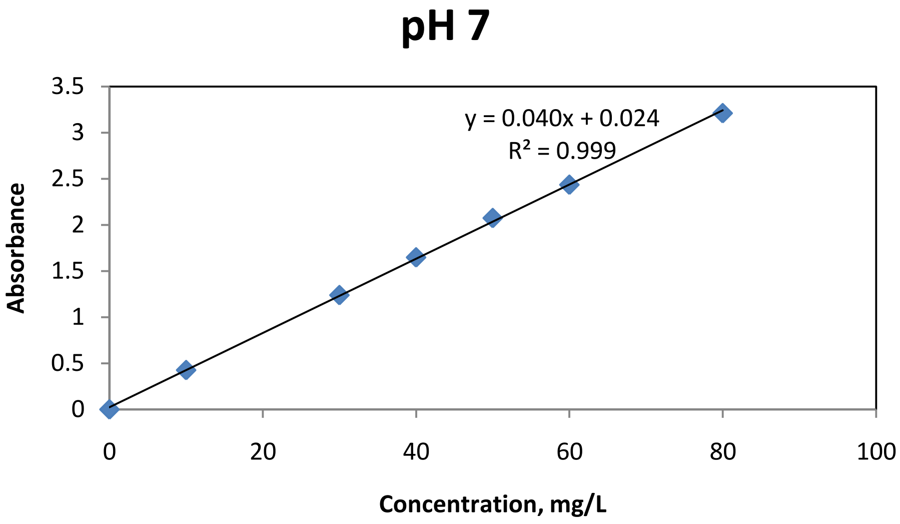
0.5
1
1.5
2
2.5
3
3.5
0
20
40
60
80
100

b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R² = 0.999
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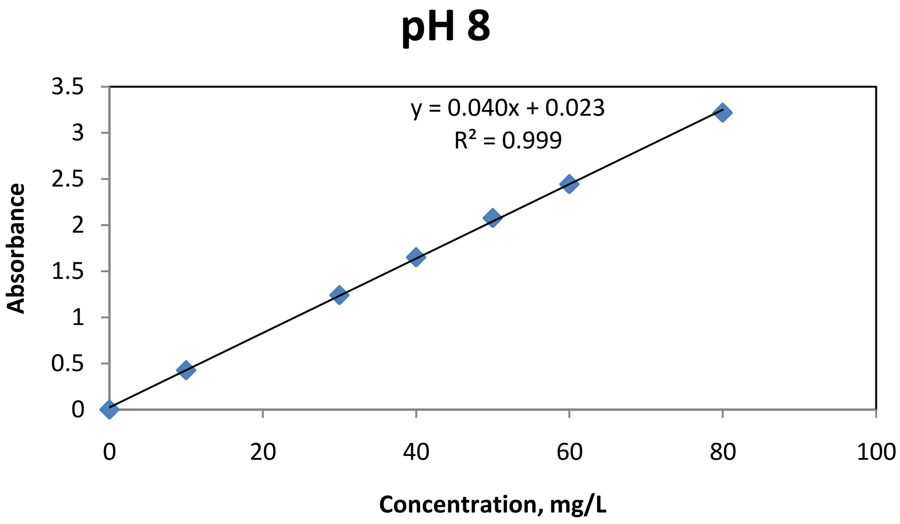
1
1.5
2
2.5
3
3.5
0
20
40
60
80
100
Concentration, mg/L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R² = 0.999
Absorbance
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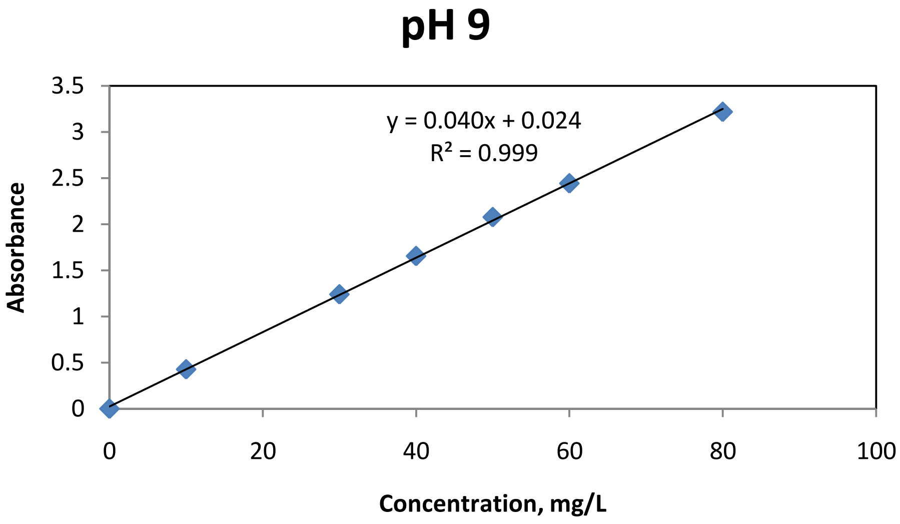
1
1.5
2
2.5
3
3.5
0
20
40
60
80
100
Concentration, mg/L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y = 0.040x + 0.024
R² = 0.999
Absorbance
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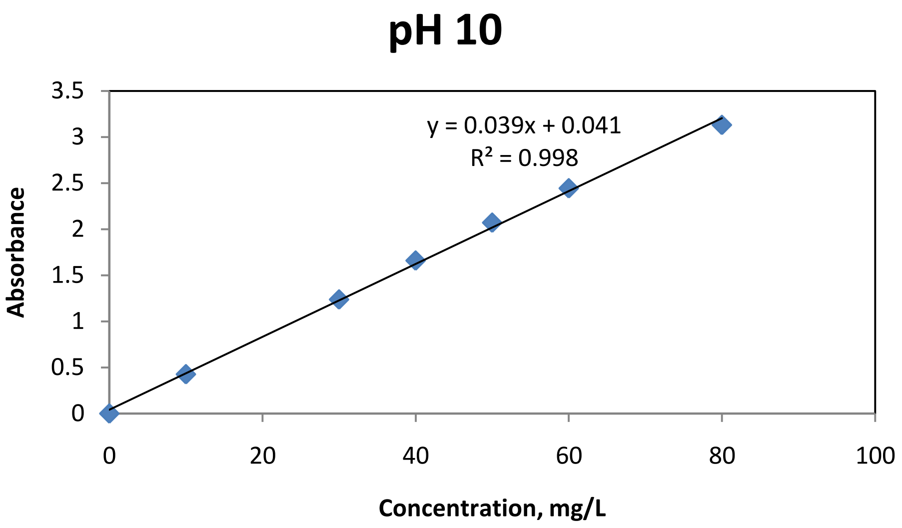
1
1.5
2
2.5
3
3.5
0
20
40
60
80
100
Concentration, mg/L


pH 10
y = 0.039x + 0.041
R² = 0.998
Absorbance
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3
2.5
2
1.5
1
0.5
0
0
20
40
60
80
100
Concentration, mg/L

d

Tabulated data for effect of pH (RH)

pH	Adsorbent	abs	Final concentration (mg/L)	Adsorbed dye	Percentage removal (%)
1	RH3	0.050	3.560	16.440	82.200
	RH4	0.040	3.160	16.840	84.200
	RH5	0.029	2.720	17.280	86.400
	RH6	0.000	0.000	20.000	100.000
	RH1	0.027	2.640	17.360	86.800
	RH2	0.023	2.480	17.520	87.600
2	RH3	0.259	8.061	11.939	59.697
	RH4	0.288	8.939	11.061	55.303
	RH5	0.192	6.030	13.970	69.848
	RH6	0.031	1.152	18.848	94.242
	RH1	0.231	7.212	12.788	63.939
	RH2	0.212	6.636	13.364	66.818
3	RH3	0.412	11.306	8.694	43.472
	RH4	0.410	11.250	8.750	43.750
	RH5	0.463	12.722	7.278	36.389
	RH6	0.526	14.472	5.528	27.639
	RH1	0.302	8.250	11.750	58.750
	RH2	0.643	17.722	2.278	11.389
4	RH3	0.644	16.892	3.108	15.541
	RH4	0.631	16.541	3.459	17.297
	RH5	0.557	14.541	5.459	27.297
	RH6	0.636	16.676	3.324	16.622
	RH1	0.398	10.243	9.757	48.784
	RH2	0.706	18.568	1.432	7.162
5	RH3	0.661	16.436	3.564	17.821
	RH4	0.652	16.205	3.795	18.974
	RH5	0.567	14.026	5.974	29.872
	RH6	0.633	15.718	4.282	21.410
	RH1	0.396	9.641	10.359	51.795
	RH2	0.742	18.513	1.487	7.436
6	RH3	0.674	16.400	3.600	18.000
	RH4	0.654	15.900	4.100	20.500
	RH5	0.568	13.750	6.250	31.250
	RH6	0.664	16.150	3.850	19.250
	RH1	0.400	9.550	10.450	52.250
	RH2	0.743	18.125	1.875	9.375

pH	Adsorbent	abs	Final concentration (mg/L)	Adsorbed dye	Percentage removal (%)
7	RH3	0.707	17.075	2.925	14.625
	RH4	0.650	15.650	4.350	21.750
	RH5	0.566	13.550	6.450	32.250
	RH6	0.676	16.300	3.700	18.500
	RH1	0.407	9.575	10.425	52.125
	RH2	0.783	18.975	1.025	5.125
8	RH3	0.711	17.200	2.800	14.000
	RH4	0.677	16.350	3.650	18.250
	RH5	0.590	14.175	5.825	29.125
	RH6	0.684	16.525	3.475	17.375
	RH1	0.450	10.675	9.325	46.625
	RH2	0.774	18.775	1.225	6.125
9	RH3	0.713	17.225	2.775	13.875
	RH4	0.691	16.675	3.325	16.625
	RH5	0.579	13.875	6.125	30.625
	RH6	0.690	16.650	3.350	16.750
	RH1	0.475	11.275	8.725	43.625
	RH2	0.803	19.475	0.525	2.625
10	RH3	0.772	18.744	1.256	6.282
	RH4	0.785	19.077	0.923	4.615
	RH5	0.621	14.872	5.128	25.641
	RH6	0.727	17.590	2.410	12.051
	RH1	0.450	10.487	9.513	47.564
	RH2	0.819	19.949	0.051	0.256

Tabulated data for effect of pH (PAC)

pH	Adsorbent	abs	Final concentration (mg/L)	Adsorbed dye	Percentage removal (%)
1	PAC	0.000	0.000	20.000	100.000
2	PAC	0.000	0.000	20.000	100.000
3	PAC	0.006	0.028	19.972	99.861
4	PAC	0.024	0.135	19.865	99.324
5	PAC	0.035	0.385	19.615	98.077
6	PAC	0.038	0.500	19.500	97.500
7	PAC	0.045	0.525	19.475	97.375
8	PAC	0.059	0.9	19.100	95.500
9	PAC	0.063	0.975	19.025	95.125
10	PAC	0.064	0.590	19.410	97.051

Tabulated data for effect of contact time and dye concentration (RH6)

Initial concentration (mg/L)	Contact time (t)	abs	Final Concentration (mg/L)	Adsorbed dye (mg/L)	Percentage Removal (%)
20	0	0.640	20.000	0.000	0.000
	3	0.270	8.394	11.606	58.030
	5	0.268	8.333	11.667	58.333
	10	0.222	6.939	13.061	65.303
	20	0.190	5.970	14.030	70.152
	30	0.164	5.182	14.818	74.091
	45	0.159	5.030	14.970	74.848
	60	0.149	4.727	15.273	76.364
	90	0.137	4.364	15.636	78.182
	120	0.129	4.121	15.879	79.394
	180	0.091	2.970	17.030	85.152
	240	0.106	3.424	16.576	82.879
40	0	1.280	40.000	0.000	0.000
	3	0.645	19.758	20.242	50.606
	5	0.582	17.848	22.152	55.379
	10	0.546	16.758	23.242	58.106
	20	0.523	16.061	23.939	59.848
	30	0.515	15.818	24.182	60.455
	45	0.494	15.182	24.818	62.045
	60	0.480	14.758	25.242	63.106
	90	0.458	14.091	25.909	64.773
	120	0.413	12.727	27.273	68.182
	180	0.395	12.182	27.818	69.545
	240	0.404	12.455	27.545	68.864
60	0	1.920	60.000	0.000	0.000
	3	0.995	30.364	29.636	49.394
	5	0.965	29.455	30.545	50.909
	10	0.955	29.152	30.848	51.414
	20	0.856	26.152	33.848	56.414
	30	0.837	25.576	34.424	57.374
	45	0.809	24.727	35.273	58.788
	60	0.803	24.545	35.455	59.091
	90	0.801	24.485	35.515	59.192
	120	0.782	23.909	36.091	60.152
	180	0.755	23.091	36.909	61.515
	240	0.777	23.758	36.242	60.404

Initial concentration (mg/L)	Contact time (t)	abs	Final Concentration (mg/L)	Adsorbed dye (mg/L)	Percentage Removal (%)
80	0	2.560	80.000	0.000	0.000
	3	1.465	44.606	35.394	44.242
	5	1.440	43.848	36.152	45.189
	10	1.411	42.970	37.030	46.288
	20	1.412	43.000	37.000	46.250
	30	1.410	42.939	37.061	46.326
	45	1.349	41.091	38.909	48.636
	60	1.302	39.667	40.333	50.417
	90	1.268	38.636	41.364	51.705
	120	1.274	38.818	41.182	51.477
	180	1.170	35.667	44.333	55.417
	240	1.210	36.879	43.121	53.902

Tabulated data for effect of contact time and dye concentration (PAC)

Initial concentration (mg/L)	Contact time (t)	abs	Final Concentration (mg/L)	Adsorbed dye (mg/L)	Percentage Removal (%)
20	0	0.640	20.000	0.000	0.000
	5	0.107	3.455	16.545	82.727
	10	0.094	3.061	16.939	84.697
	20	0.071	2.364	17.636	88.182
	30	0.058	1.970	18.030	90.152
	45	0.044	1.545	18.455	92.273
	60	0.036	1.303	18.697	93.485
	90	0.012	0.576	19.424	97.121
	120	0.009	0.485	19.515	97.576
	180	0.003	0.303	19.697	98.485
	240	0.002	0.273	19.727	98.636
40	0	1.280	40.000	0.000	0.000
	5	0.337	10.424	29.576	73.939
	10	0.298	9.242	30.758	76.894
	20	0.242	7.545	32.455	81.136
	30	0.215	6.727	33.273	83.182
	45	0.189	5.939	34.061	85.152
	60	0.164	5.182	34.818	87.045
	90	0.135	4.303	35.697	89.242
	120	0.111	3.576	36.424	91.061
	180	0.068	2.273	37.727	94.318
	240	0.068	2.273	37.727	94.318

Initial concentration (mg/L)	Contact time (t)	abs	Final Concentration (mg/L)	Adsorbed dye (mg/L)	Percentage Removal (%)
60	0	1.920	60.000	0.000	0.000
	5	0.671	20.545	39.455	65.758
	10	0.497	15.273	44.727	74.545
	20	0.423	13.030	46.970	78.283
	30	0.360	11.121	48.879	81.465
	45	0.328	10.152	49.848	83.081
	60	0.306	9.485	50.515	84.192
	90	0.282	8.758	51.242	85.404
	120	0.189	5.939	54.061	90.101
	180	0.179	5.636	54.364	90.606
	240	0.166	5.242	54.758	91.263
80	0	2.560	80.000	0.000	0.000
	5	0.961	29.333	50.667	63.333
	10	0.927	28.303	51.697	64.621
	20	0.732	22.394	57.606	72.008
	30	0.729	22.303	57.697	72.121
	45	0.625	19.152	60.848	76.061
	60	0.612	18.758	61.242	76.553
	90	0.567	17.394	62.606	78.258
	120	0.559	17.152	62.848	78.561
	180	0.471	14.485	65.515	81.894
	240	0.460	14.152	65.848	82.311

Tabulated data for effect of adsorbent dosage (RH6)

adsorbent dosage (mg/L)	abs	Initial concentration (mg/L)	Final concentration (mg/L)	Adsorbed dye (mg/L)	Percentage Removal (%)
0	0.64	20	20	0	0
1.5	0.157	20	4.969697	15.030303	75.15151515
2	0.085	20	2.787879	17.212121	86.06060606
2.5	0.056	20	1.909091	18.090909	90.45454545
3	0.038	20	1.363636	18.636364	93.18181818
3.5	0.007	20	0.424242	19.575758	97.87878788
4	0	20	0	20	100
6	0	20	0	20	100
8	0	20	0	20	100
10	0	20	0	20	100

Tabulated data for effect of adsorbent dosage (PAC)

adsorbent dosage (mg/L)	abs	Initial concentration (mg/L)	Final concentration (mg/L)	Adsorbed dye (mg/L)	Percentage Removal (%)
0	0.64	20	20	0	0
0.5	0.14	20	4.454545	15.545455	77.72727273
1	0.078	20	2.575758	17.424242	87.12121212
1.5	0.04	20	1.424242	18.575758	92.87878788
2	0.03	20	1.121212	18.878788	94.39393939
2.5	0.005	20	0.363636	19.636364	98.18181818
3	0	20	0	20	100
4	0	20	0	20	100
5	0	20	0	20	100
6	0	20	0	20	100

Tabulated data for equilibrium adsorption isotherm (RH6)

Initial conc. (mg/L)	abs	Final Conc (C_e)	Adsorbed dye (C_o-C_e)	Percentage Removal (%)	q_e	C_e/q_e	log C_e	log q_e
20	0.085	2.788	17.212	86.061	8.60606	0.32394	0.4453	0.9348
40	0.334	10.333	29.667	74.167	14.8333	0.69663	1.0142	1.17124
60	0.616	18.879	41.121	68.535	20.5606	0.9182	1.276	1.31304
80	1.113	33.939	46.061	57.576	23.0303	1.47368	1.5307	1.3623
100	1.580	48.091	51.909	51.909	25.9545	1.85289	1.6821	1.41421
120	2.086	63.424	56.576	47.146	28.2879	2.2421	1.8023	1.4516
140	2.659	80.788	59.212	42.294	29.6061	2.72876	1.9073	1.47138
160	3.219	97.758	62.242	38.902	31.1212	3.14119	1.9902	1.49306

Tabulated data for equilibrium adsorption isotherm (PAC)

Initial conc. (mg/L)	abs	Final Conc (C_e)	Adsorbed dye (C_o-C_e)	Percentage Removal (%)	q_e	C_e/q_e	log C_e	log q_e
20	0.001	0.242	19.758	98.788	9.87879	0.02454	-0.61542	0.994704
40	0.050	1.727	38.273	95.682	19.1364	0.09026	0.237361	1.281859
60	0.156	4.939	55.061	91.768	27.5303	0.17942	0.693674	1.439811
80	0.390	12.030	67.970	84.962	33.9848	0.35399	1.080277	1.531285
100	0.774	23.667	76.333	76.333	38.1667	0.620087	1.374137	1.581684
120	1.096	33.424	86.576	72.146	43.2879	0.772139	1.524062	1.636366
140	1.782	54.212	85.788	61.277	42.893939	1.263864	1.734096	1.632396
160	2.212	67.242	92.758	57.973	46.378788	1.449853	1.827643	1.666319