# PARAMETRIC AND ADSORPTION KINETIC STUDIES OF REACTIVE BLACK 5 REMOVAL FROM TEXTILE SIMULATED SAMPLE USING OIL PALM (*ELAIS GUINEENSIS*) EMPTY FRUIT BUNCH

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THIS DISSERTATION IS SUBMITTED AS A PARTIAL REQUIREMENT TO OBTAIN DEGREE OF BACHELOR OF SCIENCE WITH HONOURS

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I declare that this dissertation is based on my original work except for questions and summaries, each of which has been fully acknowledged.

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## ABSTRACT

The potential of empty fruit bunch as a low-cost biosorbent for the removal of Reactive Black 5 from aqueous solution was investigated in this study. The influences of pH, contact time, initial concentration and biosorbent dosage were studied in batch experiments at room temperature. The empty fruit bunch used in this study was characterized by FTIR spectroscopy and it was found that O-H, N-H, and C=O stretching were present. Maximum adsorption uptake of Reactive Black 5 dye was found to be at pH 2. At pH 2, the adsorption was rapid at the first 5 minutes of contact, with uptake of more than 50%, and equilibrium was achieved in 30 minutes of agitation. It was also found that higher biosorbent dosage increases dye uptake, of up to 90%. Kinetic studies showed good correlation coefficient for a pseudo-second-order kinetic model. Langmuir, Freundlich, and BET isotherm models were applied to describe the biosorption of Reactive Black 5 dyes onto empty fruit bunch. Langmuir model fitted the equilibrium data better, giving correlation coefficient of 0.9999 and a maximum adsorption capacity of 7.3421 mg/g. Both pseudo-second-order kinetic model and Langmuir model indicates monolayer coverage on adsorbent. The results showed that empty fruit bunch have the potential to be applied as alternative low-cost biosorbent in the remediation of dye contamination in waste water.

***KAJIAN PENJERAPAN KINETIC UNTUK PENYINGKIRAN PEWARNA REACTIVE BLACK 5 DARIPADA LARUTAN AKUES TEKSTIL MENGGUNAKAN TANDAN BUAH KELAPA SAWIT KOSONG***

## *ABSTRAK*

*Potensi tandan kelapa sawit sebagai penjerap berkos rendah dalam penyingkiran pewarna Reactive Black 5 daripada larutan akues telah dikaji. Pengaruh pH, masa sentuhan, kepekatan ion permulaan, dan dos penjerap telah dikaji secara kelompok pada suhu bilik. FTIR spektroskopi telah dijalankan ke atas tandan buah kelapa sawit kosong telah digunakan dalam kajian ini dan regangan O-H, N-H, and C=O telah dijumpai. Keupayaan sorpsi maksimum telah dijumpai pada pH 2. Penyingkiran ion adalah cepat dalam masa 5 minit sentuhan pertama, dengan penjerapan lebih daripada 50%, dan keseimbangan telah dicapai dalam masa 30 minit. Dos penjerap yang tinggi juga menunjukkan kadar penjerapan pewarna yang tinggi, sehingga mencapai 90%. Kajian kinetik menunjukkan korelasi yang tinggi bagi model kinetik pseudo-kadar-kedua. Model-model isoterma Langmuir, Freundlich, dan BET telah digunakan untuk menggambarkan penjerapan pewarna Reactive Black 5 ke atas buah tandan kelapa sawit kosong. Model Langmuir menunjukkan keputusan yang lebih memuaskan, dengan korelasi 0.9999 dan keupayaan penjerapan maksimum 7.3421 mg/g. Kedua-dua model kinetic pseudo-kadar-kedua dengan model isoterma Langmuir menunjukkan lingkupan lapisan mono ke atas buah tandan kelapa sawit kosong. Keputusan kajian menunjukkan buah tandan kelapa sawit kosong mempunyai potensi sebagai penjerap berkos rendah alternatif dalam memulihkan air sisa yang dicemari pewarna.*

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## LIST OF SYMBOLS AND ABBREVIATIONS

| | |
|---|---|
| AOP | Advance Oxidation Process |
| BET | Brunauer–Emmett–Teller |
| BOD | Biological Oxygen Demand |
| Ce | Equilibrium concentration of adsorbate |
| $C_e$ | Final concentration |
| $C_i$ | Initial concentration |
| COD | Chemical Oxygen Demand |
| EDX | X-ray Spectroscopy |
| EFB | Empty Fruit Bunch |
| FTIR | Fourier Transform Infrared Spectroscope |
| $K_L$ | Langmuir adsorption constant related to the free energy adsorption |
| $q_e$ | Amount of adsorbate adsorbed at equilibrium |
| $q_{max}$ | Maximum monolayer adsorption capacity of the adsorbent |
| $Q_o$ | Langmuir constant related to rate of adsorption |
| $q_t$ | Amount of dye absorbed |
| $R^2$ | Correlation coefficient |
| RB5 | Reactive Black 5 |
| SEM | Scanning Electron Microscope |
| TOC | Total Organic Carbon |
| UV-VIS | Ultraviolet-Visible |
| XRD | X-ray Diffraction |
| λmax | Maximum wavelength |

# CHAPTER 1

# INTRODUCTION

## 1.1 Background of Study

Approximately more than 70% of the Earth's surface is covered by water. However, of all the water available on Earth, only 2.5% are fresh water. Freshwater is characterized as having low concentration of dissolved salts and other dissolved solids. Of the freshwater available, most of it, almost 70% are frozen in the icecaps of Antarctica and Greenland. The remaining freshwater lies in deep underground aquifers as groundwater or exist as the moisture found in soil, unfit for human use. This means that the available freshwater accessible to direct human use is less than 1% (Gleick, 2000).

Water is of great importance to us, and water pollution has become a major concern nowadays. One of the contribution to water pollution are dyes used in industries such as printing, paper-making, plastic, coating, food and especially textile industry, which uses dyes and pigments to color their products (Elizalde-Gonzalez and Pelaez-Cid, 2003). The wastewater discharges from these industries are worrisome and it has been reported that 10 - 15% of the dye is released to the environment after dyeing process (Chowdhury *et al.*, 2011).

The degree of pollution of water is determined by biochemical oxygen demand (BOD), chemical oxygen demand (COD), suspended solids, bad smell, toxicity and color (Ali & Sreekrishnan, 2001). Wastewater contaminated with dyes, gives it color which reduces light penetration, affecting aquatic life, and more importantly increases toxicity making it unfit for human use (El-Geundi, 1991). Most dyes and color effluents are toxic, having carcinogenic and mutagenic effects that not only affect the human health, but also causes harmful effects on the environment. Hence, removal of these dyes has become a major concern and much effort is constantly put into the search of a proper and efficient way to achieve better treatment of dye containing effluents.

The primary functions of dyes are to give the chosen raw material, a coating layer of long lasting colours. Therefore, dyes are designed to have properties which are resistant to soap, water, light and oxidizing agents. Furthermore, synthetic dyes have complex aromatic structures, increasing its stability and resistance to biodegradation. Hence, finding an efficient dye-removal method for the treatment of dye containing effluents proves to be a challenging problem (Khataeė *et al.*, 2013) and (Akar *et al.*, 2009).

The conventional treatment of dye containing effluents may include processes that combine physical, chemical or biological in nature. The aforementioned processes includes coagulation/flocculation, advanced oxidation processes, ozonation, membrane filtration, electroflotation, electrokinetic coagulation, electrochemical destruction, ion exchange, irradiation, precipitation and biological treatment. These processes however, have its disadvantages. The most significant include high reagent and energy necessity, low selectivity, high capital and operational cost and also the generation of secondary wastes that is difficult to be removed or degraded (Haddad *et al.*, 2013). An effective alternative process to treat dye containing effluents is by means of adsorption. Currently, a popular and widely used adsorption technique is by using activated carbon as the biosorbent. This is due to its high adsorption capacity, surface area and degree of surface reactivity as well as microporous structures. But it still shows common problems such as high operating costs and regeneration problems (Akar *et al.*, 2009).

This causes an increasing interest in using low cost, easily available materials for the adsorption of dye colours in recent years. Considerable amount of researches have been done in search of a low cost adsorbent, one which is abundant in nature, or is a by-product or waste material from another industry (Khalaf, 2008). Different agricultural biomasses such as tree fern, peat and rice husk, peanut hull, sugarcane dust, apple pomace and wheat straw, bark, palm kernel fibre, banana peel and orange peel, coir pith, linseed cake and sawdust have been previously tried for the removal of different types of dyes. However, the search for a more effective and economical biosorbent materials is still needed (Akar *et al*,. 2009).

## 1.2 Objectives of Study

The objectives of this study are:

i. to determine suitability of empty fruit bunch of *Elaeis guineensis* (Oil palm) as biosorbent for removal of reactive black 5 from textile effluents,

ii. to study adsorption, kinetic, isothermal of empty fruit bunch in the removable of reactive black 5 from textile effluents,

iii. to conduct parametric study of biosorption in regard to pollution concentration, absorbent dosage and solution pH.

## 1.3 Scope of Study

The purpose of this research was to further investigate the parametric and adsorption kinetic studies of Reactive Black 5 removal from textile simulated sample by using empty fruit bunch. The experiment to be described below was an attempt to provide some further data on the role and position of a summary in a research report, using empty fruit bunch.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 Dyes

Application of colors to different textiles, such as leather, paper, plastics and especially fabrics makes it much more attractive (Haddad *et al.*, 2013). Hence, the application of a process known as dyeing, a technique used to impart long lasting colour onto a desired substance was introduced. The first historical records of dyeing dates back to 3500 BC, where natural dyes are extracted from certain vegetables, fruits and flowers. These however, provided a dull and limited range of colours. In 1856, W.H Perkins made the discovery of synthetic dyes, which were far more superior to its predecessors. Synthetic dyes show a bright and wider range of colours, together with its better ability to "fasten" itself onto different fabrics and textiles. This results in the bloom of the dye application industry (Rita, 2012).

### 2.1.1 Classification of Dyes

Two important components of dyes are chromophores, responsible for producing the desired color, and auxochromes, which enhances the affinity of the dye toward the fibers. In general, common dyes used in the textile industries are basic dyes, acid dyes, reactive dyes, direct dyes, azo dyes, mordant dyes, vat dyes, disperse dyes and sulfur dyes, where azo derivatives are the major class of dyes that are used in the industry today. Table 2.1 show the typical dyes used in textile dyeing operations.

Dyes can be classified into cationic, anionic and non-ionic dyes. Cationic dyes are basic dyes while the anionic dyes include direct, acid and reactive dyes (Salleh *et al.*, 2011).

**Table 2.1** Typical dyes used in textile dyeing operations (Source: Demirbas, 2009).

| Dye class | Description |
|---|---|
| Acid | Water-soluble anionic compounds |
| Basic | Water-soluble, applied in weak acidic dyebaths. Very bright dyes |
| Direct | Water-soluble, anionic compounds; can be applied directly to cellulosics without mordants (or metals such as Chromium and copper) |
| Disperse | Not water soluble |
| Reactive | Water-soluble, anionic compounds. Largest of the dye class |
| Sulfur | Organic compounds containing sulphur or sodium sulphide |
| Vat | Water-soluble. Oldest dyes which are chemically more complex |

### 2.1.2 Cationic Dyes

Cationic dyes are mainly used in acrylic, wool, nylon and silk dyeing industries. Cationic dyes include different chemical structures, which are based on substituted aromatic groups. These dyes are also known as basic dyes which depend on a positive ion; generally hydrochloride or zinc chloride complexes.

Cationic dyes carry a positive charge in their molecule. They are water soluble and yield colored cations in solution. Cationic functionality is found in various types of dyes, mainly in cationic azo dyes and methane dyes, also in anthraquinone, di- and tri-arylcarbenium, phthalocyanine dyes, various polycarbocyclic and solvent dyes (Salleh *et al.*, 2011).

### 2.1.3 Anionic Dyes

Anionic dyes are mainly used in silk, wool, polyamide, modified acrylic and polypropylene fiber industries. Anionic dyes have good water solubility. Unlike cationic dyes, anionic dyes depend on a negative ion. Anionic dyes includes many compounds from the most varied classes of dyes, which exhibit characteristic differences in structure (e.g., azoic, anthraquinone, triphenylmethan and nitro dyes) but possess as a common feature, water-solubilizing, ionic substituents. The anionic dyes also include direct dyes, and from the chemical standpoint the group of anionic azo dyes includes a large proportion of the reactive dyes.

### 2.1.4 Toxicity of Dyes

Textile dyes are mainly composed of polyaromatic compounds and are resistant to biological degradation. This means that when a body of water is contaminated or polluted with textile dyes, they cannot be broken down to simple harmless compound through action of biological agents (Gross, 2002).

The textile dye industry uses more than 8000 chemicals in a series of different processes of textile manufacture, which includes dyeing and printing. Some harmful and hazardous chemicals include aniline, dioxin and formaldehyde, which are highly carcinogenic. Other chemicals are listed in Table 2.2. Most of these chemicals are highly toxic and damaging to human health, directly or indirectly (Handa, 1991).

**Table 2.2** Major chemicals and dyes used in synthetic textile mills (Source: Handa, 1991).

| No. | Chemical | Quantity Kg/month |
|---|---|---|
| 1 | Acetic Acid | 1611 |
| 2 | Ammonium Sulphate | 858 |
| 3 | P V Acetate | 954 |
| 4 | Wetting Agent | 125 |
| 5 | Caustic Soda | 6212 |
| 6 | Softener | 856 |
| 7 | Organic Solvent | 247 |
| 8 | Organic Resin | 5115 |
| 9 | Formic Acid | 1227 |
| 10 | Soap | 154 |
| 11 | Hydrosulphites | 6563 |

| | | |
|---|---|---|
| 12 | Hydrochloric Acid | 309 |
| 13 | Hydrogen Peroxide | 1038 |
| 14 | Leveling & Dispersing Agent | 547 |
| 15 | Solvent 1425 | 321 |
| 16 | Oxalic Acid | 471 |
| 17 | Polyesthylene Emulsion | 1174 |
| 18 | Sulphuric Acid | 678 |
| 19 | Disperse Dyes (Polyester) | 1500 |
| 20 | Vat Dyes (Viscose) | 900 |
| 21 | Sulphur Dyes | 300 |
| 22 | Reactive Dyes | 45 |

## 2.2 Reactive Black 5

In this study, C.I. Reactive Black 5 commercial dye is considered in order to establish an optimized experimental condition to dye degradation. Reactive Black 5 is an anionic dye. Anionic dyes affix to fibers by means hydrogen bonding, Van der Waals forces and ionic bonding. They are usually commercially available as the sodium salt; therefore they are in anionic in solutions. There is an attraction of anionic dye molecule towards the cationic site on the fiber. Figure 2.1 shows the molecular structure of Reactive Black 5.

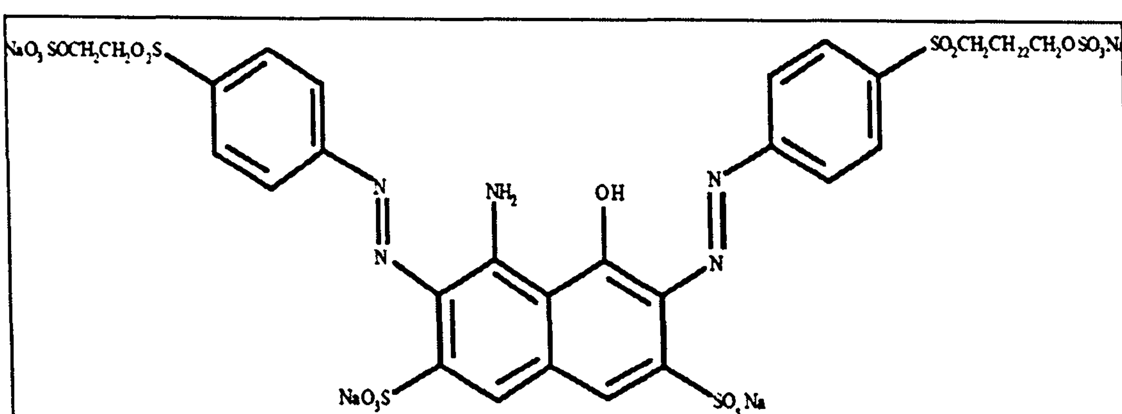

**Figure 2.1** Molecular structure of C.I. Reactive Black 5 (Esteves & Sousa, 2007).

This dye is chosen due the harmful effects it causes. While it is non toxic by itself, when inhaled and ingested by the human body, the dye molecule is metabolized and broken back down into the original intermediates used in manufacture. These intermediates, such as ortho-toluidine and benzidine, are highly toxic and found to be carcinogenic.

## 2.3 Wastewater Treatments

As dye polluted waters are of a major concern, many treatment methods and processes are widely researched and utilized in search of the optimum wastewater treatment technique. Of the conventional methods studied, cost, effectiveness, secondary reactions and scale of operations are some of the concerns (Wang *et al.*, 2012).

### 2.3.1 Comparisons of Conventional Textile Wastewater Treatment Processes

#### a. Coagulation/Flocculation

Currently, dye removal from wastewater by coagulation/flocculation is one of the most popular treatment processes. It is relatively harmless as no toxic intermediate produced during the flocculation process of dye polluted wastewater (Wang *et al.*, 2012). Flocculation/coagulation using alum, ferric sulphate, lime and some synthetic organic polymers was found to be effective for color and organic removal from textile wastewater (Mishra & Bajpai, 2006). However, for this method to be cost effective, a large scale flocculation operation is required (Wang *et al.*, 2012).

#### b. Advance Oxidation Process (AOP)

Advanced oxidation processes (AOPs), is a process where oxygen-based radicals (•OH, HOO• and $O_2^-$•) are generated *in situ* from water and $O_2$, to be applied to dye degradation. These generated radicals take part in several different reactions to degrade dye molecules completely. AOP are clean processes because harmful dye molecules are decomposed to low-molecular-weight compounds (e.g., small aldehydes, carboxylic acids or small inorganic compounds), $CO_2$, and $H_2O$, and no significant or solid secondary pollution is generated (Hisaindee *et al.*, 2013).

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