REMOVAL OF TEXTILE DYE BY

ELECTROCHEMICAL TREATMENT

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ABSTRAK

Salah satu masalah utama yang berhubungkait dengan air sisa daripada industri tekstil adalah warna. Scara amnya, pewarna reaktif yang dibebaskan daripada industri adalah susah diuruskan disebabkan oleh ketidakserasian serta ketidakpatuhan dengan rawatan biologi. Kajian ini dijalankan ke atas air sisa yang mengandungi *"reactive Red dye" (Cibacron Red CR)* dengan menggunakan kaedah elektrokimia yang terdiri daripada cast iron sebagai elektrod dan sodium chloride (NaCl) sebagai elektrolit bagi tujuan menyahwarnakan bahan pewarna tersebut. Objektif utama kajian ini dijalankan adalah untuk menentukan optimum bagi kombinasi faktor tahap pengaruh bagi penyahwarnaan pewarna. Suatu eksperimen telah dijalankan bagi menentukan kesan beberapa parameter yakni kepekatan eletrolit, kepekatan pewarna, ketumpatan serta masa reaksi bagi penyahwarnaan dengan menggunakan elektrod besi beracuan. Spektrometer turut digunakan dengan penetapan pada 525mm bagi mengukur tahap penyerapan sebelum and selepas sampel dinyahwarnakan. Kepekatan NaCl tidak mempengaruhi kesan penyahwarnaan disebabkan kepekatan yang digunakan dalam kajian ini adalah jauh lebih tinggi daripada keperluan optimum. Tahap penyahwarnaan didapati bergantung kepada ketumpatan semasa, kepekatan pewarna dan juga masa reaksi. Penambahan ketumpatan semasa dan masa reaksi serta pengurangan kepekatan pewarna akan meningkatkan keberkesanan proses penyahwarnaan. Eksperimen ini dijalankan dengan merujuk kepada faktor rekabentuk iaitu 4 faktor pada 3 tahap masing-masing. Keputusan menunjukkan bahawa lebih 99% penyahwarnaan tercapai dibawah keadaan operasi yang optima.

ABSTRACT

One of the main problems associated with wastewater from textile industries is colour. Reactive dyes in industrial discharge, in particular, are difficult to manage as they are not readily amenable to biological treatment. The present study employs an electrochemical method using cast iron electrodes and sodium chloride (NaCl) as electrolyte to decolourise synthetic wastewater containing reactive Red dye (Cibacron Red CR). The main objective of this study was to determine the optimum combination of influencing factor levels for dye removal. A laboratory-scale batch experiment was used to investigate the effect of operating parameters, electrolyte (NaCl) concentration (0.6-1.0 g/l), initial dye concentration (20-100 mg/l), current density (1-3 mA/cm^2) and reaction time (20-60 min), on colour removal using cast iron electrodes. A scanning UV-visible spectrophotometer set at 525 nm was used to measure the absorbance before and after the dye solution was treated. NaCl concentration did not effect dye removal because its concentration used in this study was found to be greater than the optimum required. Removal was found to be highly dependent on, current density, initial dye concentration and reaction time. Increasing the current density and reaction time and lowering the initial dye concentration showed an increase in the colour removal. The experiment was conducted following a factorial design with 4 factors at 3 levels each. The results show that over 99% dye removal may be achieved under optimum operating conditions. The multiple correlation coefficient of determination, R-Squared was 0.8148. This shows that the actual data give a quite good fit to the predicted data.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Large amounts (around 10^6 tons) of chemically different dyes are produced annually world-wide for use in various industrial applications. They are used extensively in the dye and printing industries, and 5-10% of the dyestuffs are lost in the industrial effluents (Sanroman *et al*., 2005). As for textile industries billions of litres of aqueous waste streams are generated every day. These effluents usually possess colour, high electrolyte concentration and a substantial amount of residual dyes that can produce environmental problems. Colour is usually the first contaminant to be recognized in wastewater; a very small amount of dye in water (10-20 mg/l) is also highly visible and affects water transparency and gas solubility of waterbodies (Cameselle *et al*., 2005). These contaminated wastewater must first be treated before being released into the environment to avoid pollution and adverse impact. The treated wastewater must comply with the environmental regulatory standards set by the Government.

Textile manufacturing begins with the production or harvest of raw fiber. After the raw natural or synthetic fibers are shipped from the farm or the chemical plant, they pass through four main stages of processing: yarn production, fabric production, finishing, and fabrication. For most uses, these fabrics must undergo further processing, which includes bleaching, printing, dyeing, mechanical finishing, preshrinking, and shaping. Many different textures can also be obtained through the

application of resins and sizings and the use of high temperature and pressure (Yang and Jared, 2005).

The textile industry produces large volumes of wastewater in their dyeing and finishing processes. These effluents have common characteristics due to their high colouration since a small amount of residual dye (of the order mg/l) can be sufficient to cause a significant visual effect (Lopez and Gutierrez, 2005). The textile activities have a high potential environmental impact, principally due to the release of large volumes of wastewaters that contain high organic charge and strong coloration. Many dyes used in textile processes show toxicity to the aquatic biota (or can be biologically transformed to toxic species) and could cause interference in natural photosynthetic processes (Ronaldo *et al.*, 1999).

Textile dye processes are among the major industrial water users; in many areas, this industry has the wastes most difficult to treat satisfactorily. Textile dye wastewater is well known to contain strong colour, high pH, temperature and COD and low biodegradability, especially the effluent from the dyeing stages of the dyeing and finishing process. The removal of dyes is therefore a challenge to both the textile industry and the wastewater-treatment facilities that must treat it (Vlyssides *et al.*, 1999).

Several processes have been used for the removal of dyes and colour from wastewater. Biological processes show limited colour removal due to the toxic nature of some dyes and the high salt concentration, which makes a pre-treatment mandatory. Adsorption on activated carbon is technically easy but has a highly expensive waste disposal. Membrane filtration provides the production of pure water that can be reused, but low mass dyes can pass through the membranes, which also have a considerable cost. Coagulation-flocculation processes with either ferric or aluminium salts or lime produce large amounts of waste sludge. Chemical oxidation with ozone or other oxidizing chemicals normally results in very high costs (Cameselle *et al*., 2005). These technologies have been applied at industrial scale, offering a good colour removal, but they have high cost as a common factor (Lopez and Gutierrez, 2005).

The aim of this study is to obtain a low cost, effective electrochemical method to remove colour from synthetic dyeing wastewater using different degree of factors (current density, electrolyte concentration, initial dye concentration and time). This study focuses on the electrochemical decoloration of dye using cast-iron electrodes.

Electrochemical treatment is an important technique used in water and water waste industry for the removal of colour and other organic pollutants. An optimum combination of influencing factors (current density, initial dye concentration, electrolyte concentration and reaction time) is to be determined from this study. This

study uses cast-iron electrodes as they have been found to be cheap and effective in the removal of colour (Sheng *et al*., 1997; Rajesh, 2002; Rajesh *et al*., 2003). Without ignoring the importance of electrodes and factors in electrochemical treatment, the optimum usage of current density, electrolyte concentration and time will be proposed by the end of this study to treat the different dye concentration of wastewater.

The removal of colour from wastewater is a persistent problem. There is still a lot of room for improvement in wastewater treatment. Researchers are still on the go to search for the most effective method.

1.2 Objectives of the Study

The main objectives of this study are:

1. To determine the efficiency of electrochemical treatment for textile dye removal from wastewater.

2. To determine the optimum current density, initial dye concentration, electrolyte concentration and reaction time in colour removal from wastewater.

3. To treat the textile dye from wastewater in an environmentally friendly way.

1.3 Scope of Study

This study is focused on the use of cast iron electrodes and combination of different levels of current density, electrolyte concentration, dye concentration and time in the removal of colour from wastewater. The scope of the study is as follows:

1. Study is carried out with decolourisation time varying from 20 minutes to 60 minutes.
2. Current density of 1 mA/cm^2 to 3 mA/cm^2 was tested for its efficiency in colour removal.
3. The concentration of electrolyte (NaCl) was 0.6 g/l, 0.8 g/l, 1.0 g/l.
4. The concentration of synthetic textile dye wastewater was 20 mg/l, 60 mg/l, 100 mg/l.
5. The effective area of anode and cathode was 64 cm^2 each.
6. The sample of wastewater for each experiment was 500 ml.
7. The initial pH of the solution for each experiment was set at 7 using dilute sodium hydroxide or dilute sulfuric acid.

CHAPTER 2

LITERATURE REVIEW

This chapter consists mainly of two parts. The first section addresses the general overview of dyes, classification of dyes and characteristics of dyes. Meanwhile the second section reviews published methods of coloured wastewater treatment including physical treatment, biological treatment, chemical treatment and physical-chemical treatment.

2.1 Dyes

2.1.1 General

Generally, a dye can be described as a coloured substance that has affinity to the substrate to which it is being applied. Archaeological evidence shows that dyeing has been carried out for more than 5000 years, particular in India and the Middle East. The first synthetic textile colorant was produced in the 1860´s when Perkin oxidized aniline to produce Mauvine.

Synthetic dyes have been extensively used in many industries such as textile, leather tanning, paper production, food technology, photoelectrochemical cells, hair colorings etc. Although the exact number and amount of dyes produced in the world is not known, it is estimated that more than 100,000 dyes are commercially available with

over 7×10^5 tons of dye-stuff produced annually (Lee *et al.*, 2005).

However, it has to be emphasized that the overwhelming majority of synthetic dyes currently used are the highly water soluble azo-reactive dyes. Azo dyes are characterized by the existence of nitrogen-nitrogen double bonds and the presence of bright colour is due to these azo bonds and associated chromospheres. Even the presence of very low concentrations of dyes (less than 1 mg/l) in the effluent is highly visible and is considered undesirable. These reactive dyes are the most problematic compared to other forms of dyes and must be removed from wastewater completely. (Lee *et al.,* 2005).

Apart from being aesthetically displeasing these synthetic dyes cause considerable environmental pollution, are toxic to some aquatic organisms and are of serious health risk to human beings. They lead to greater public concern and present legislation problem. Thus, removal of these dyes from wastewater is a major environmental challenge and there is a constant need to have an effective process that can efficiently remove these dyes (Lee *et al.,* 2005).

2.1.2 Classification of dyes

Nowadays, dyes used by the textile industry are largely synthetic and are derived from coal tar and petroleum-based intermediates. The primary classification of dyes is based on the fibers to which they can be applied, and the chemical nature of each dye determines the fibers for which the dye has affinity (Yang and Jared, 2005).

It is advantageous to consider the classification of dyes by use or method of application before considering chemical structures in detail because of the dye nomenclature and jargon that arises from this system. Classification by usage or application is the principal system adopted by the Colour Index (Table 2.1). Because the most important textile fibers are cotton and polyester, the most important dye types are those used for dyeing these two fibers, including polyester-cotton blends. Other textile fibers include nylon, polyacrylonitrile, and cellulose acetate (Hunger, 2003).

Table 2.1 Usage classification of dyes

Class	Principal substrates	Method of application	Chemical types
Acid	Nylon, wool, silk, paper, inks, and leather	Usually from neutral to acidic dyebaths	Azo (including premetallized), anthraquinone, triphenylmethane, azine, xanthene, nitro and nitroso
Azoic components and compositions	Cotton, rayon, cellulose acetate and polyester	Fiber impregnated with coupling component and treated with a solution of stabilized diazonium salt	Azo
Basic	Paper, polyacrylonitrile, modified nylon, polyester and inks	Applied from acidic dyebaths	Cyanine, hemicyanine, diazahemicyanine, diphenylmethane, triarylmethane, azo, azine, xanthene, acridine, oxazine, and anthraquinone

From Hunger (2003)

Table 2.1 Usage classification of dyes (continued)

Class	Principal substrates	Method of application	Chemical types
Direct	Cotton, rayon, paper, leather and nylon.	Applied from neutral or slightly alkaline baths containing additional electrolyte.	Azo, phthalocyanine, stilbene, and oxazine.
Disperse	Polyester, polyamide, acetate, acrylic and plastics	Fine aqueous dispersions often applied by high temperature/pressure or lower temperature carrier methods; dye may be padded on cloth and baked on or thermofixed.	Azo, anthraquinone, styryl, nitro, and benzodifuranone.
Fluorescent brighteners	Soaps and detergents, all fibers, oils, paints, and plastics	From solution, dispersion or suspension in a mass	Stilbene, pyrazoles, coumarin, and naphthalimides

From Hunger (2003)

Table 2.1 Usage classification of dyes (continued)

Class	Principal substrates	Method of application	Chemical types
Food, drug, and cosmetic	Foods, drugs, and cosmetics		Azo, anthraquinone, carotenoid and triarylmethane.
Mordant	Wool, leather, and anodized aluminium	Applied in conjunction with Cr salts	Azo and anthraquinone
Oxidation bases	Hair, fur, and cotton	Aromatic amines and phenols oxidized on the substrate	Aniline black and indeterminate structures
Reactive	Cotton, wool, silk, and nylon.	Reactive site on dye reacts with functional group on fiber to bind dye covalently under influence of heat and pH (alkaline).	Azo, anthraquinone, phthalocyanine, formazan, oxazine, and basic.

From Hunger (2003)

Table 2.1 Usage classification of dyes (continued)

Class	Principal substrates	Method of application	Chemical types
Solvent	Plastics, gasoline, varnishes, lacquers, stains, inks, fats, oils, and waxes.	Dissolution in the substrate	Azo, triphenylmethane, anthraquinone, and phthalocyanine.
Sulfur	Cotton and rayon	Aromatic substrate vatted with sodium sulfide and reoxidized to insoluble sulfur-containing products on fiber.	Indeterminate structures.
Vat	Cotton, rayon, and wool.	Water-insoluble dyes solubilized by reducing with sodium hydrogensulfite, then exhausted on fiber and reoxidized.	Anthraquinone (including polycyclic quinines) and indigoids.

From Hunger (2003)

2.1.3 Characteristics of dyes

2.1.3.1 Acid dyes

Acid dyes are water-soluble anionic compounds applied to nylon, wool, silk, and some modified acrylic textiles in an acidic medium (Yang and Jared, 2005). They are called acid dyes because they are normally applied to the nitrogenous fibers of fabrics in organic or inorganic acid solutions. Chemical reactions between the dye and fiber form an insoluble colour molecule on the fiber (Kulkarni *et al.,* 1985).

They have one or more sulfonic or carboxylic acid groups in their molecular structure. The dye-fiber affinity is the result of ionic bonds between the sulfonic acid part of the dye and the basic amino groups in wool, silk, and nylon fibers (Yang and Jared, 2005). Although acid dyes may be used for transfer printing, this use is not commercially important. In general, these dyes have poor wet fastness. Molecular weights range from 200 to 900, and the higher the molecular weight, the poorer the leveling (uniformity of dye uptake) property (Kulkarni *et al.,* 1985).

2.1.3.2 Disperse dyes

Disperse dyes have a very low water solubility, so they are applied as a dispersion of finely ground powders in the dyebath. The dyes dissolve at low concentration in the aqueous dyeing bath but transfer into the synthetic fiber polymer because of their

higher solubility in the substrate. High temperatures and pressures are consistently used for application. Disperse dyes are used for oleophylic fibers and polyester and other synthetics that do not accept water-soluble dyes (Yang and Jared, 2005).

2.1.3.3 Direct Dyes

These water-soluble anionic dyes, when dyed from aqueous solution in the presence of electrolytes, are substantive to, i.e, have high affinity for, cellulosic fibers. Their principal use is the dyeing of cotton and regenerated cellulose, paper, leather, and, to a lesser extent, nylon. Most of the dyes in this class are polyazo compounds, along with some stilbenes, phthalocyanines, and oxazines. After treatments, frequently applied to the dyed material to improve washfastness properties, include chelation with salts of metals (usually copper or chromium), and treatment with formaldehyde or a cationic dye-complexing resin (Hunger, 2003).

2.1.3.4 Vat Dyes

These water-insoluble dyes are applied mainly to cellulosic fibers as soluble leuco salts after reduction in an alkaline bath, usually with sodium hydrogensulfite. Following exhaustion onto the fiber, the leuco forms are reoxidized to the insoluble keto forms and then treated, usually by soaping, to redevelop the crystal structure. The principal chemical classes of vat dyes are anthraquinone and indigoid (Hunger, 2003).

2.1.3.5 Sulfur Dyes

Sulfur dyes are used primarily for cotton and rayon. The application of sulfur dyes requires carefully planned transformations between the water-soluble reduced state of the dye and the insoluble oxidized form. Sulfur dyes can be applied in both batch and continuous processes; continuous applications are preferred because of the lower volume of dye required (Kulkarni *et al.,* 1985). These dyes are applied to cotton from an alkaline reducing bath with sodium sulfide as the reducing agent. Numerically this is a relatively small group of dyes. The low cost and good washfastness properties of the dyeings make this class important from an economic standpoint. However, they are under pressure from an environmental viewpoint (Hunger, 2003).

These dyes generally have a poor resistance to chlorine. In general, sulfur blacks are the most commercially important colours and are used where good colour fastness is more important than shade brightness. Sulfur dyes are not applicable to wool or silk because the fibers are chemically damaged by the dyeing process (Kulkarni *et al.,* 1985).

2.1.3.6 Cationic (Basic) Dyes

These water-soluble cationic dyes are applied to paper, polyacrylonitrile (e.g. Dralon), modified nylons, and modified polyesters. Their original use was for silk, wool, and tannin-mordanted cotton when brightness of shade was more important than fastness to light and washing. Basic dyes are water-soluble and yield colored cations in solution. For this reason they are frequently referred to as cationic dyes. The principal chemical classes are diazahemicyanine, triarylmethane, cyanine, hemicyanine, thiazine, oxazine, and acridine. Some basic dyes show biological activity and are used in medicine as antiseptics (Hunger, 2003).

2.1.3.7 Solvent Dyes

These water-insoluble but solvent-soluble dyes are devoid of polar solubilizing groups such as sulfonic acid, carboxylic acid, or quaternary ammonium. They are used for coloring plastics, gasoline, oils, and waxes. The dyes are predominantly azo and anthraquinone, but phthalocyanine and triarylmethane dyes are also used (Hunger, 2003).

2.1.3.8 Reactive Dyes

Reactive dyes are extensively used in the textile industry, fundamentally due to the capacity of their reactive groups to bind on textile fibers by covalent bonds formation. This characteristic facilitates the interaction with the fiber and reduces energy consumption. Reactive dyes represent an important fraction of the commercialized synthetic pigments used (approximately 12% of the worldwide production). The major environmental problem associated with the use of reactive dyes is their loss in the dyeing process. Frequently, the fixation efficiency range between 60 % and 90 %; consequently, substantial amounts of unfixed dyes are released in the wastewater. In the particular case of the Reactive Blue 19, the relatively low fixation efficiency (75-80 %) is due to the competition between the formation of the reactive form (vinyl sulfone) and the hydrolysis reactions (Appendix A 1). The latter reaction leads to the formation of 2-hidroxyethylsulfone which does not fix on the fiber (Ronaldo *et al.,* 1999). A General structure of reactive dye is shown in (Appendix A 2) (Edyta and Edward, 2003).

Reactive dyes are water-soluble, anionic dyes that require relatively simple dyeing methods. They are mainly used for dyeing cellulosic fibers, such as cotton and rayon, but are also used for silk, wool, nylon, and leather. Reactive dyes have largely replaced direct, azoic, and vat dyes and are the largest dye class in the United States (Yang and Jared, 2005). This class of dyes, first introduced commercially in 1956 by

Dulux, made it possible to achieve extremely high washfastness properties by relatively simple dyeing methods. A marked advantage of reactive dyes over direct dyes is that their chemical structures are much simpler, their adsorption spectra show narrower absorption bands, and the dyeings are brighter. The principal chemical classes of reactive dyes are azo (including metallized azo), triphendioxazine, phthalocyanine, formazan, and anthraquinone. High-purity reactive dyes are used in the ink-jet printing of textiles, especially cotton (Hunger, 2003). The dyes form covalent bonds with the fiber and become part of the fiber. To exhaust the dyes, large amounts of salt are generally essential, and significant amounts of dye can remain unfixed at end of the process (Yang and Jared, 2005).

The high consumption of reactive dyes, mainly in the cotton industry, increases this environmental and aesthetic problem, due to their low degree of exhaustion (60-90 %). Therefore, a significant percentage of the dye (10-40 %) remains in its hydrolysed and unfixed form in the exhausted dyebath or is removed in the washing liquors (Lopez and Gutierrez, 2005).

Reactive dyes, as well as many other textile dyes, are only partially removed under the aerobic conditions of the conventional biological treatment, mainly by adsorption on the activated sludge. As biological treatment is insufficient to remove the colour and to accomplish with current regulations, the application of specific treatment is required (Lopez and Gutierrez, 2005).

2.2   Treatment Methods for Colour Removal

2.2.1 Physical Treatment

2.2.1.1 Adsorption

Adsorption processes have been applied for concentrating organic compounds from industrial effluents for years. The most widely used adsorbent is granular activated carbon (GAC). GAC has been proven to be effective for the removal of colour from textile and pulp and paper effluents (Yang and Jared, 2005).

However, adsorption on activated carbon (C*) without pretreatment is impossible because the suspended solids rapidly clog the filter. This procedure is therefore only feasible when combined with a flocculation–decantation treatment or a biological treatment. This combination permits a reduction of suspended solids and organic substances as well as a slight influence on the color but the cost of activated carbon is still high (Allegre *et al.,* 2005).

Due to the high capital, regeneration, and disposal costs, full scale GAC adsorption systems for effluent decolorization have yet to be accepted by the industry. An adsorption capacity of 1 g/g makes this material a candidate for further development. However high pH and salt concentrations may have an adverse effect on the adsorption (Yang and Jared, 2005).

2.2.1.2 Membrane processes

The increasing cost of water and its wasteful consumption have now induced a treatment process which is integrated in in-plant water circuits rather than as a subsequent treatment. From this standpoint, membrane filtration offers potential applications. Processes using membranes provide very interesting possibilities of separating hydrolyzed dyestuffs and dyeing auxiliaries, thus simultaneously reducing coloration and the BOD/COD of the wastewater. Some particular membrane process (MF, UF, NF and RO), are as described below (Allegre *et al.,* 2005).

(a) Microfiltration (MF)

Microfiltration is suitable for treating dye baths containing pigment dyes as well as subsequent rinsing baths. The auxiliaries remain in the retentate. Microfiltration can also be used as a pretreatment for nanofiltration or reverse osmosis (Allegre *et al.,* 2005).

(b) Ultrafiltration (UF)

Ultrafiltration enables the elimination of macromolecules and particles but the elimination of polluting substances, such as color is never complete (between 31 % and 76 %). And even in the best of cases, the quality of the treated wastewater does

not permit its reuse for feeding sensitive processes, such as the dyeing of textile. Rott and Minke (1999), emphasize that 40 % of the water treated by ultrafiltration can be recycled to feed processes termed "minor" in the textile industry (rinsing, washing) in which salinity is not a problem. Ultrafiltration can only be used as a pretreatment for reverse osmosis or in combination with a biological reactor (Allegre *et al.,* 2005).

(c) Nanofiltration

Nanofiltration membranes retain organic compounds of low molecular weight, divalent ions or large monovalent ions, such as hydrolyzed reactive dyes as well as dyeing auxiliaries. The effect of the concentration of dyes has been frequently reported in dye house effluents as well as the concentration of salt and the pressure. In most published studies concerning dye house effluents, the concentration of mineral salts does not exceed 20 g L^{-1} and the concentration of dyestuff 1.5 g L^{-1}. The effluents are reconstituted with generally only one dye and the volume studied is low. The treatment of dyeing wastewater by nanofiltration thus represents one of the rate applications possible for the treatment of solutions with highly concentrated and complex solutions (Allegre *et al.,* 2005).

(d) Reverse osmosis

Reverse osmosis membranes have a retention rate of 90 % or more for most types of

ionic compounds and produce a high quality of permeate. Decolouration and the elimination of chemical auxiliaries in dye house wastewater can be carried out in a single step. Reverse osmosis permits the removal of all mineral salts, hydrolyzed reactive dyes and chemical auxiliaries. The problem involved is that the higher the concentration of salt, the more important the osmotic pressure becomes and therefore the greater the energy required (Allegre *et al.,* 2005).

2.2.2   Biological treatment

Biological treatments reproduce, artificially or otherwise, the phenomena of self-purification that exist in nature. Self-purification comprises the whole process by which an aquatic environment achieves the re-establishment of its original quality after pollution. Biological treatments differ according to the presence or absence of oxygen (Allegre *et al.,* 2005). Aerobic treatment of industrial effluents typically removes most of the biochemical oxygen demand (BOD) and 60 % to 80 % of COD in wastewater. However, these treatments are ineffective for colour removal from textile industry wastewater because most of the synthetic dyes are non-biodegradable. In anaerobic processes, the azo bonds can be used as electron acceptors in the electron transport pathways. The products of the azo bond cleavage are lower molecular weight aromatic amines. Although they are colourless, they cannot be further degraded under anaerobic conditions. Therefore, various combined processes, such as anaerobic/aerobic, chemical/biological, and electrochemical/biological, processes

have been under intensive study to completely mineralize organic dyes (Yang and Jared, 2005).

2.2.3 Physical chemical treatment

Coagulation–flocculation treatments are generally used to eliminate organic substances. The products normally used have no effect on the elimination of soluble dyestuffs. Even though this process, which is widely used in Germany, makes it possible to effectively eliminate insoluble dyes, it is of doubtful value owing to the cost of treating the sludges and the increasing number of restrictions concerning their disposal (Allegre *et al.,* 2005).

2.2.4 Chemical Treatment

2.2.4.1 Ozonation

Ozone is an attractive and increasingly important method for the degradation of organic pollutants in aqueous solution (Huang *et al.*, 2005). Ozone is now used widely in water treatment, and (either alone or in combination with other treatments, such as O_3-UV or O_3-H_2O_2) in the treatment of industrial effluents. Ozone especially attacks the double bonds which are responsible for coloration. For this reason, decolorization of wastewater by ozone alone is not always accompanied by a significant reduction of the COD and the installation of an ozonation process involves additional costs

(Allegre *et al.,* 2005). However, the refractory organic compounds are usually not totally oxidized and only a small mineralization is achieved. Moreover, the practical use of ozonation for wastewater treatment is limited by its high-energy demand (Huang *et al.*, 2005).

2.2.4.2 Electrochemical method

In recent years, there has been increased interest in the electrochemical methods to decolourise and degrade dye molecules. The electric current induces redox reactions resulting in the transformation/destruction of the organic compounds and their virtually complete oxidation to CO_2 and H_2O. There are different approaches to the electrochemical technique as shown below (Cameselle *et al.,* 2005).

The electrochemical decolourisation could be done by reduction or by oxidation. In the case of reduction, the chromophore of azo dyes is broken to generate two amino groups. In the second case, the indirect oxidation by hypochlorite ions generated in a brine solution using a graphite rod as anode is reported. The electrochemical oxidation of dyes can also be assisted by photocatalysis. During the dyeing process with reactive dyes, the addition of high concentrations of an electrolyte is necessary to obtain a better fixation and exhaustion. Generally, an amount of 50-80 g/l of a salt is added as electrolyte, NaCl or Na_2SO_4 being the most common. This salt also acts as electrolyte during the electrochemical treatment (Lopez and Gutierrez, 2005).