# Performance Evaluation of an Electrocoagulation Reactor in remediating a basic dye (Rhodamine B) from aqueous solution - A laboratory scale study

A thesis submitted for the partial fulfillment of the requirements for the degree of **Master of Engineering** in **Civil Engineering** for the session 2021-2023

## Prepared by

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2023

### RECOMMENDATION CERTIFICATE

It is hereby certified that this thesis titled "Performance" Evaluation of an Electrocoagulation Reactor in remediating a basic dye (Rhodamine B) from aqueous solution - A laboratory **scale study**" is submitted for the partial fulfilment for the degree of Master of Engineering in Civil Engineering with specialization in Environmental Engineering of Jadavpur University by Biswajit Chakraborty (Class Roll No. 002110402013; Registration No. 141026 of 2017-2018; Examination Roll No. M4CIV23003), a student of the said course for the session 2021-2023 under the supervision of **Prof.** Debsarkar, Department of Civil Anupam Engineering (Environmental Engineering Division), Jadavpur university.

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## **DECLARATION**

This thesis entitled "Performance Evaluation of an Electrocoagulation Reactor in remediating a basic dye (Rhodamine B) from aqueous solution - A laboratory scale study" is prepared and submitted for the partial fulfillment of degree of Master of Engineering in Civil Engineering (Specialization in Environment Engineering) of Jadavpur University for the session 2021-2023. It is declared that no part of this said thesis work has been presented or published elsewhere.

Date:

Place: Kolkata

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# **Chapter-1**

# Introduction

Water pollution by dyes is one of the major problems facing worldwide. Effluents from industries like textile, paper and pulp, leather, plastic and food contain different kinds of synthetic dyes. Approximately 12% of synthetic textile dyes used each year is lost during manufacture and processing operation, and 20% of these dyes enter the environment through effluents that result from the treatment of residual industrial waters (Selvarani 2000). The discharge of these highly coloured effluents into natural water bodies is aesthetically displeasing and it also inhibits light penetration that negatively affects the biological processes within a stream (Rajeshkannan et al. 2010). Dyes can pose hazardous effects on the living systems because of its carcinogenicity and toxic nature (Akar et al. 2009). The dyes present in wastewater adversely affect the aqueous ecosystem because the intense color of the aqueous body imparted by dyes inhibits the access of sunlight to the interior of the aqueous body. Due to lack of sunlight, photosynthesis activity gets affected which results in the deficiency of oxygen causing disruption to ecosystem. This problem continuously occurs year by year. Accumulation of dyes on a food chain will offer resistance to biological degradation of decaying process from organism such as bacteria, plankton and fungi (Anwer et al., 2019). Most of the dyes being used in different industries have not been evaluated for their impact on health and the environment. The synthetic dyes are resistant to degradation by conventional wastewater treatment plants and are released into the environment untreated thus causing pollution of surface and ground waters in the areas harboring such industries.

Various treatment processes have been used for the treatment of dyecontaminated wastewater. The biological treatment methods including both aerobic and anaerobic methods are popular as they offer the possibility of energy recovery and are practical and comparatively less costly. However, these biological methods have certain limitations too. For example, they require a long time for operation, and are very sensitive to experimental conditions like accumulation of ammonia, concentration of pollutants, etc. The biological treatment methods fail to treat wastewater containing high dye concentration (Garcia et al. 2020). The physicochemical treatment methods such as adsorption, coagulation, precipitation, reverse osmosis, flocculation and anaerobic oxidation are not effective because these techniques generate secondary pollution due to use of chemicals. Alternatively, the Electrocoagulation (EC) process is considered to be a potential and promising technique for the treatment of dye contaminated wastewater. The EC technology offers an alternative to the use of metal salts or polymers and poly-electrolyte addition for breaking stable emulsions and suspensions. The technology removes metals, colloidal solids and particles and soluble inorganic pollutants from aqueous media by introducing highly charged polymeric metal hydroxide species. These species neutralize the electrostatic charges on suspended solids to facilitate agglomeration or coagulation and resultant separation from the aqueous phase. EC process provides a simple, reliable and cost-effective method for the treatment of wastewater without any need for additional chemicals and thus the chance of secondary pollution is reduced. Electro-coagulation would subsequently generate less solid waste, thus offering savings on disposal costs. The EC process also has the advantage of removing the smallest colloidal particles, because the applied electric field sets them in faster motion, thereby facilitating the coagulation. Electrocoagulation thus deserves a thorough study as a technique for treatment of dye bearing wastewater.

# **Chapter-2**

# **Review of Literature**

Most of the synthetic dye wastes are toxic, harmful to human, animal and plant life. Textile industries consume large volumes of water and chemicals for wet processing of textiles. The chemical reagents used are very diverse in chemical composition, ranging from inorganic compounds to polymers and organic products (Mishra and Tripathy, 1993; Banat et al., 1996). The presence of very low concentrations of dyes in effluent is highly visible and undesirable (Nigam et al., 2000). There are more than 100,000 commercially available dyes. Due to their chemical structure, dyes are resistant to fading on exposure to light, water and many chemicals. Most of the dyes are difficult to decolorize due to their complex structure and synthetic origin. Extensive work has been carried out on methods for treatment of dye bearing wastewater.

#### 2.1 Dyes and its classification

Dyes are the coloured organic substances that are used in solution to impart colour to various substances like fabric, paper, leather, hair, drugs, cosmetics, plastics and food material. It is a substance with a suitable colour and the ability to fix itself or be fixed to the fabric. It must be fast and resistant towards water, diluted acids, alkalis and various organic solvents. Dyes can be natural as well as synthetic. Natural dyes are obtained from natural sources like plants (including fungi and lichens), animals, invertebrates or minerals. Some of the examples of natural dyes are Indigo dyes (from stems and leaves of indigo), Alizarin dyes (from roots of madder plant), Logwoods dyes (from the trunk for black colour to silk and cotton fabrics) etc. These dyes are not that much harmful in nature. Synthetic dyes are prepared in labs/factories for dyeing purposes. Examples of synthetic dyes are mordant dyes, azo dyes etc. Most of the dyes used today are synthetic because they are easy to use, cheaper, brighter, fast and have wider range of colours. These synthetic dyes are toxic in nature. If it is present in water sources, it can cause several health hazards for human beings.

A dye molecule has two main components:

#### (i) Chromophores, and

#### (ii) Auxochromes.

Chromophores are responsible for colour while auxochromes facilitate attachment towards fibers. Based on chromophores and auxochrome the dyes are nitro and nitroso dye, azo dye, triarylmethane dye, anthraguinone dye, Indigo dyes etc. In nitro and nitroso dye contain nitro group (-NO<sub>2</sub>) and nitroso group (-NO) as the chromophores respectively and hydroxyl group (-OH) as auxochrome. One of the examples of nitro dye is Naphthol yellow S and nitroso dye is Gambine-Y. In azo dyes the Azo group (-N=N-) is present as primary chromophore in their molecular structure which is present between two aromatic rings. Tartrazine (yellow), Methyl Orange, Congo Red, Bismarck Brown, Chrysoidine and other azo dyes are examples. Azo dyes account for 60-70% of dyes used in food and textile production. A central carbon is bonded to three aromatic rings in triarylmethane dyes, one of which is guinoid. The examples of triarymethane dyes are Malachite Green (used as a direct dye for wool and silk), Crystal violet, and Phenolphthalein etc. In Anthraguinone dye Anthraguinone ring is the chromophore and -OH, -SO<sub>3</sub>H, -NH<sub>2</sub> as auxochrome. One of the examples of this dye is Alizarin-red textile dye. Indigo dyes have carbonyl chromospheres and indigoid structure (-CO=C=C-CO-). These dyes are used in cotton yarn, which is primarily used to make blue jeans denim cloth.

Dyes can be classified in three broad categories:

- i) cationic (basic dyes)
- ii) anionic (direct, acid and reactive dyes) and
- iii) non-ionic (disperse dyes and vat dyes).

#### 2.1.1 Cationic dyes

Cationic dyes contain a cationic functional group that may dissolve into positively charged ions in an aqueous solution. The onium group is the most common cationic functional group. Methylene Blue (MB), Rhodamine B (RhB), Rhodamine 6G (Rh6G), Safranin O (SO), Crystal Violet (CV), and Malachite Green (MG) are some examples of cationic dyes, with most cations being N+. As a result, cationic dyes are also called basic dyes. RhB is a cationic dye that is widely used in

the textile, leather, and food industries. Because of its higher toxic colourants, RhB is carcinogenic and mutagenic to aquatic wildlife and humans. The chemical properties of some cationic dyes are given in Table 2.1.

Table 2.1: Chemical properties of some Cationic Dyes (Chiu et al., 2019)

Dyes	Abbreviation	Molecular Weight (g/mole)	Structure	λ max
Methylene Blue	МВ	799.81	H <sub>3</sub> C N-CH <sub>3</sub>	664
Rhodamine B	RhB	479.02	H <sub>3</sub> C	554
Rhodamine 6G	Rh6G	479.02	H <sub>3</sub> C H <sub>3</sub> CH <sub>3</sub> CH <sub>3</sub>	534
Malachite Green	MG	364.91	CH <sub>3</sub> CH <sub>3</sub> CH <sub>3</sub> N CH <sub>3</sub>	614
Safranin O	SO	350.85	H <sub>3</sub> C CH <sub>3</sub> N+ NH <sub>2</sub> CI	520

Crystal Violate	CV	407.98	H <sub>3</sub> C-N  CI  CH <sub>3</sub> CH <sub>3</sub> CH <sub>3</sub> CH <sub>3</sub> CH <sub>3</sub>	573
Victoria Blue B	VB B	506.08	H <sub>3</sub> C N-CH <sub>3</sub> Cr Cr CH <sub>3</sub>	614
Auramine O	AO	303.83	H <sub>3</sub> C <sub>N</sub> CH <sub>3</sub> CH <sub>3</sub> CH <sub>3</sub>	420

#### 2.1.2 Anionic Dyes

Anionic dyes contain anionic functional groups (e.g., sulfonic or carboxylic acid groups) that are water soluble and may interact easily with photocatalysts with hydrophilic surfaces. As a result, anionic dyes are also referred as acidic dyes. Some of the anionic dyes are Acid Orange 7 (AO7), Eosin Y (EY), Methyl Orange (MO), Acid Red 14 (AR14), Alizarin Red S (ARS), Rose Bengal (RB), and Phenol Red (PR). The chemical properties of some anionic dyes are given in Table 2.2.

Table 2.2: Chemical properties of some Anionic Dyes (Chiu et al., 2019)

Dyes	Abbreviation	Molecular Weight (g/mole)	Structure	λ max
Methyl Orange	МО	327.33	O	464
Eosin Y	EY	691.85	Br O Br O Na <sup>+</sup>	518
Acid Orange 7	AO7	350.32	N N N N N N N N N N N N N N N N N N N	484
Acid Red	AR14	502.43	OH Na <sup>+</sup> O Na <sup>+</sup>	515
Alizarin Red S	ARS	240.21	O O Na <sup>+</sup> O OH	426
Rose Bengal	RB	973.67	CI CI CI CI CI Na <sup>+</sup>	550

			<del>-</del>	
Phenol Red	PR	354.38	Na <sup>+</sup> O O OH	560
Congo Red	CR	696.67	NH <sub>2</sub> O=S=O NN <sub>N</sub> NH <sub>2</sub> NH <sub>2</sub> NH <sub>2</sub> NH <sub>2</sub>	497
Acid Violet 7	AV7	566.47	H <sub>3</sub> C H <sub>N</sub> H <sub>O</sub> N <sub>a</sub> +	522
Reactive Black 5	RB5	991.82	Na*  Na*  Na*  Na*  Na*  Na*	602

#### 2.1.3 Non-ionic Dyes

Non-ionic Dyes are basically of two types.

#### i) Disperse dyes and ii) Vat dyes

#### 2.1.3.1 Disperse Dyes

Disperse dye is described as a substantially water-insoluble dye associated with one or more than one hydrophobic nature of fibers, such as polyester or cellulose acetate. Being non-ionic, it is easily volatile and vapors of dye are highly absorbed by the hydrophobic fiber. Some examples of dispersed dyes are C.I. Disperse Yellow 13 which is suitable for acetates and nylon, C.I. Disperse Violet 1 which is suitable for acetate, nylon and polyester, C.I. Disperse Blue 56 which is

suitable for dyeing polyester fibers, C.I. Disperse Orange 30 is applied on acetate and triacetate, C.I. Disperse Blue 183 for dying of polyester. The chemical structure of some dispersed dyes is shown in Fig. 2.1 (Clark, 2011).

Fig. 2.1: Chemical Structures of some Disperse Dyes

#### **2.1.3.2** Vat Dye

Vat dye is identified for better colour fastness and excellent brightness properties (Burkinshaw & Son, 2010). They are mainly soluble in hot water. Some vat dyes are also soluble in sodium carbonate. The most important natural vat dye is Indigo or indigotin obtained from the plant Indigofera. Some examples of the vat dyes are C.I. Vat Blue 1, C.I. Vat Black 25, C.I. Vat Green 1. The chemical structure of some vat dyes is shown in Fig. 2.2 (Sharma et al., 2021).

Fig. 2.2: Chemical Structures of some Vat Dyes

#### **2.1.3.3 Sulfur dyes**

Sulphur dyes are highly coloured, water insoluble compounds. They are the most commonly used dyes manufactured for cotton in terms of volume. They are

predominantly black, brown, and dark blue. These dyes can be categorized into four groups such as sulfur, Leuco sulfur, solubilized and condensed sulfur dyes. Some of the sulfur dyes are C.I. Sulfur Blue 15, C.I. Leuco Sulfur Black1 and C.I. Sulfur Green 3. The chemical structure of some sulfur dyes are shown in Fig. 2.3 (Sharma et al., 2021).

Fig 2.3: Chemical Structures of some Sulfur dyes

#### 2.2 Harmful effects of dyes

During the colouration process, a huge amount of dye does not bind to the fabric even after many cycles of fabrication. The remaining un-useful dye is discharged over the earth's surface and into natural water bodies like rivers and lakes, etc. The long exposure of the dyes can cause health hazards to the human being and they must be treated before release to the environment. Exposure of dyes can cause breathing problems due to the inhalation of the dye molecules which affect the immune system of the human. Other symptoms like itching, sore eyes, sneezing, coughing, and wheezing can also be the results of the impact of dye. On the other hand, in comparison with the other dyes, the anionic and cationic dyes can easily penetrate living body cells and can cause carcinogenic effects even at the low concentration.

Rhodamine B (RhB) is widely used as a colorant in textile industries. It is also utilized in the manufacturing of numerous products such as ball pens, paints, leather, dye lasers, carbon sheets, stamp pad inks, crackers, and explosives It is a weakly basic nitrogenous molecule that is undergoing dissociation and produces a highly stable and non-biodegradable colorful cation. These colorful cations enter the surface and ground water reservoirs and cause significant alterations in the aquatic ecosystem. The widespread presence of Rhodamine B in the aquatic environment could be hazardous to both human and animal health. It causes mutagenic and oncogenic variations in living things. It is also categorized as a neurotoxic dye in

both humans and animals, causing infections of the eyes, skin, gastrointestinal tract, and respiratory tract. It's Predicted No Effect Concentration (PNEC) was 14  $\mu$ g/L in an aquatic organism based on ecotoxicological research. It is the lowest value for Rhodamine B's aquatic ecotoxicity that has been documented. The so-called Annual Average Quality Standard (AA-QS), the Environmental Quality Standard (EQS) for longer-term exposure, can be simply computed from these PNEC values. The AA-QS for RhB will be 14  $\mu$ g/L, which is equal to the PNEC. The Maximum Acceptable Concentration (MAC-QS) for RhB is 140  $\mu$ g/L. RhB is frequently employed as a systemic marker in several animals due to its high level of persistence. One study found that detectable quantities of RhB occurred in the Myocastor coypus for up to 225 days after feeding them 0.5 g/kg of the dye for three days. RhB is difficult to remove because of its persistence, which raises the possibility that it can cause several problems (Sharma et al., 2022).

Textile industries usually release a large amount of Methylene Blue (MB) dyes in natural water sources, which becomes a health threat to human beings and microbes. MB dye is harmful to human health above a certain concentration due to its substantial toxicity. MB is toxic, carcinogenic, non-biodegradable and can cause a serious threat to human health and destructive effects on the environment. MB causes several risks to human health such as respiratory distress, abdominal disorders, blindness, and digestive and mental disorders. It also causes nausea, diarrhoea, vomiting, cyanosis, shock, gastritis, jaundice, methemoglobinemia, tissue necrosis, and increased heart rate, causing the death of premature cells in tissues and skin/eye irritations. MB contacts with skin may result in skin redness and itching. The no observed adverse effect level (NOAEL) for the MB in rats was observed to be 25 mg/kg. MB discharge into the environment is a significant threat for aesthetical and toxicological reasons. It also reduces light penetration and is a toxic supply to food chains for organisms. MB presence in water bodies, even at a very low concentration, makes highly coloured sub-products. Owing to its high molar absorption coefficient ( $\sim 8.4 \times 10^4$  L/mol/cm at 664 nm), which reduces sunlight transmittance, it decreases oxygen solubility, affects the photosynthetic activity of aquatic life, and decreases the diversity and aesthetics of the biological community (Sarkar Phyllis et al., 2022).

Methyl Orange (MO) is a carcinogenic water-soluble azo dye, and also acknowledged as an acidic or anionic dye. It can cause vomiting and diarrhea. High levels of exposure to MO can results in death. It is also metabolized into aromatic amines by intestinal microorganisms. Methyl orange is stable, shows low biodegradability and is soluble in water hence it is difficult to remove from aqueous solutions by common water purification or treatment methods.

Congo red (CR) is a diazo dye associated with benzidine which is a cancercausing substance. Therefore, CR is also known as a human carcinogen and toxic compound for animals and plants (Farias et al., 2018). The toxicity of Congo red is primarily connected to its intermediary metabolites, which directly disturbed DNA and produce apoptosis in HL-60 cells (human promyelocytic leukemia cell line)(Hernández-Zamora et al., 2016). Apart from dyes, phenol and its derivatives are also considered toxic to human beings if long term exposed. These cause mainly diarrhea, anorexia, vertigo, progressive weight loss, excessive salivation, dark coloured urine, blood, and liver effects (McCall et al., 2009).

Knowing the bad impact of the dyes and toxic compounds on the human's life, plants, and animals, it is crucial to eliminate the dyes from the wastewater prior to releasing into the environment.

## 2.3 Dye removal methods

The majority of synthetic dyes is poisonous in nature and constitutes a risk to aquatic life. To meet the stringent environmental requirements, numerous methods for removing colours from water and wastewater have been used. These methods are classified in Fig. 2.4.

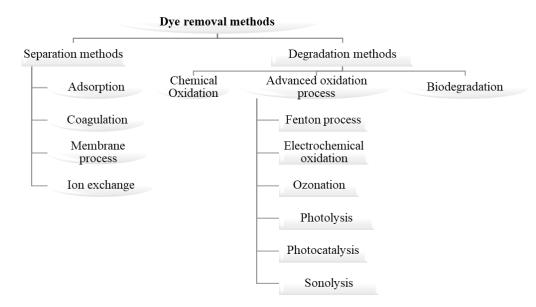


Fig 2.4: Classification of various methods for dye treatment

Some of the techniques for removal of dyes are discussed in the following subsections.

#### 2.3.1 Chemical coagulation & flocculation

Coagulation and flocculation are commonly used as physicochemical procedures for cleaning industrial wastewater because they destabilize and create flocs, which remove colloidal particles, very small solid suspensions, and a few soluble chemicals that were previously present in the wastewater. According to several researchers, the coagulation process has proven to be a more effective approach compared to other processes such as anaerobic reduction, oxidation, and adsorption. It is also one of the simplest and cheapest procedures that may be easily employed in industrial settings (Kos, 2016). Coagulation removes around (70 – 80) % of the colour and reduces the concentration of organic chemicals by about the same amount. The efficacy of this procedure is determined by the qualities of raw wastewater, as well as the temperature and pH of the solution, as well as the type and dosage of coagulants used, and the mixing time. Inorganic salts like alum or ferric chloride, as well as synthetic organic polymers, are common coagulants. Although chemical coagulants are used to remove colours and suspended particles from wastewater, they have several drawbacks, including their impact on humans

by creating diseases like Alzheimer's disease, which is caused by inorganic salts, and some of them are also carcinogenic in nature.

Chemical coagulation is a complex process involving several interconnected characteristics, thus it's crucial to know how well a coagulant will perform under specific conditions. As a coagulant, alum, ferric chloride, ferric sulphate, and magnesium chloride can be employed. Destabilization is affected differently by different coagulants. The higher the counter ion's valence, the more destabilizing it is and the lower the required coagulation dose. Positively charged polymers will prevail when the pH is below the isoelectric point of metal hydroxide and colloids are precipitated by various coagulants supported by a suitable polymer. Adsorption of these positively charged polymers can destabilize negatively charged colloids by charge neutralization. Anionic polymers will predominate above the isoelectric point when particle instability may occur due to adsorption and bridge formation. When a large dose of metal ions (coagulant) is used, a sufficient degree of oversaturation occurs, resulting in the rapid precipitation of a large amount of metal hydroxide, enmeshing the colloidal particles known as sweep flocs. Monomeric and polymeric ferric species are produced when Fe (III) salts are used as coagulants, with the generation of monomeric and polymeric ferric species being pH dependent. According to several studies, the natural colour of ferric chloride solution is acidic, but successful removal can only be done when the pH is close to neutral, therefore adding a base to keep the pH stable becomes a must. Lime or sodium hydroxide (NaOH) might be used for this. Lime, on the other hand, may result in more sludge. The use of polyelectrolyte as a coagulant aid, on the other hand, often improves the coagulant's performance. As can be observed, the ideal pH for alum is close to neutral, allowing for greater colour removal efficiency. Furthermore, the inclusion of polyelectrolyte improves the colour removal performance in general. The vast amount of sludge generated by this technique, however, makes it unappealing. Magnesium chloride's optimal pH ranges from 9 to 12. When used with lime, it provides excellent colour removal. However, it produces a huge amount of sludge, which may provide a sludge disposal concern as well as additional costs. Because of the enormous volume of sludge generated and the formation of basic effluent after treatment, alum and magnesium chloride may not be regarded good coagulants. Though both ferric chloride and alum have a high efficiency, ferric chloride has

lower colour removal effectiveness at low concentrations. However, when ferric chloride is combined with a little amount of cationic polymer, a considerable improvement in colour removal has been recorded.

#### 2.3.2 Membrane process (Reverse Osmosis (RO) & Nano Filtration (NF))

Membranes are widely used in a variety of separation processes because of their ability to control materials passing through the membrane, resulting in a high degree of separation that is always achieved, making these processes widely acceptable. The membrane is a barrier that allows some substances to pass through it (permeate) while preventing others from doing so (retentate) in a very specific way. Membranes can be used to separate fluids, dissolved solids, suspended solids, and colloidal dissolved solids. The ability to remove or recover valuable or harmful components, as well as the ability to close water systems, which reduces the consumption of fresh water, are the main characteristics of membrane processes in the treatment of consumables. The use of membrane processes allows wastewater to be purified to a level that is difficult to achieve with traditional methods. Reverse Osmosis (RO) and Nano Filtration are the most widely used and significant membrane filtration techniques (NF). The operating pressure of NF is between 5 and 40 bars, and the membrane pore size is between 0.5 and 2 nm. On the one hand, it's used to separate sugars, other organic molecules, and multivalent salts from monovalent salts, ions, and water, and on the other hand, it's used to separate monovalent salts, ions, and water. In RO or hyper filtration, the membrane pore size is in the range of 0.5 nm. Operating pressures in RO are usually between 7 and 100 bars. The importance of membrane processes is demonstrated by the membrane areas installed in various industrial sectors. RO membranes are a popular choice for treating contaminated drinking water supplies because of their ability to remove both organic and inorganic compounds. Hardness, colour, a variety of bacteria and viruses, as well as organic contaminants like agricultural chemicals and trihalomethane precursors, can all be removed simultaneously using reverse osmosis. Avlonitis et al. looked at effluents from the cotton textile industry that were treated with a nanofiltration membrane to reduce the amount of water that was discarded while also allowing the treated water to be reused (Avlonitis et al.,

2008). The results showed that NF membranes were capable of completely decolorizing the cotton dye effluent and reducing the total salt concentration by more than 72%. Even at high recoveries and low pressures, these membranes can produce high-quality water that can be reused. Gozálvez-Zafrilla et al. (2008) looked into the treatment of secondary effluent for wastewater reuse in the textile industry. They used (NF90) membrane in their research. The results showed that NF90 had the highest salt rejection and a 99% COD reduction (75-95%). The levels of COD removal and salt rejection were unaffected by fouling, and the permeate quality allowed for a high flux percentage to be recovered after cleaning. Abid et al. used the RO and NF process to test acid red, reactive black, and reactive blue dye (Abid et al., 2012). They discovered that RO and NF membranes can effectively remove dye from wastewater, and that dye removal was proportional to applied pressure, pH, TDS, and dye concentration in the feed solution, but not to feed temperature.

#### 2.3.3 Biological process

The biological process employs microorganisms such as fungi, bacteria, and algae, which are capable of biodegrading and absorbing dyes found in wastewater. The use of microorganisms to remove dyes from wastewater has several advantages, including a low cost, an environmentally friendly process that produces less secondary sludge, and nontoxic final products for complete mineralization. Many studies have been conducted that show the ability of microorganisms like Cunninghamellaelegans, Aspergillusniger, Bacillus cereus, Chlorella Spand, and Citrobacter sp. to remove dye from industrial wastewater. The most important factors influencing the decolourization effectiveness of microbial activity are the adaptability and activity of each microorganism. Colour is removed using biological treatment, but because most dyes are designed to resist light and oxidation degradation, they are less likely to be treated with traditional aerobic therapy. The removal of water-soluble dyes by aerobic processes is a particular problem. On wastewater sludge, some of these dyes are adsorbed. Direct biological treatment with bacteria or fungi is also possible, but the nutritional and physiological requirements of microorganisms limit the applicability of these bioremediation methods. The use of enzymes to replace traditional non-biological methods has gained popularity as a result of research into effective and environmentally friendly oxidation techniques. Biological treatment methods have proven to be effective in reducing dye house effluents, and when used properly, they are less expensive to operate than other methods. In the water treatment process, which combines chemical and biological or physical and biological, have also been found to be effective. The biological treatment of textile azo dyes has been shown to be an effective method for degrading all dye materials while also overcoming many of the disadvantages of physicochemical processes. Both aerobic and anaerobic metabolism can be used by microbes and their enzymes to degrade dye. Several studies have been published on the degradation of environmental contaminants by different bacteria. Many bacteria have been identified as relying solely on hydrocarbons. Bacteria with hydrocarbon-degrading bacteria can degrade hydrocarbons. Because they are biodegradable and easy to maintain, these bacterial bio flocculants provide an affordable and clean alternative for replacing or supplementing current treatment processes to remove dyes from wastewater effluents. Daneshvar et al. looked into the ability of microalgae Cosmarium species to decolourize a solution containing Malachite Green, a cationic textile dye (Daneshvar et al., 2007). They looked at algal stability and reusability during repeated decolourization operations, as well as the relationship between kinetic properties and dye concentration and other rate-dependent environmental variables (temperature, pH, dye concentration, and algal concentration).

#### 2.3.4 Adsorption with conventional adsorbent (activated carbon)

Adsorption is the method of choice and produces the best results among the many dye removal techniques available because it can be used to remove a variety of colouring materials. If the adsorption system is properly designed, the treated effluent will be of high quality. Depending on the nature of the forces involved, adsorption processes can be classified as either physical or chemical. Adsorbate/adsorbent interaction, adsorbent surface area and pore structure, surface chemistry, nature of the adsorbate, effect of other ions, particle size, pH, temperature, contact time, and other physico-chemical factors all influence the adsorption process. In comparison to other techniques such as coagulation,

flocculation, precipitation, and activated sludge, the adsorption technique has proven to be more effective and convenient due to its low cost, simple design, easy handling, and sludge-free cleaning operations. Adsorption processes are a viable treatment option, particularly when the adsorbent is cheap and readily available. Activated carbon is the most widely used adsorbent among commercial adsorbents. The use of activated carbon as a dye removal technique has been highlighted as a viable option. Activated carbon has an extremely high affinity for many types of dyes, including basic dyes, due to its unique molecular structure. Because of its excellent adsorption ability, activated carbon is currently used as a sorbent in most commercial systems to remove dyes from wastewater. One of the best available control technologies, according to the US Environmental Protection Agency, is activated carbon adsorption. Activated carbon, on the other hand, is a preferred sorbent, but its widespread use is limited due to its high cost. Efforts have been made to find low-cost alternative adsorbents to reduce treatment costs. El Qada et al. investigated activated carbon and compared the adsorption capacity of three different activated carbons (PAC1 and PAC2 developed at QUB for this work, and commercially available Filtrasorb 400) (El Qada et al., 2008).

#### 2.3.5 Adsorption with non-conventional adsorbents (fly ash)

Apart from traditional adsorbents like activated carbon, a variety of approaches to developing cheaper and more effective adsorbents have been investigated. Several researchers have proposed a variety of non-traditional low-cost adsorbents, including natural materials, biosorbents, and waste materials from industry and agriculture. These materials could be used to remove dyes from solutions as sorbents. Biosorbents (chitosan, peat, biomass), clay materials (bentonite, kaolinite), zeolites, siliceous materials (silica beads, alunite, perlite), agricultural wastes (bagasse pith, maize cob, rice husk, coconut shell), industrial waste products (waste carbon slurries, metal hydroxide sludge), and others have all been reported as sorbents (starch, cyclodextrin, cotton). G. Crini looked at the technical feasibility of using nontraditional low-cost adsorbents to remove dye from contaminated wastewater (CRINI, 2006). He believes that low-cost adsorbents have a lot of potential for commercial applications in the future. Chitosan-based sorbents have demonstrated superior dye removal capabilities when compared to

activated carbon. Despite several papers on low-cost adsorbents being published, there is still a scarcity of information containing a comprehensive comparison of sorbents. Although much progress has been made in the field of low-cost sorbents, much more work is needed (i) to predict the performance of adsorption processes for dye removal from real industrial effluents under a variety of operating conditions, (ii) better understand adsorption mechanisms, and (iii) demonstrate the use of lowcost sorbents on an industrial scale. For the removal of crystal violet and rosaniline hydrochloride (basic fuchsine) from wastewater, Mohan et al. used fly ash generated in thermal power plants as a low-cost adsorbent (Mohan et al., 2002). They discovered that as the temperature rises, the adsorption of the two dyes increases, indicating that the process is endothermic. Both the Freundlich and Langmuir models can be used to fit the data and estimate model parameters, according to the findings. Overall, the nonlinear Freundlich adsorption isotherm fits the data better. Crystal violet and basic fuchsine dyes have higher or comparable adsorption capacities on fly ash than other adsorbents used for the same or other cationic dyes. According to their findings, fly ash can be used as an effective adsorbent for dye removal.

#### 2.3.6 Advanced oxidation process (AOP)

Traditional oxidation treatments have struggled to oxidize dyestuffs and organic compounds with complex structures at low concentrations or when they are particularly resistant to oxidants. Advanced oxidation processes (AOPs) have been developed to generate hydroxyl free radicals using various techniques to alleviate the problems mentioned. The AOPs, which combine ozone (O<sub>3</sub>), hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) and UV irradiation, to treat textile wastewater, have shown the most promise. These oxidants effectively decolourized dyes but did not eliminate COD completely. Any AOP's goal is to generate and use hydroxyl free radical (OH•) as a strong oxidant to destroy compounds that cannot be oxidized by conventional oxidants. Advanced oxidation processes are defined by the production of OH• radicals and attack selectivity, which is a useful property for an oxidant. The fact that AOP can be used in a variety of ways for OH• adds to their versatility. Combining O<sub>3</sub>, H<sub>2</sub>O<sub>2</sub>, TiO<sub>2</sub>, UV radiation, electron-beam irradiation and ultrasound is a common way to speed up the production of OH•. The most promising are

O<sub>3</sub>/H<sub>2</sub>O<sub>2</sub>, O<sub>3</sub>/UV and H<sub>2</sub>O<sub>2</sub>/UV for oxidizing textile wastewater. In recent decades photocatalysis has appeared as an emerging advance oxidation process.

#### 2.4 Selection of Methodology

A wide range of techniques and methods exist for the treatment of dye bearing wastewater. There are Physical processes, are used to substantially remove floating and settleable solids in wastewater. This process includes screening, sedimentation, adsorption, membrane filtration, ion exchange, Irradiation, or Chemical Processes, which enhance the degradation of volatile organic compounds present at low concentrations in contaminated media. Biological treatment, in which bacteria break down the organic parts of wastewaters; usually accomplished by bringing the wastewater and bacteria together in trickling filters, aerobic, anaerobic or in the activated sludge process. Several membrane-based processes also exist as treatment methods. As there is a wide range in pH and dyes are highly coloured materials, which are difficult to decompose biologically, conventional Physico-chemical and biological treatment processes are not effective in removing dyes from wastewaters. Moreover, all these processes are costly and cannot be used by small industries to treat the wide range of dye wastewater. However, after a brief review of available literature and consultation with guide we have decided to test the effectiveness of electro coagulation on treatment of textile dye bearing waste waters with Rhodamine B which is a cationic dye extensively used in textile industry. The EC technology offers an alternative to the use of metal salts or polymers and poly-electrolyte addition for breaking stable emulsions and suspensions. The technology removes metals, colloidal solids and particles, and soluble inorganic pollutants from aqueous media by introducing highly charged polymeric metal hydroxide species. These species neutralize the electrostatic charges on suspended solids to facilitate agglomeration or coagulation and resultant separation from the aqueous phase. EC is a technique with large potential as seen from the literature review presented in and one of the lesser explored ones. It has already proved to be an efficient technique in a wide variety of other processes of metal removal and wastewater treatment. The EC process provides a simple, reliable and cost-effective method for the treatment of wastewater without any need for additional chemicals, and thus the secondary pollution. Electro-coagulation would subsequently generate less solid waste, thus saving on disposal costs. The EC process also has the advantage of removing the smallest colloidal particles, because the applied electric field sets them in faster motion, thereby facilitating the coagulation. Electro coagulation thus deserves a thorough study as a technique for treatment of textile reactive dye bearing wastewater.

#### 2.5 Electrocoagulation as a removal technique

Electro coagulation systems have been in existence since Dieterich, patented one in 1906, however only recently have they aroused interest for treatment of textile dye bearing wastes using a variety of anode and cathode geometries, including plates, balls, fluidized bed spheres, wire mesh, rods, and tubes. It is increasing being suggested for separation of solids from the water molecules and treatment of heavy metals, virus, bacteria, pesticides, arsenic, MTBE, cyanide, BOD, TDS, and TSS along with treatment of Municipal, Industrial, wastewater contaminated with sewage treatment, dairy, plating shops, contaminated soil, steam cleaner and car wash water, mining, laundries, and textile dyes. Recently a lot of work is being done on treatment of dye bearing wastewater from textile industry using this method.

Oeguetveren et al. (1992) tried to remove a dye such as Acilan Blau using electrochemical method. Effects of several factors on the removal rate were examined such as pH, presence of supporting electrolyte, duration of electrolysis, initial dye concentration and applied potential. A removal rate of 100% was practically achieved in a very short time with synthetic solutions. Additionally, samples of wastewater taken from the operating line of a local textile plant have also been treated in the reactor. Complete removal of the dyes (98-100%) from these solutions has been achieved with a little increase in time.

Do and Chen (1993) studied effects of various parameters during decolorization treatment of textile waste waters. In the electrocoagulation of Drimarene Discharge X-3LG ® (R12S) and Samaron Yellow 4 the effects of sacrificial anodes, initial pH, rate of stirring and concentrations of dyes on the fraction of decolourization were tested. The experimental results revealed that the

colour of dyes in the aqueous phase was effectively removed when iron and aluminium were used as sacrificial anodes and the concentration of the dyes was less than 100 ppm. When the concentration of R128 exceeded 300 ppm, iron was a more suitable sacrificial anode for electrocoagulation of R12S than aluminium. For the electrocoagulation of DO5H in aqueous solution aluminium as a sacrificial anode was superior to iron when the concentration of DOS5H exceeded 300p.p.m. Using iron as sacrificial anode, the residual concentration fraction of R12S was decreased below 10% when the initial concentration of R12S and charge passed were 500p.p.m. and 120C, respectively. The decolourization fraction of DOSH was 99% when aluminium was used as sacrificial anode.

Silva and Filho (2001) investigated into the application of electrolysis technology as an alternative for removal of color from textile wastewater. The study included a series of experiments with aqueous solutions of two commonly used sulphur dyes: Brown Sodyesul 2GBB ECO and Blue Sodyesul 7RB ECO. The experimental program involved experiments of chemical coagulation and electrolytic treatment to compare the performance. In the experiments of electrolytic treatment using steel electrodes, an investigation was carried out on the effects of parameters such as applied potential, initial pH and conductivity on the efficiency of colour removal. The experimental results are assessed in terms of the abatement of colour as measured in absorbance values. These measurements were used to determine the percent efficiency of the process. Colour abatement of at least 99% was achieved in a contact time of 10 minutes with an applied potential of 5.0 V. The electrolytic process proved to be an effective route for the removal of colour from wastewater. Overall, the experimental results suggest the main removal mechanism is the electrocoagulation of the dye.

Szpyrkowicz et al. (2001) described the removal of colour from textile wastewater bearing a mixture of three disperse dyes. The experiments were carried out in a reactor equipped with an Al sacrificial anode (100 cm<sup>2</sup>) and a stainless-steel cathode of the same dimensions. Decolorisation occurred because of destabilization of colloids due to an effect of an electric field generated between the electrodes (polarization coagulation) and the reactions with coagulating compounds, formed in situ during anode oxidation (volumetric coagulation), followed by a subsequent

flotation of agglomerates of the particles. During the study the following parameters were varied: initial pH of the solution, current density (between 100 and 300 A/m²) and an initial concentration of the dyes. The best results of elimination of colour, equal to 99%, were obtained during 5 minutes of treatment under the conditions of pH of 9.

Butnaru et al. (2003) discussed the possibility to treat wastewater from cellulose dyeing with direct dyes by electrocoagulation. Four direct dyes are analyzed, at different concentrations. COD decrease and color of the residual solution are investigated, as well as the volume of the sludge produced in the process. Optimum treatment parameters are determined for the electro-coagulation process.

Pathak et al. (2004) successfully worked on EC treatment of orange II dye solution in a continuous flow cell by using sacrificial iron electrodes. The cell contained five parallel iron electrodes, which form four parallel cells. The internal volume of the cell is approximately 450 ml. Inter electrode distance was 0.6 cm and experiments were run at 25°C. This study showed the 99% removal of the dye from the waste stream under the optimum conditions. The optimum conditions were current = 4.0A (42 V); initial pH = 7.3; flow rate = 350 mL/min; and dye concentration = 10 ppm

Sanroman et al. (2004) worked on electrochemical decolorization of structurally different dyes (bromophenol blue, indigo, poly R-478, phenol red, methyl orange, fuchsin, methyl green and crystal violet). This study showed that the electrochemical process allowed a colour removal of all dyes studied, although the decolorization rate largely depended on the chemical structure of the different dyes. Nearly complete decolorization was achieved in 30 min for bromophenol blue followed by methyl orange (95% in 30 min) and methyl green (90% in 60 min), whereas phenol red was hardly decolorized (30% in 60 min). In mixtures of two dyes, the decolorization rate became similar for both dyes.

Alinsafi et al. (2005) worked with Al electrode for the removal of Reactive Blue dye with an electrode spacing of 2.0 cm and optimum current density of 12 mA/cm<sup>2</sup> and operating time of 105 min. It was found that color removal was greater than 90%.

Kobya et al. (2005) have worked on electrocoagulation of levafix orange textile dye solution by using aluminum sacrificial anode and inter electrode distance was 1.1cm. It was found that almost complete removal of pollutants (99.9%) was obtained with typical operating conditions; 100 A/m² current density, 15 min operating time and initial pH 3.0. On the other hand, 95% decolorization efficiency may be obtained for the same current density in 12 min and at pH 6.4. The corresponding electrode and energy consumptions during the electrolysis were found to be 1.8 kg Al/kg dye and 35 kWh/kg dye. The decolorization efficiency for Al electrode falls in a linear fashion from 99.6 to 88% with increase in concentration of the dye from 100 to 500 mg/L.

Kashefialasl et al. (2005) have studied the batch removal of the reactive textile dye viz. Colored Index (C.I.) Acid yellow 36 from an aqueous medium by electrocoagulation using iron electrodes. The effects of electrolyte concentration, initial pH, current density, electrode area, interelectrode distance, dye concentration, and treatment time on the decolorization efficiency have been investigated. The results showed that when the initial concentration of the dye was 50 ppm, the dye was effectively removed (83%) at pH ranging from 7.0 – 9.0, time of electrolysis of approximately 6 min, current density of approximately 127.8 A/m², temperature of approximately 298 °K, and interelectrode distance of 2.5 cm. It was also found that the color removal percent with increasing of dye concentration decrease.

Ghalwa et al. (2005) have worked on electrochemical degradation of Acid Green dye (C.I. 61570) in aqueous wastewater dyestuff solutions using a lead oxide coated Titanium electrode. results of the electrocatalytic oxidation process of the dyestuff solutions were expressed in terms of the remaining dye concentration and chemical oxygen demand (COD) values. Electrolytic cell volume was kept 150 cm<sup>3</sup> and distance between electrode was 3.0 cm. The optimum operating conditions of the treatment process were current density of 15 mA/cm<sup>2</sup>, temperature of 30°C, initial dye load concentration of 100 mg/l, and electrolysis time of 10 min. This

study concluded that the prepared PbO,/Ti electrode has a high efficiency in the removal of acid green dye from their aqueous solutions, at low current densities, short electrolysis times, and mild temperatures.

Daneshvar et al. (2002) has studied decolorization of orange II by electrocoagulation method. EC of dye-containing aqueous solutions were carried out in the beakers (250 ml). The results showed that the orange II was effectively removed at initial pH of 7.5-8.5. When the stirring rate exceeded 100 rpm, the removal efficiency decreased. The results also indicated that the removal efficiency of orange II and COD of the sample were raised to > 98 and > 84%, respectively. When iron was used as sacrificial anode and the initial concentration of orange II was about 50 ppm, the optimum current density, stirring rate, initial concentration, distance between the electrodes, were 34.62 A/m², 100 rpm, (50-200 ppm), 2.0 cm, respectively.

Daneshvar et al. (2004) studied electrocoagulation of Acid Red 14 dye. The EC unit consisted of an 0.5 L electrochemical reactor with iron anode and steel cathode. It was found that for dye solutions with COD of approximately 30 ppm and dye concentrations less than 150 ppm, high color removal (93%) was obtained when the pH ranged from 6 to 9, time of electrolysis was approximately 4 min, current density was approximately 80 A/m², the temperature was approximately 300°K, and inter electrode distance was 1.0 cm. During the EC process under these conditions, the COD decreased by more than 85%. The experimental results also showed that an EC cell with several electrodes was more effective than a simple electrochemical cell in color removal.

Daneshvar et al. (2006) worked on electrocoagulation for the removal of color from solutions containing C. J. Basic Red 46 (BR46) and C. I. Basic Blue 3 (BB3). The study shows that an increase in the current density up to 60-80 A/m<sup>2</sup> enhanced the color removal efficiency, the electrolysis time was 5.0 min and the range of pH was determined between 5.5 and 8.5 for two mentioned dye solutions. It was found that for, the initial concentration of dye in solutions should not be higher than 80 mg/L in order to achieve a high color removal percentage. The optimum conductivity was found to be 8.0 mS/cm<sup>2</sup>, which was adjusted using proper amount

of NaCl with the dye concentration of 50 mg/L.

Modirshahla et al. (2006) investigated the effect of different electrode connections on the removal efficiency of Tartrazine from aqueous solutions by electrocoagulation. In this work it was shown that the EC treatment achieves a fast and effective removal of Tartrazine. For 250 ml dye solution with COD of approximately 30 mg/l, dye concentration of 40 mg/l, color and COD elimination of 100% and 90% were obtained, respectively, when the pH was about 5.78, time of electrolysis was approximately 6 min, current density was approximately 120 A/m², iron anode and aluminium cathode and interelectrode distance was 1.5 cm. Electrocoagulationwith Fe/Al (anode/cathode) was more effective for the treatment process than Fe/Fe electrode pair.

In the study conducted by Chrysafopoulou et al. (2006), an electrolytic pilot scale unit was manufactured for the tertiary treatment of biologically processed wastewater aiming at its reuse for groundwater recharge or for agricultural process. The integrated pilot unit consisted of two compartments, the electrolytic cell (reactor) and the unit of flotation and precipitation of the coagulated colloid particles. The anode of the cell was a DSA type electrode with iridium and platinum oxide mixture deposited on a titanium substrate; Stainless Steel 316 was used for the cathode. The unit has been operating at the Wastewater Treatment Plant (WWTP) of the Herakleion Industrial Park (Crete, Greece). The results obtained from the operation of the unit for the tertiary treatment of industrial wastewater are very encouraging. Complete decolorization and disinfection of the wastewater as well as essentially the complete removal of suspended solids.

Aleboyeh et al. (2007) studied the decolorization of C.I. Acid Red 14 (AR14) azo dye by EC process in a batch reactor. Response surface methodology (RSM) was applied to evaluate the simple and combined effects of the three main independent parameters, current density, time of electrolysis and initial pH of the dye solution on the colour removal efficiency and optimizing the operating conditions of the treatment process. Analysis of variance (ANOVA) showed a high coefficient of determination value ( $R^2 = 0.928$ ) and satisfactory prediction second-order regression model was derived. Maximum color removal efficiency was predicted and

experimentally validated. The optimum current density, time of electrolysis and initial pH of the dye solution were found to be 102Am 2, 4.47 min and 7.27, respectively. Under optimal value of process parameters, high removal (>91%) was obtained for Acid Red 14. Graphical response surface and contour plots were used to locate the optimum point.

## 2.6 Research Gap

It is evident from the literature reviewed that there exists a wide gap in regard of assessment of efficacy of a cost-effective dye removal method. Electrocoagulation, now-a-days is being widely applied in water and wastewater related research. Some initial studies conducted on electrocoagulation-based system demonstrated satisfactory performances in regard of decolourization. Considering the paucity of studies carried out in this particular field, especially in Indian context in-depth research may be undertaken using different types of electrodes to explore the efficacy of the system.

# **Chapter-3**

# **Theoretical Considerations**

EC technique uses a direct current source between metal electrodes immersed in polluted water. The electrical current causes the dissolution of metal plates including iron or aluminum into wastewater. The metal ions, at an appropriate pH, can form wide ranges of coagulated species and metal hydroxides that destabilize and aggregate the suspended particles or precipitate and absorb dissolved contaminants (Daneshwar et al., 2006). Compared with traditional flocculation and coagulation, electro-coagulation has, in theory, the advantage of removing small colloidal particles; they have a larger probability of being coagulated because of the electric field that sets them in motion. Addition of excessive amount of coagulants can be avoided, due to their direct generation by electro-oxidation of a sacrificial anode (Alinsafi et al., 2005). The EC process is characterized by a fast rate of pollutant removal, compact size of the equipment, simplicity in operation, energy efficiency, and low operating and equipment costs (N. Daneshvar et al., 2004). The EC is a simple and efficient method for the treatment of water and many kinds of wastewater. It has been tested successfully for the treatment of various effluents such as wastewater containing food and protein wastes, textile wastewater, aqueous suspensions containing ultrafine particles, fluoride-containing water, restaurant wastewater, textile dye solutions, and smelter wastewater containing harmful arsenic.

#### 3.1 Mechanism of EC

Electrocoagulation (EC) is one of the emerging alternative methods utilized now a days to desalinate water. This process has been successfully applied to treat pollutants and nutrients from several types of wastewaters (Bakshi et al., 2020; Chen, 2004; Sharma and Naushad, 2020). In recent years, EC has increasingly gained the attention of many researchers, who have been able to employ EC to eliminate numerous water contaminants. Widespread applications were found for this technology owing to its high capacity for treating wastewater, such as dyes

from the textile industry (Naje et al., 2019). Elements like lead and copper (Bouguerra et al., 2015), fluoride (Betancor-Abreu et al., 2019), glyphosate removal (Danial et al., 2019), and heavy metals (Al-Shannag et al., 2015) are also used to treat high-salinity water (Al-Raad et al., 2019).

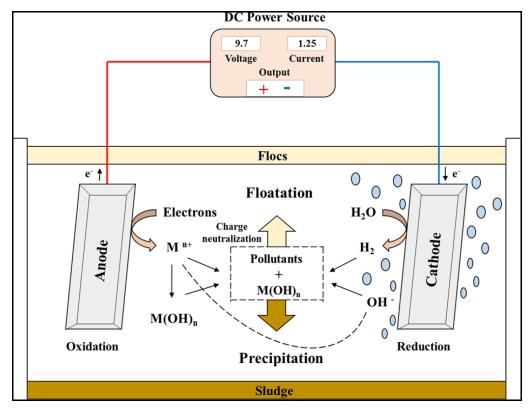


Figure 3.1 - Schematic Diagram of the Electrocoagulation process

The mechanism of EC is based on the generation of in-situ coagulants through dissolution of the polar material. Different electric currents are applied on the sacrificial metal electrodes immersed in targeting water inside an electrolytic reactor, which dissolves metallic material and produces a coagulant species in situ. More precisely, EC technology is used for delivering metallic hydroxide to target water through coagulant species income in situ by electro dissolution of the utilized material (Garcia-Segura et al., 2017; Gilhotra et al., 2018). EC is also considered an environmentally friendly technology because it produces a low amount of secondary waste that can easily be eliminated by filtration with the assistance of generated bubbles, no added chemicals, and low operating costs as well as can be used with a combination of renewable energies from solar or wind power (Dura and Breslin, 2019, Sharma et al., 2011). The most common

materials used in this technology include aluminium (Al) and iron (Demirbas and Kobya, 2017; Deng et al., 2019; Keyikoglu et al., 2019; Yang et al., 2020). From these studies we can see that electrocoagulation has been proven to treat any type of water. Also, studies have shown that electrocoagulation is an effective process because it destabilizes the finely dispersed particles in wastewater and thus, removing pollutants such as greases, hydrocarbons, heavy metals, and suspended solids (Chunjiang et al., 2017; Rodriguez et al., 2020). Following this, electrocoagulation was proven able to remove contaminants from waste that resulted in minimal sludge production as compared to the chemical coagulation process (Garcia et al., 2017; Maitlo et al, 2019). Electrocoagulation is a cheap treatment process in comparison to conventional chemical (Collivignarelli et al., 2019). Specifically, an effective cost in electrocoagulation lies behind the metal ion's dissociation during the treatment process. The formation of hydroxide flocs has resulted from the dissolution of aluminum or iron as the anode electrodes have defined the effectiveness of the electrocoagulation process (Collivignarelli et al., 2019). The anode electrodes act as the coagulant, leads to the generation of metal ions before reacting with the hydroxyl group in the wastewater to form coagulant as formulated in Equation 1, Equation 2, Equation 3, and Equation 4 (Nasrullah et al., 2019)

Metal electrodes such as Fe<sub>3</sub><sup>+</sup> and Al<sub>3</sub><sup>+</sup> ions will immediately undergo spontaneous reactions that resulted in the production of hydroxides or polyhydroxides. Polyhydroxides act as the counter ions that have a strong affinity for dispersed particle leads to coagulation along with the gases evolved at the electrodes may impinge on and thus causes flotation of the coagulated pollutants (Qiang Gao et al., 2019). Usually, flocs that are formed through electrocoagulation are large, stable, acid-resistant, and contain less water-bound. These allow the flocs to be easily separated through filtration since it contains less total dissolved solids.

As a result, electrocoagulation made water treatment a low-cost process since it merges coagulation and flocculation in one reactor, leading to low water recovery cost since the bubbles carry pollutants easily to the top (Mamelkina et al., 2019). Overall, the electrocoagulation treatment unit is suitable in rural areas even though with no access to electricity since the solar panel attached to the unit is sufficient to run the whole process. Apart from that, electrocoagulation consists of chemical and physio-chemical phenomena due to the electrolysis effects. This means electricity is needed for the formation of coagulants by anode dissolution, destabilization of contaminant and particulate suspension, and aggregation of the destabilized phase causes the formation of the floc (M. Sadik, 2019). A study on electrocoagulation indicated that a process that occurred in an electrolyte leads to the possibility of ion transfer between two (2) electrodes as result of the applied current to the treatment unit (Zazou et Al., 2019). The positive ions will transfer to the cathode whereas negative ions will transfer to the anode during electrocoagulation. There is various type of unit operation for electrocoagulation treatment unit such as (i) basic reactors that consist of tank cells, plate and frame cells, rotating cells and (ii) complicated three-dimensional reactor system like fluidized bed, packed bed cell, and even porous carbon packing cells. Thus, it is necessary to improve these arrangements since electrodes with large surface area are needed for a workable rate of metal dissolution as shown in the following figure.

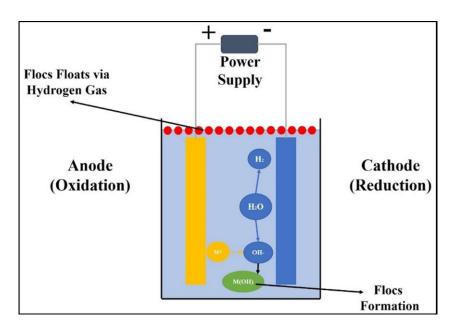


Figure 3.2 - Metal Dissolutions in Electrocoagulation

Electrocoagulation system consists of a pair of metal sheets called an electrode, arranged in two pairs (anode and cathode). Electrocoagulation works based on the principles of electrochemistry in which the cathode oxidizes or loses electrons while the water is reduced or gains electrons that resulted in wastewater treatment. The electro dissolution causes an increased metal concentration in the wastewater and finally precipitates as oxides or hydroxides. Electrocoagulation generates highly metal hydroxides, functioning as the neutralizer for the electrostatic charge present among the colloid particles, to promote agglomeration or coagulation of suspended materials. The reactions happen in three (3) general successive steps which are (i) formation of coagulation through sacrificial anode electrodes, (ii) breaking of emulsion and coagulation of colloid particles, and (iii) flocculation or adsorption of aggregated destabilized metal hydroxide flocs. Coagulants are formed when soluble anodes are hydrolyzed to polymeric hydroxides to cause the destabilization of colloid particles through reaction with released metal ions. The metal ions destabilized the stable suspension by breaking the emulsion of colloid particles to form flocs in the water. These reactions indicate that electrocoagulation is indeed environmentally friendly because it is characterized by minimal usage of chemicals in the process along with easy operations that resulted in minimal sludge production. Certainly, electrocoagulation has been under great interest for commercialization due to its environmental compatibility. Electrocoagulation does not require the addition of chemical coagulants since the coagulants are generated in situ during the treatment. After all, electrocoagulation has been used widely to treat various types of water and wastewater. Optimization of electrical power consumption through maximum effluent throughput rates has been conducted for minimum energy consumption. Thus, a sustainable electrocoagulation treatment unit can be achieved with proper control of various operating parameters such as current density, pH, electrode arrangement, and reaction time as mentioned by Nidheesh et al. (2018).

# 3.2 Electrocoagulation Practices

EC systems are run either on batch or on continuous basis. The following text gives a brief account of both types of systems as in practice.

#### 3.2.1 Batch Electrocoagulation System

In a batch EC process, electrocoagulation of dye solution is carried out in a reactor with a magnetic stirrer to agitate the solution. The electrodes are placed inside a glass reactor and connected to a digital power supply. In each run, fixed volume of dye solution is decanted into the electrolytic cell and the run is started. Samples were withdrawn at appropriate time intervals and then filtered and analyzed for the residual dye concentration using double beam UV/VIS spectrophotometer. Batch reactor applications typically operate with a fixed wastewater volume per treatment cycle but suffer from the perceived disadvantage (from a design and operational standpoint) that conditions within the reactor change with time.

#### 3.2.2. Continuous Electrocoagulation System

In this process Electrocoagulation of dye-containing solution is carried out in a flow-through EC apparatus consisting of a flow-through cell, the electrode assembly, the feed pump and the DC power supply unit. At the beginning of a run, the dye solution of desired concentration is pumped through the cell at a predetermined flow rate and after the treatment of desired volume of dye solution, the EC run is stopped. The concentration of the dye stuff in the treated sample is analyzed using the UV-Vis spectrophotometer.

Continuous EC systems can treat large volumes of waste wasters. Flow regime should be laminar because it favours the growth of large flocs that are easier to remove. A key advantage for such reactor systems is that their coagulant requirements are essentially fixed, a major advantage in terms of both design and operation (Holt et al., 2005).

In a continuous stirred tank reactor, the concentration of materials in the feed stream was immediately lowered to the concentration in the tank and effluent. Therefore, the overall rate of reaction, and consequently the conversion of the reactant to product, will be less than that for a plug flow tubular reactor (PFTR), where no such lowering of concentration upon entering the reactor occurs. If the gap between the electrodes is closer, and the ratio of length/width is much larger than 1, it can be considered that there is no mixing of the fluid in a longitudinal direction. Therefore, the reactor can be considered a PFTR (Kim et al., 2002).

## 3.3 Factors Controlling Electrocoagulation

Decolorization of dye solution process by EC is influenced by several factors such as:

- 1. pH
- 2. Initial concentration
- 3. Electrolysis time
- 4. Current density
- 5. Electrode material
- 6. Inter electrode distance
- 7. Number of electrodes

## 3.3.1 pH

EC strongly depends on pH of the solution, concentrations and conductivity of the continuous phase. When iron electrode is connected by DC power source in an electrolytic system, it produces coagulant  $Fe(OH)_n$ , where n = 2 or 3. Two mechanisms for the production of the metal hydroxide have been proposed

#### (Daneshvar et al., 2006)

#### **Mechanism 1**:

Anodic reactions:  $4\text{Fe(s)} - 8\text{e}^{-} \rightarrow 4\text{Fe}^{2+}$  (ag)

$$4\text{Fe}^{2+}$$
 (aq) +  $10\text{H}_2\text{O} + \text{O}_2 \rightarrow 4\text{Fe}(\text{OH})_3$  (s) +  $8\text{H}^+$ 

Cathodic reaction:  $8H^+$  (aq)  $+ 8e^- \rightarrow 4H_2$ 

Overall reaction:  $4\text{Fe(s)} + 10\text{H}_2\text{O} + \text{O}_2 \rightarrow 4\text{Fe(OH)}_3 \text{ (s)} + 4\text{H}_2$ 

$$nFe(OH)_3 \rightarrow Fe_n(OH)_{3n}$$
 (s)

#### **Mechanism 2:**

Anodic reactions:  $4\text{Fe(s)} - 2\text{e}^{-} \rightarrow \text{Fe}^{2+}$  (aq)

$$Fe^{2+}$$
 (aq) +  $2OH^{-}$  (aq)  $\rightarrow Fe(OH)_2$  (s)

Cathodic reaction:  $2H_2O + 2e^- \rightarrow 2OH^- + 4H_2$ 

Overall reaction: Fe(s) +  $2H_2O \rightarrow Fe(OH)_2 + H_2$ 

$$nFe(OH)_2 \rightarrow Fe_n(OH)_{2n}$$
 (s)

The insoluble metal hydroxide of iron remains in the aqueous phase as a suspension, which removes the pollutants from wastewater by precipitation and adsorption mechanisms (Kobya et al., 2003). NaCl increases the production rate of mono-nuclear and poly-nuclear ferric hydroxide by chain reaction as described in

previous equations and improves the pollutant removal (Kobya et al., 2003).

Precipitation: Dye + Monomeric Fe  $\rightarrow$  [Dye – Monomeric Fe] (s)

Dye + Polymeric Fe  $\rightarrow$  [Dye – Polymeric Fe] (s)

Adsorption: Dye +  $Fe_n(OH)_n(s) \rightarrow [sludge]$ 

[Dye – Polymeric Fe] (s) + Fe<sub>n</sub>(OH)<sub>n</sub> (s)  $\rightarrow$  [sludge]

#### 3.3.2 Initial Concentration

Percent removal decreases with the increase in the initial concentration of the dye. This could be due to the presence of intermediate products produced during the electrolysis process at higher concentrations, which then could compete for the active sites on the electrode, or these intermediate products may be insoluble in water and would block the electrode's active sites. On the other hand, one of the most important pathways of colour removal by EC process is adsorption onto metallic hydroxide flocs and the adsorption capacity of flocs is limited.

#### 3.3.3 Electrolysis Time

The colour removal efficiency depends directly on the concentration of ions produced by the electrodes. When the electrolysis period increases, an increase occurs in the concentration of ions and their hydroxide flocs. Therefore, percent removal increases with increase in time of electrolysis.

#### 3.3.4 Current Density

In all EC processes, current density is the most important parameter for controlling the reaction rate within the reactor. When the current density increases, the efficiency of ion production on the anode and cathode increases. Therefore, there is an increase in floc production in the solution and hence an improvement in the efficiency of colour removal. Moreover, bubble generation rate increases, and the bubble size decreases with increasing current density, which are both beneficial for high pollutant removal efficiency by H<sub>2</sub> flotation.

#### 3.3.5 Electrode Material

The electrode material impacts markedly on the performance of the EC reactor. The anode material determines the cation introduced into solution. Several researchers have studied the choice of electrode material with a variety of theories as to the preference of a particular material. The most common electrodes are aluminium or iron plates. **Do and Chen (1994)** compare the performance of iron

and aluminium electrodes for removing colour from dye-containing solutions. Their conclusion was that the optimal EC conditions varied with the choice of iron or aluminium electrodes, which in turn is determined by some factors such as initial pollutant concentration, pollutant type.

#### 3.3.6 Inter Electrode Distance

Percent removal decreases with the increase in electrode gap. With increasing distance between electrodes, less interaction of ions with hydroxyl polymers expected. In the other words, decreasing local concentration and electrostatic attraction are the reasons for decreasing removal of dye.

#### 3.3.7. Number of Electrodes

There is a direct relation between removal efficiency and total no. of electrodes. As the number of electrodes increases, the distribution of the coagulation agents becomes more effective. This can produce the related coagulation and completes the removal of samples.

# **Chapter-4**

# **Objective and Scope of the study**

## 4.1 Objective of the Study

Based on the available research gap the objective of the proposed investigation can be stated as "To assess the efficacy of an electrocoagulation-based laboratory-scale novel reactor system for removal of Rhodamine B from wastewater".

## 4.2 Scope of the Study

The specific scopes of studies included are –

- Fabrication of a novel reactor for electrocoagulation process.
- Preparation of synthetic Rhodamine B wastewater and its characterization.
- Evaluation of the performance of the proposed laboratory-scale novel reactor system in achieving the decolourization of the synthetic-coloured water.
- Optimization of the operational parameters for the proposed laboratory-scale electrocoagulation system in achieving the decolourization of the synthetic-coloured water

# **Chapter-5**

# **Materials and Methods**

## **5.1 Materials and equipment**

The following materials and equipment were used during conducting the experiment.

- i) Rhodamine B
- ii) Sodium Chloride (Nacl)
- iii) UV-Visible spectrophotometer
- iv) Clamp Meter for measuring DC current.
- v) Verilog (AC to DC converter)
- vi) Fibre Reactor (30 cm \* 15 cm \* 25 cm)
- vii) Iron plate (12 cm \* 21 cm)
- viii) Weighing Machine
- ix) Distilled Water
- x) Pipette

#### **5.1.1 Rhodamine B**

Rhodamine B (RhB) dye is a synthetic dye widely used as a coloring in textiles and food products. It is a bright red organic fluorescent pigment used as a coloring agent in various industries such as textiles, paper, paint, etc. Its high solubility in water and its low-cost nature makes it a commonly used pigment in various industries. RhB dye, like other dyes, stable to light, heat, oxidation, and is not biologically degradable. Some studies suggest that RhB dye is considered as carcinogenic and mutagenic origin in animals and humans. It causes biological issues such as skin, respiratory inflammation, hemolysis, degenerative changes in the liver, and kidneys, etc. (Li et al., 2019). It is known as water tracer fluorescent as it is used as a tracker inside the water to evaluate the flow rate and direction of the water flow. This dye belongs to the xanthine family and is commonly used in the biological, analytical, and optical sciences. Among its uses is that it is frequently used in the manufacture of paper, textile and leather dyeing, and fluorescent cell dyeing, and it is also found in wastewater (Yu et al.,

2018). Expanding release of a significant overabundance of dyes by color fabricating and material business leads to extreme environmental issues. RhB dye is broadly utilized for coloring, and their release in water bodies gives antagonistic impacts on human and creature.

Table 5.1: Properties of Rhodamine B

Common name	Rhodamine B
Chemical name	[9-(2-carboxyphenyl)-6-diethylamino-3-xanthenylidene]-diethylammonium chloride
Chemical formula	$C_{28}H_{31}CIN_2O_3$
Molecular weight	479.017 g/mol
Adsorption maximum	545 nm
Class	Triphenylmethane
Appearance	Basic Violet 10; Brilliant Pink B
Molecular structure	H <sub>3</sub> C CH <sub>3</sub> CH <sub>3</sub> CH <sub>3</sub> CH <sub>3</sub> CH <sub>3</sub>

Concentration of Rhodamine B in aqueous solution was measured by a UV-Visible Spectrophotometer. Standard curve was prepared from solution of known concentration at 554 nanometre wavelengths. The standard curve is given below.

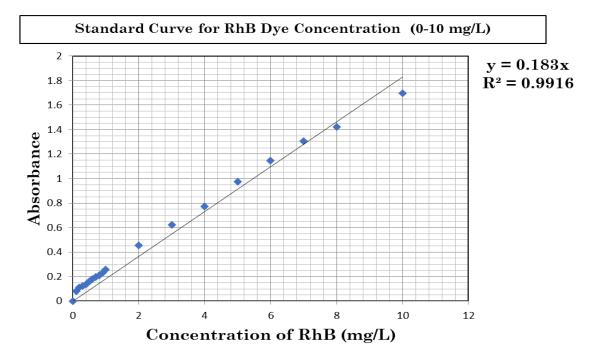


Fig. 5.1: Standard Curve of Rhodamine B





Fig. 5.2: Manufacturing details of Rhodamine B

## **5.1.2** UV-Visible spectrophotometer

For measuring the concentration of Rhodamine B, UV-Visible spectrophotometer was used. It was manufactured by PerkinElmer. UV grade Cuvette used in this spectrophotometer was manufactured by LARK. Photo of spectrophotometer and cuvette is given below



Fig. 5.3: UV-Visible spectrophotometer



Fig. 5.4: UV grade cuvette

## **5.1.3 Clamp Meter**

Clamp Meter was used for measuring DC current. The clamp meter was manufctured by Fluke. It can measure DC current up to 200 ampere.



Fig. 5.5: Clamp Meter

## **5.1.4** Electrocoagulation Reactor

For electrocoagulation, a fiber glass reactor setup was manufactured from a local shop. Thickness of glass fiber was 0.5 cm. Dimension of the reactor was length = 30 cm, Width = 15 cm and Height = 25 cm. For holding the electrode plates, a cascade was inside the reactor. Dimension of the cascade was length = 25 cm, Width = 13 cm and Height = 22 cm. Picture of reactor and cascade is given below.



Fig. 5.6: Fiber Reactor

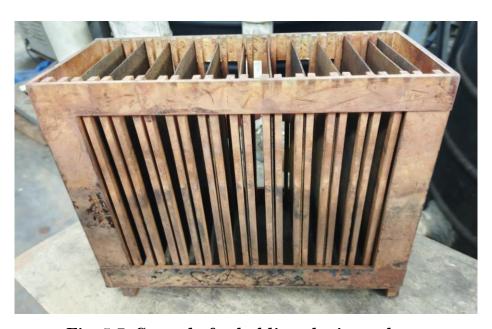


Fig. 5.7: Cascade for holding the iron plates

# **5.1.5 Iron Plate**

Iron plate was used as electrode in the electrocoagulation setup. The plates were manufactured from a local shop. Dimension of those plates was 12 cm (width) \* 21 cm (height). Maximum 12 number of plates can be placed inside the cascade. Picture of the iron plates is given below.



Fig. 5.8: New Iron Plate



Fig. 5.9: Corrosion of Iron Plate



Fig. 5.10: Sludge formed during removal of Rhodamine B



Fig. 5.11: Electrocoagulation Setup





Fig. 5.12: Pictorial representation of Removal of Rhodamin B with time

# **Chapter-6**

# **Result and discussion**

A detailed discussion on the results of the experiments conducted is given in this chapter. These results include:

- 1) Variation of removal percentage with initial concentration of Dye.
- 2) Variation of removal percentage with initial pH concentration.
- 3) Variation of removal percentage with current density.
- 4) Variation of removal percentage with Inter Electrode Distance (IED).
- 5) Variation of removal percentage with respect to electrolyte concentration.
- 6) Variation of removal percentage with respect to turbulence.

# 6.1 Variation of removal percentage with initial concentration of Dye

In the batch reactor, 3 initial concentrations were taken for testing purposes. These concentration are

- i) 30 ppm solution of Rhodamine B
- ii) 20 ppm solution of Rhodamine B
- iii) 10 ppm solution of Rhodamine B

6 Liters of sample was taken in the reactor. During testing the initial pH of the solution water was kept 7. Electrolyte concentration was 2 g/L. Inter Electrode Distance was kept at 2 cm. Current density was 2.26 mA/cm<sup>2</sup>.

#### **6.1.1 Initial Concentration = 30 ppm**

The following table shows the variation of removal when initial concentration is 30 ppm. From the below table we can see that after 30 min almost 89.20 % of the dye was removed and after that the rate of removal percentage was very low. After 90 min of batch timing, final removal percentage was almost 97 %. Fig. 6.1 shows the variation of remaining dye concentration with time and Fig. 6.2 shows the variation of percentage dye removal with time.

Table 6.1: Operating conditions of batch reactor [Initial Dye concentration = 30 ppm]

Sl. No.	Parameter	range
1	Initial concentration (ppm)	30
2	рН	7
3	Inter Electrode Distance (cm)	2
4	Current Density (mA/cm <sup>2</sup> )	2.26
5	Electrolyte concentration (g/L)	2

Table 6.2: Variation in Dye concentration and percentage of dye reduction with time

[Initial Dye Concentration = 30 ppm]

Time (min)	Dye Concentration remaining (ppm)	Percentage Removal (%)
0	30.00	0.00
10	15.00	50.00
20	4.30	85.67
30	3.24	89.20
40	2.50	91.67
50	1.73	94.23
60	1.41	95.29
70	1.31	95.62
80	1.14	96.20
90	0.89	97.03

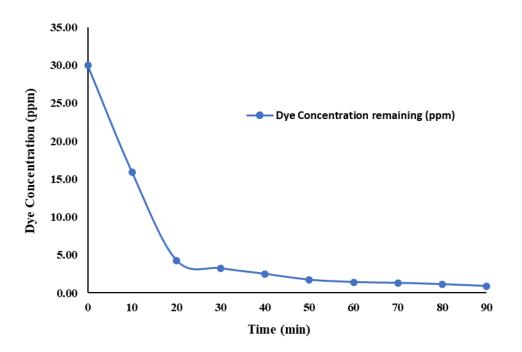


Fig. 6.1: Concentration of remaining Dye with time (30 ppm)

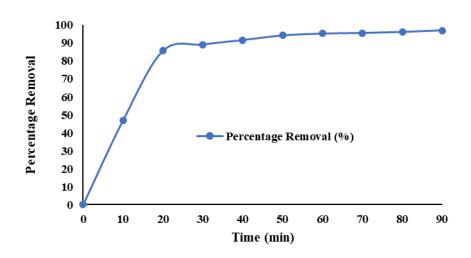


Fig. 6.2: Dye removal percentage with time (30 ppm)

#### **6.1.2** Initial Concentration = **20** ppm

The following table shows the variation of removal when initial concentration is 20 ppm. From the below table we can see that after 40 min almost 92.50 % of the dye was removed and after that rate of removal percentage was very low. After 90 min of batch timing, final removal percentage was almost

98.43 %. Figure 6.3 shows the variation of remaining dye concentration with time and figure 6.4 shows the variation of percentage dye removal with time.

Table 6.3: Operating conditions of batch reactor [Initial Dye Concentration = 20 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	20
2	рН	7
3	Inter Electrode Distance (cm)	2
4	Current Density (mA/cm <sup>2</sup> )	2.26
5	Electrolyte concentration (g/L)	2

Table 6.4: Variation in Dye concentration and percentage of dye reduction with time

[Initial Dye Concentration = 20 ppm]

Time	Dye Concentration	Percentage Removal
(min)	remaining (ppm)	(%)
0	20.00	0.00
10	8.52	57.39
20	3.15	84.25
30	2.14	89.29
40	1.50	92.50
50	0.79	96.07
60	0.44	97.82
70	0.34	98.32
80	0.31	98.43
90	0.31	98.43

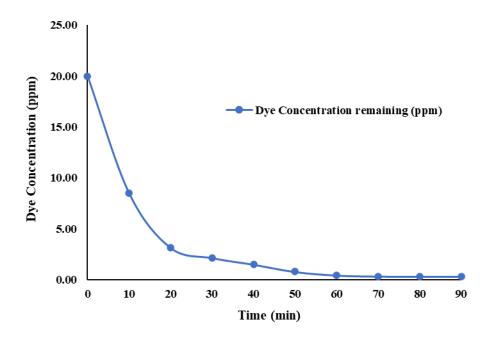


Fig. 6.3: Concentration of remaining Dye with time (20 ppm)

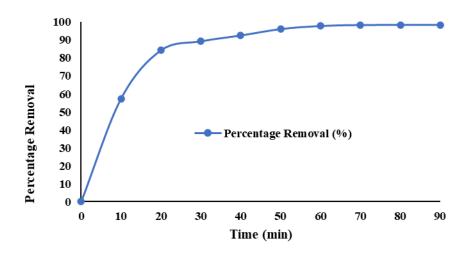


Fig. 6.4: Dye removal percentage with time (20 ppm)

#### 6.1.3 Initial Concentration = 10 ppm

The following table shows the variation of removal when initial concentration is 20 ppm. From the below table we can see that after 50 min almost 93.90 % dye was removed and after that rate of removal percentage was very low. After 90 min of batch timing, final removal percentage was almost 97.70 %. Figure 6.5 shows the variation of remaining dye concentration with time and figure 6.6 shows the variation of percentage dye removal with time.

Table 6.5: Operating conditions of batch reactor
[Initial Dye Concentration = 10 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	10
2	рН	7
3	Inter Electrode Distance (cm)	2
4	Current Density (mA/cm <sup>2</sup> )	2.26
5	Electrolyte concentration (g/L)	2

Table 6.6: Variation in Dye concentration and percentage of dye reduction with time

[Initial Dye Concentration = 10 ppm]

Time	Dye Concentration remaining	Percentage Removal
(min)	(ppm)	(%)
0	10.00	0.00
10	6.95	30.51
20	2.55	74.50
30	1.65	83.50
40	1.02	89.80
50	0.61	93.90
60	0.39	96.10
70	0.28	97.20
80	0.25	97.50
90	0.23	97.70

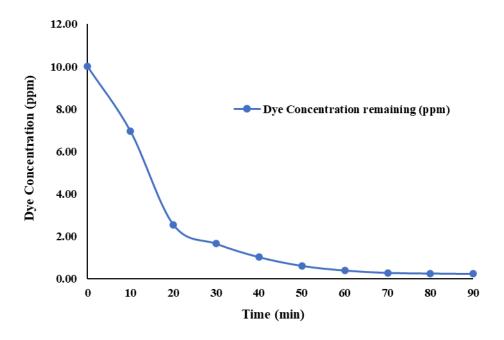


Fig. 6.5: Concentration of remaining Dye with time (10 ppm)

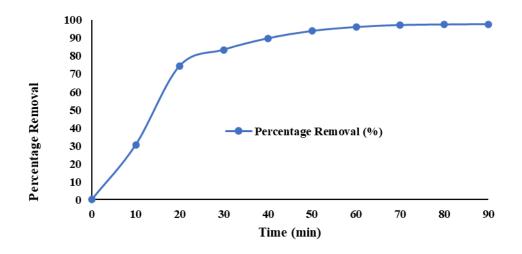


Fig. 6.6: Dye removal percentage with time (10 ppm)

**Outcome:** It is quite clear that under the present experimental conditions, in lower dye concentration removal efficiency is better. When initial concentration of dye was 30 ppm, after 50 min remaining dye concentration was 1.73 ppm whereas after same time, for initial concentration of 10 ppm, remaining dye concentration was 0.61 ppm. Increasing the initial concentration of dye required more electrolysis time for electro-coagulation of dye for achieving similar quality of water. The trend shows similarity with the results of **Do and Chen, 1994.** 

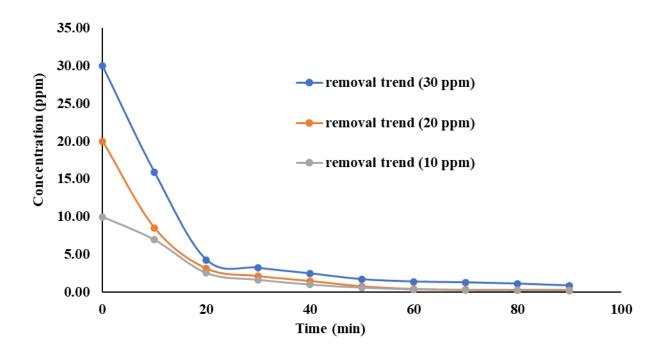


Fig. 6.7: Comparison of dye removal with respect to various initial concentrations

# 6.2 Variation of removal percentage with initial pH concentration

pH is an important parameter that influences the performance of the EC process. To examine the effect of pH, the dye solution was adjusted to the different initial pH ranging from 5 to 9 by adding 0.1 N NaOH or 0.1 N H<sub>2</sub>SO<sub>4</sub> solution. In the batch reactor, for testing purposes, 3 initial pH concentrations were taken. Apart from pH 7 (which is shown above) 2 other initial pH condition were pH 5 and pH 7.

During testing Current density was 2.26 mA/cm<sup>2</sup>. Electrolyte concentration was 2 g/L. Inter Electrode Distance was kept 2 cm. In each initial pH concentration, 3 different initial dye concentration was tested.

## 6.2.1 Initial pH = 5.0, Initial Dye Concentration = 30 ppm

The following table shows the variation of removal when initial concentration is 30 ppm. From the below table we can see that after 60 min almost 87.63 % of the dye was removed and after that rate of removal percentage

was very low. After 90 min of batch timing, final removal percentage was almost 89.93 %. Figure 6.8 shows the variation of remaining dye concentration with time at pH = 5 and figure 6.9 shows the variation of percentage dye removal with time.

Table 6.7: Operating conditions of batch reactor [Initial Dye Concentration = 30 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	30
2	рН	5
3	Inter Electrode Distance (cm)	2
4	Current Density (mA/cm²)	2.26
5	Electrolyte concentration (g/L)	2

Table 6.8: Variation in Dye concentration and percentage of dye reduction with time

[Initial Dye concentration= 30 ppm]

Time	Dye Concentration remaining	Percentage Removal
(min)	(ppm)	(%)
0	30.00	0.00
10	23.72	20.93
20	17.94	40.20
30	12.91	56.97
40	8.12	72.93
50	5.34	82.20
60	3.71	87.63
70	3.52	88.27
80	3.19	89.37
90	3.02	89.93

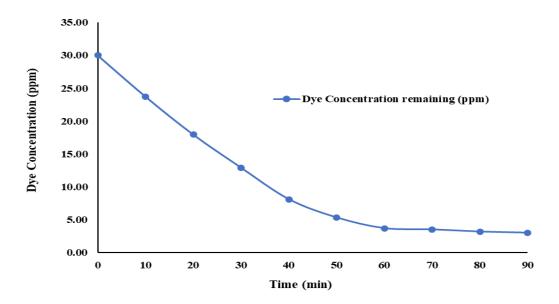


Fig. 6.8: Concentration of remaining Dye with time (Initial pH = 5, 30 ppm)

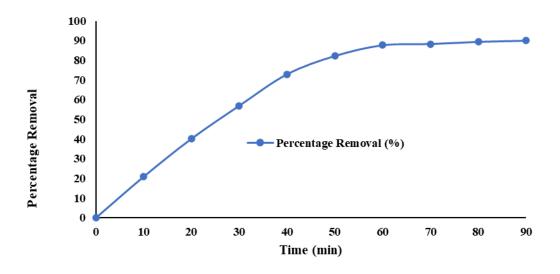


Fig. 6.9: Dye removal percentage with time (Initial pH = 5, 30 ppm)

# 6.2.2 Initial pH = 5.0, Initial Dye Concentration = 20 ppm

The following table shows the variation of removal when initial concentration is 30 ppm. From the below table we can see that after 60 min almost 86.15 % dye was removed and after that rate of removal percentage was

very low. After 90 min of batch timing, final removal percentage was almost 90.95 %. Figure 6.10 shows the variation of remaining dye concentration with time at pH = 5 and figure 6.11 shows the variation of percentage dye removal with time.

Table 6.9: Operating conditions of batch reactor [Initial Dye Concentration = 20 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	20
2	рН	5
3	Inter Electrode Distance (cm)	2
4	Current Density (mA/cm²)	2.26
5	Electrolyte concentration (g/L)	2

Table 6.10: Variation in Dye concentration and percentage of dye reduction with time

[Initial Dye Concentration = 20 ppm]

Time	Dye Concentration remaining	Percentage
(min)	(ppm)	Removal (%)
0	20.00	0.00
10	16.00	19.99
20	12.33	38.38
30	7.87	60.65
40	5.04	74.80
50	3.54	82.30
60	2.77	86.15
70	2.04	89.80
80	1.87	90.65
90	1.81	90.95

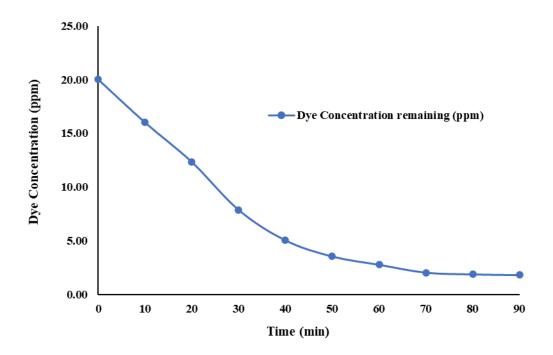


Fig. 6.10: Concentration of remaining Dye with time (Initial pH = 5, 20 ppm)

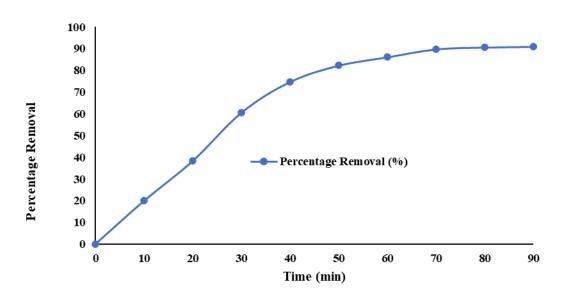


Fig. 6.11: Dye removal percentage with time (Initial pH = 5, 20 ppm)

## 6.2.3 Initial pH = 5.0, Initial Dye Concentration = 10 ppm

The following table shows the variation of removal when initial concentration is 30 ppm. From the below table we can see that after 60 min almost 81.10 % dye was removed and after that rate of removal percentage was very low. After 90 min of batch timing, final removal percentage was almost 88.40 %. Figure 6.12 shows the variation of remaining dye concentration with time at pH = 5 and figure 6.13 shows the variation of percentage dye removal with time.

Table 6.11: Operating conditions of batch reactor
[Initial Dye Concentration = 10 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	10
2	рН	5
3	Inter Electrode Distance (cm)	2
4	Current Density (mA/cm²)	2.26
5	Electrolyte concentration (g/L)	2

Table 6.12: Variation in Dye concentration and percentage of dye reduction with time [Initial Dye Concentration = 10 ppm]

Time (min)	Dye Concentration remaining (ppm)	Percentage Removal (%)
0	10.00	0.00
10	9.18	8.20
20	8.30	17.05
30	6.50	35.00
40	4.18	58.20
50	2.27	77.30
60	1.89	81.10
70	1.52	84.80
80	1.28	87.20
90	1.16	88.40

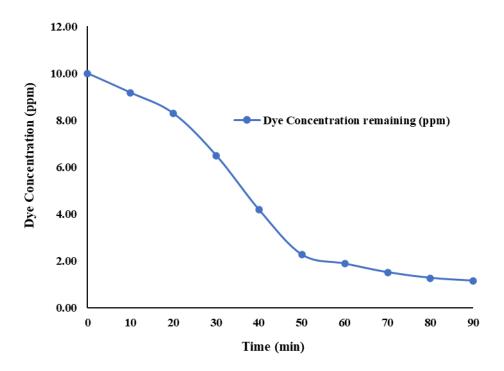


Fig. 6.12: Concentration of remaining Dye with time (Initial pH = 5, 10 ppm)

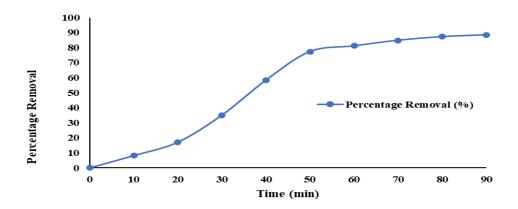


Fig. 6.13: Dye removal percentage with time (Initial pH = 5, 10 ppm)

## 6.2.4 Initial pH = 9.0, Initial Dye Concentration = 30 ppm

The following table shows the variation of removal when initial concentration is 30 ppm. From the below table we can see that after 60 min almost 88.20 % dye was removed and after that rate of removal percentage was

very low. After 90 min of batch timing, final removal percentage was almost 91.60 %. Figure 6.14 shows the variation of remaining dye concentration with time at pH = 9 and figure 6.15 shows the variation of percentage dye removal with time.

Table 6.13: Operating conditions of batch reactor [Initial Dye Concentration = 30 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	30
2	рН	9
3	Inter Electrode Distance (cm)	2
4	Current Density (mA/cm²)	2.26
5	Electrolyte concentration (g/L)	2

Table 6.14: Variation in Dye concentration and percentage of dye reduction with time [Initial Dye Concentration = 30 ppm]

Time	Dye Concentration remaining	Percentage Removal
(min)	(ppm)	(%)
0	30	0.00
10	21.33	28.90
20	15.54	48.20
30	11.19	62.70
40	7.57	74.77
50	4.38	85.40
60	3.54	88.20
70	3.18	89.40
80	2.78	90.73
90	2.52	91.60

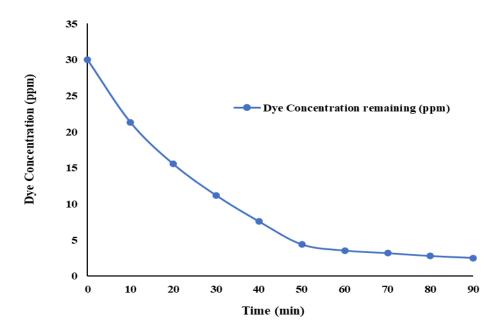


Fig. 6.14: Concentration of remaining Dye with time (Initial pH = 9, 30 ppm)

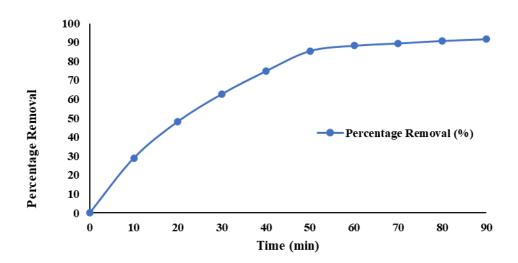


Fig. 6.15: Dye removal percentage with time (Initial pH = 9, 30 ppm)

# **6.2.5** Initial pH = 9.0, Initial Dye Concentration = 20 ppm

The following table shows the variation of removal when initial concentration is 20 ppm. From the below table we can see that after 60 min

almost 89.30 % dye was removed and after that rate of removal percentage was very low. After 90 min of batch timing, final removal percentage was almost 91.90 %. Figure 6.16 shows the variation of remaining dye concentration with time at pH = 9 and figure 6.17 shows the variation of percentage dye removal with time.

Table 6.15: Operating conditions of batch reactor [Initial Dye Concentration = 20 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	20
2	pН	9
3	Inter Electrode Distance (cm)	2
4	Current Density (mA/cm²)	2.26
5	Electrolyte concentration (g/L)	2

Table 6.16: Variation in Dye concentration and percentage of dye reduction with time [Initial Dye Concentration = 20 ppm]

Time	Dye Concentration remaining	Percentage
(min)	(ppm)	Removal (%)
0	20.00	0.00
10	12.52	37.42
20	8.69	56.55
30	5.38	73.10
40	3.32	83.40
50	2.82	85.90
60	2.14	89.30
70	1.98	90.10
80	1.68	91.60
90	1.62	91.90

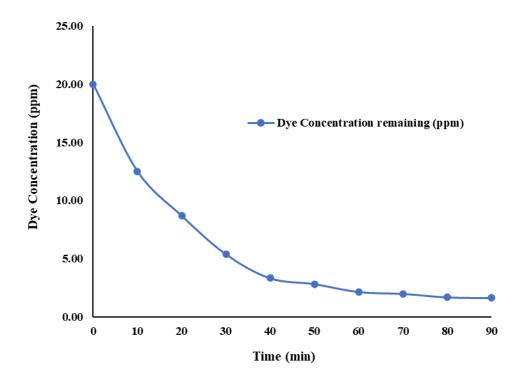


Fig. 6.16: Concentration of remaining Dye with time (Initial pH = 9, 20 ppm)

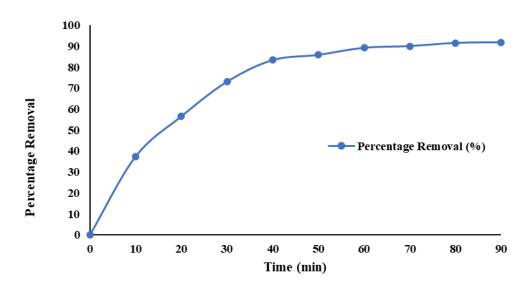


Fig. 6.17: Dye removal percentage with time (Initial pH = 9, 20 ppm)

## 6.2.6 Initial pH = 9.0, Initial Dye Concentration = 10 ppm

The following table shows the variation of removal when initial concentration is 10 ppm. From the below table we can see that after 60 min almost 89.30 % dye was removed and after that rate of removal percentage was very low. After 90 min of batch timing, final removal percentage was almost 91.90 %. Figure 6.18 shows the variation of remaining dye concentration with time at pH = 9 and figure 6.19 shows the variation of percentage dye removal with time.

Table 6.17: Operating conditions of batch reactor
[Initial Dye Concentration = 10 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	10
2	рН	9
3	Inter Electrode Distance (cm)	2
4	Current Density (mA/cm²)	2.26
5	Electrolyte concentration (g/L)	2

Table 6.18: Variation in Dye concentration and percentage of dye reduction with time [Initial Dye Concentration = 10 ppm]

Time	Dye Concentration remaining	Percentage Removal
(min)	(ppm)	(%)
0	10.00	0.00
10	7.93	20.70
20	5.64	43.60
30	3.98	60.20
40	2.54	74.60
50	2.02	79.80
60	1.64	83.60
70	1.38	86.20
80	1.19	88.10
90	1.1	89.00

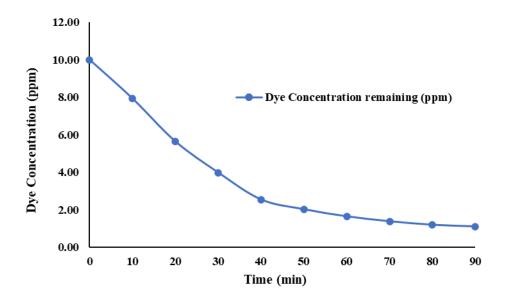


Fig. 6.18: Concentration of remaining Dye with time
(Initial pH = 9, 10 ppm)

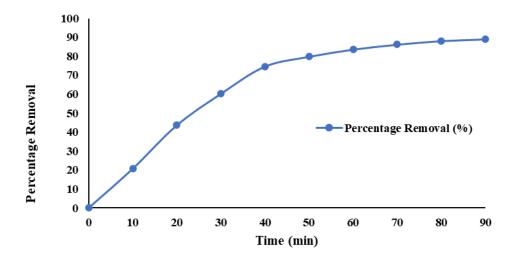


Fig. 6.19: Dye removal percentage with time (Initial pH = 9, 10 ppm)

**Outcome:** From the above-mentioned graphical representations it is clear that higher removal efficiency is achieved at pH 7. At pH 7, removal is more than 97% whereas in case of pH 5 and 9, removal is almost 89% and 91%. Graphical representation of removal at all 3 pH is shown below

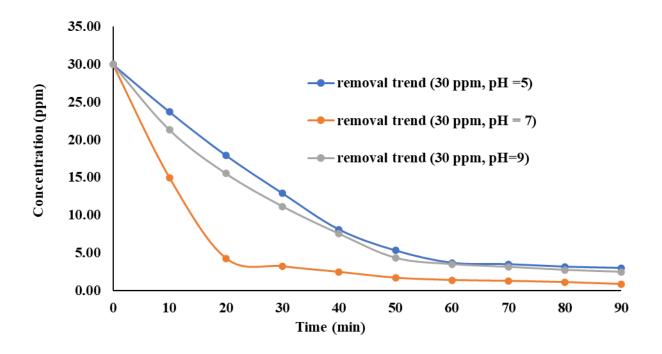


Fig. 6.20: Comparison of dye removal with respect to initial concentration 30ppm

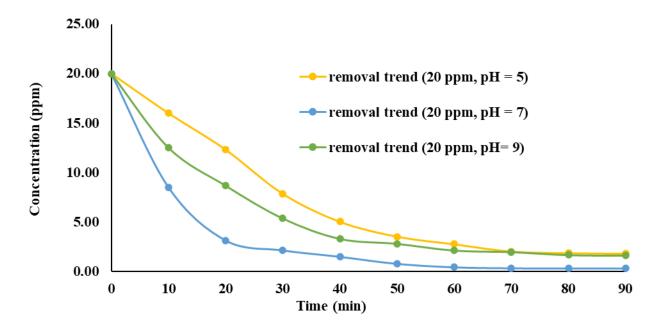


Fig. 6.21: Comparison of dye removal with respect to initial concentration 20 ppm

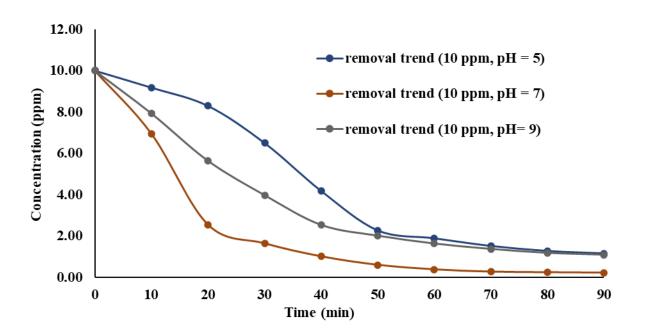


Fig. 6.22: Comparison of dye removal with respect to initial concentration 10 ppm

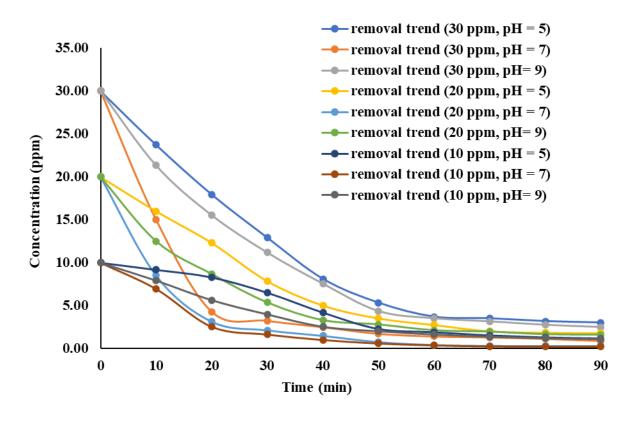


Fig. 6.23: Overall Comparison of dye removal on different pH

## 6.3 Variation of removal percentage with current density

Current density is an important parameter that influences the performance of the EC process. Current density was measured from the input current of cathode and anode. Input current is divided by submerged plate area to find the current density.

In the batch reactor, for testing purpose, 3 current densities were taken. Apart from current density 2.26 (which is shown above) 2 other conditions were -

- i) current density = 1.41 mA/cm<sup>2</sup>
- ii) Current density = 3.11 mA/cm<sup>2</sup>

During testing initial pH of the solution 7. Electrolyte concentration was 2 g/L. Inter Electrode Distance was kept at 2 cm. In each condition, 3 different initial dve concentration was tested.

## 6.3.1 Current density = $1.41 \text{ mA/cm}^2$ , Initial Dye Concentration = 30 ppm

The following table shows the variation of removal when initial concentration is 30 ppm. From the below table we can see that after 60 min almost 90 % dye was removed and after that rate of removal percentage was very low. After 90 min of batch timing, final removal percentage was almost 92 %. Figure 6.24 shows the variation of remaining dye concentration with time at and figure 6.25 shows the variation of percentage dye removal with time.

Table 6.19: Operating conditions of batch reactor [Initial Dye Concentration = 30 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	30
2	pН	7
3	Inter Electrode Distance (cm)	2
4	Current Density (mA/cm <sup>2</sup> )	1.41
5	Electrolyte concentration (g/L)	2

Table 6.20: Variation in Dye concentration and percentage of dye reduction with time [Initial Dye Concentration = 30 ppm]

Time	Dye Concentration remaining	Percentage Removal
(min)	(ppm)	(%)
0	30.00	0.00
10	23.32	22.27
20	16.10	46.35
30	10.05	66.50
40	6.50	78.32
50	3.46	88.47
60	2.95	90.16
70	2.79	90.70
80	2.41	91.97
90	2.39	92.03

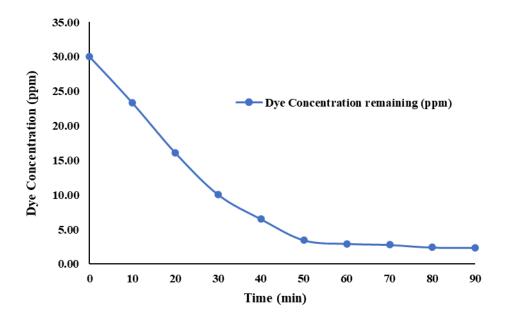


Fig. 6.24: Concentration of remaining Dye with time

(Current density = 1.41 mA/cm<sup>2</sup>, 30 ppm)

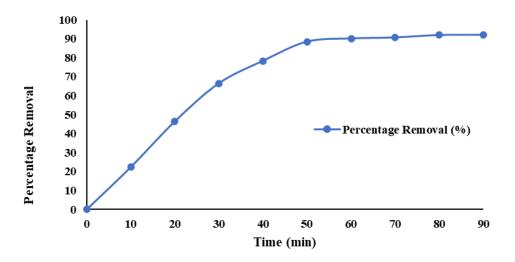


Fig. 6.25: Dye removal percentage with time (Current density = 1.41 mA/cm², 30 ppm)

## 6.3.2 Current density = 1.41 mA/cm<sup>2</sup>, Initial Dye Concentration = 20 ppm

The following table shows the variation of removal when initial concentration is 20 ppm. From the below table we can see that after 50 min almost 90 % dye was removed and after that rate of removal percentage was very low. After 90 min of batch timing, final removal percentage was almost 94.95 %. Figure 6.26 shows the variation of remaining dye concentration with time at and figure 6.27 shows the variation of percentage dye removal with time.

Table 6.21: Operating conditions of batch reactor [Initial Dye Concentration = 20 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	20
2	рН	7
3	Inter Electrode Distance (cm)	2
4	Current Density (mA/cm²)	1.41
5	Electrolyte concentration (g/L)	2

Table 6.22: Variation in Dye concentration and percentage of dye reduction with time [Initial Dye Concentration = 20 ppm]

Time (min)	Dye Concentration remaining	Percentage Removal
Time (min)	(ppm)	(%)
0	20.00	0.00
10	15.34	23.30
20	10.91	45.46
30	7.08	64.61
40	3.58	82.08
50	1.86	90.70
60	1.25	93.75
70	1.19	94.05
80	1.03	94.85
90	1.01	94.95

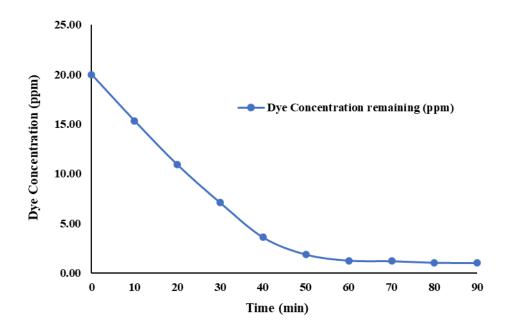


Fig. 6.26: Concentration of remaining Dye with time (Current density = 1.41 mA/cm², 20 ppm)

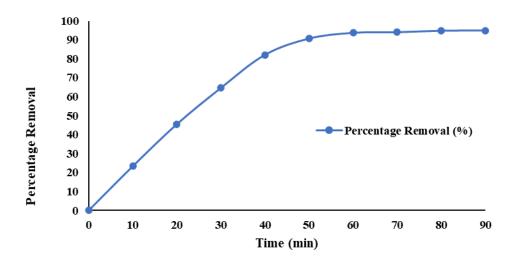


Fig. 6.27: Dye removal percentage with time (Current density = 1.41 mA/cm<sup>2</sup>, 20 ppm)

## 6.3.3 current density = 1.41 mA/cm<sup>2</sup>, Initial Dye Concentration = 10 ppm

The following table shows the variation of removal when initial concentration is 10 ppm. From the below table we can see that after 50 min almost 90 % dye was removed and after that rate of removal percentage was very low. After 90 min of batch timing, final removal percentage was almost 92 %. Figure 6.28 shows the variation of remaining dye concentration with time at and figure 6.29 shows the variation of percentage dye removal with time.

Table 6.23: Operating conditions of batch reactor [Initial Dye Concentration = 10 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	10
2	рН	7
3	Inter Electrode Distance (cm)	2
4	Current Density (mA/cm <sup>2</sup> )	1.41
5	Electrolyte concentration (g/L)	2

Table 6.24: Variation in Dye concentration and percentage of dye reduction with time

[Initial Dye Concentration = 10 ppm]

Time	Dye Concentration remaining	Percentage Removal
(min)	(ppm)	(%)
0	10.00	0.00
10	8.11	18.90
20	5.62	43.80
30	3.95	60.50
40	1.83	81.72
50	1.06	89.40
60	0.95	90.50
70	0.89	91.10
80	0.85	91.50
90	0.80	92.00

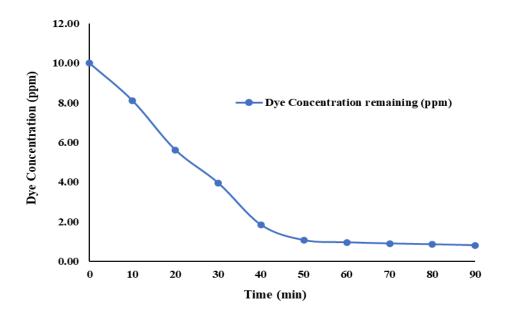


Fig. 6.28: Concentration of remaining Dye with time (Current density =  $1.41 \text{ mA/cm}^2$ , 10 ppm)

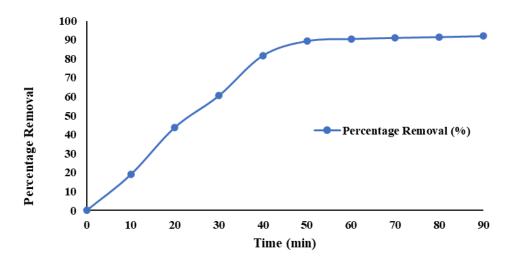


Fig. 6.29: Dye removal percentage with time (Current density = 1.41 mA/cm², 10 ppm)

## 6.3.4 Current density = 3.11 mA/cm<sup>2</sup>, Initial Dye Concentration = 30 ppm

The following table shows the variation of removal when initial concentration is 30 ppm. From the below table we can see that after 30 min almost 93.17 % dye was removed and after that rate of removal percentage was very low. After 90 min of batch timing, final removal percentage was almost 99 %. Figure 6.30 shows the variation of remaining dye concentration with time at and figure 6.31 shows the variation of percentage dye removal with time.

Table 6.25: Operating conditions of batch reactor [Initial Dye Concentration = 30 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	30
2	рН	7
3	Inter Electrode Distance (cm)	2
4	Current Density (mA/cm <sup>2</sup> )	3.11
5	Electrolyte concentration (g/L)	2

Table 6.26: Variation in Dye concentration and percentage of dye reduction with time [Initial Dye Concentration = 30 ppm]

Time	Dye Concentration remaining	Percentage Removal
(min)	(ppm)	(%)
0	30.00	0.00
10	10.91	63.63
20	2.92	90.27
30	2.05	93.17
40	1.91	93.62
50	1.34	95.54
60	0.68	97.72
70	0.45	98.51
80	0.36	98.80
90	0.27	99.11

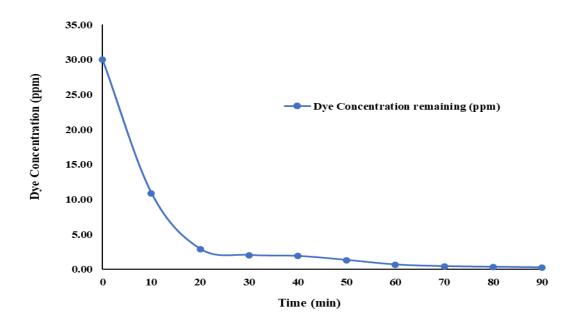


Fig. 6.30: Concentration of remaining Dye with time (Current density = 3.11 mA/cm², 30 ppm)

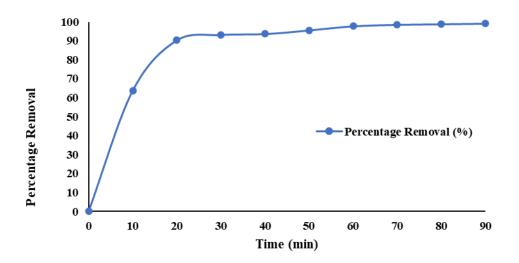


Fig. 6.31: Dye removal percentage with time

(Current density =  $3.11 \text{ mA/cm}^2$ , 30 ppm)

#### 6.3.5 Current density = 3.11 mA/cm<sup>2</sup>, Initial Dye Concentration = 20 ppm

The following table shows the variation of removal when initial concentration is 20 ppm. From the below table we can see that after 20 min almost 94.54 % dye was removed and after that rate of removal percentage was very low. After 90 min of batch timing, final removal percentage was almost 99.23 %. Figure 6.32 shows the variation of remaining dye concentration with time at and figure 6.33 shows the variation of percentage dye removal with time.

Table 6.27: Operating conditions of batch reactor [Initial Dye Concentration = 20 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	20
2	рН	7
3	Inter Electrode Distance (cm)	2
4	Current Density (mA/cm <sup>2</sup> )	3.11
5	Electrolyte concentration (g/L)	2

Table 6.28: Variation in Dye concentration and percentage of dye reduction with time [Initial Dye Concentration = 20 ppm]

Time	Dye Concentration remaining	Percentage Removal
(min)	(ppm)	(%)
0	20.00	0.00
10	5.70	71.50
20	1.09	94.54
30	0.79	96.06
40	0.70	96.52
50	0.56	97.20
60	0.31	98.44
70	0.28	98.61
80	0.17	99.17
90	0.15	99.23

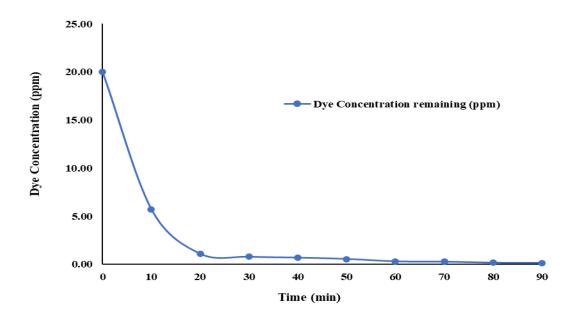


Fig. 6.32: Concentration of remaining Dye with time (Current density = 3.11 mA/cm², 20 ppm)

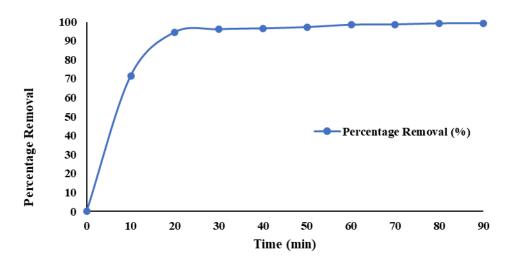


Fig. 6.33: Dye removal percentage with time (Current density = 3.11 mA/cm², 20 ppm)

#### 6.3.6 Current density = 3.11 mA/cm<sup>2</sup>, Initial Dye Concentration = 10 ppm

The following table shows the variation of removal when initial concentration is 10 ppm. From the below table we can see that after 30 min almost 97.29 % dye was removed and after that rate of removal percentage was very low. After 90 min of batch timing, final removal percentage was almost 99.70 %. Figure 6.34 shows the variation of remaining dye concentration with time at and figure 6.35 shows the variation of percentage dye removal with time.

Table 6.29: Operating conditions of batch reactor [Initial Dye Concentration = 10 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	10
2	рН	7
3	Inter Electrode Distance (cm)	2
4	Current Density (mA/cm <sup>2</sup> )	3.11
5	Electrolyte concentration (g/L)	2

Table 6.30: Variation in Dye concentration and percentage of dye reduction with time [Initial Dye Concentration = 10 ppm]

Time	Dye Concentration remaining	Percentage Removal
(min)	(ppm)	(%)
0	10.00	0.00
10	3.76	62.39
20	0.70	92.97
30	0.27	97.29
40	0.19	98.10
50	0.10	98.96
60	0.08	99.18
70	0.07	99.29
80	0.05	99.50
90	0.03	99.70

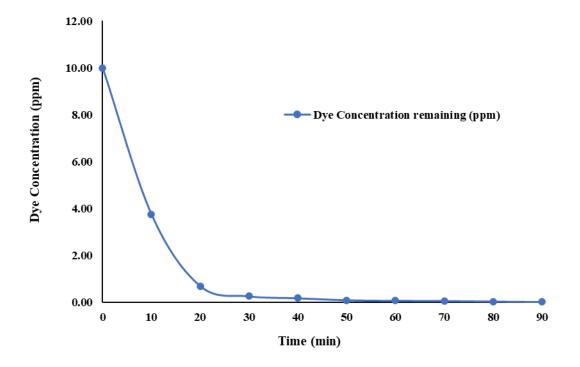


Fig. 6.34: Concentration of remaining Dye with time (Current density = 3.11 mA/cm², 10 ppm)

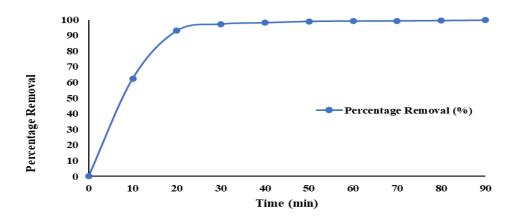


Fig. 6.35: Dye removal percentage with time

(Current density =  $3.11 \text{ mA/cm}^2$ , 10 ppm)

Outcome: Current density determines the coagulant production rate, adjusts the rate and size of the bubble production and hence affects the growth of flocs (Daneshvar et al. 2003; Mollah et al. 2004). The effect of current density on the efficiency of colour removal by electrocoagulation process was carried out using various current densities. From the above figure, it is glaring that as the current density is increased, the rate of colour removal also increased. Increasing current density results in a corresponding increase in the production of coagulant in the solution leading to high efficiency. A graphical comparison of the removal at different current density is shown below.

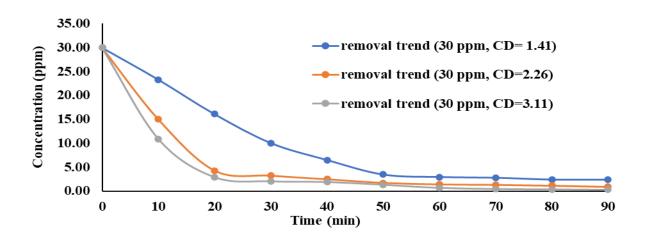


Fig. 6.36: Comparison of dye removal with respect to initial concentration 30ppm

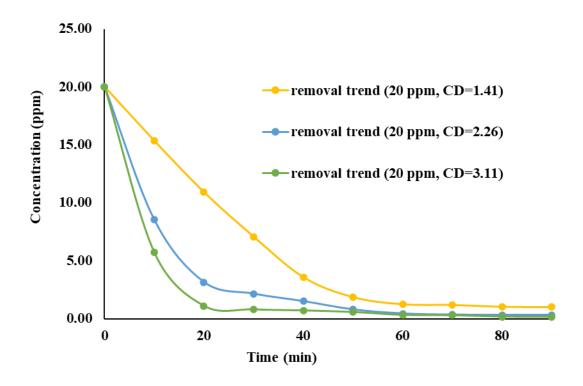


Fig. 6.37: Comparison of dye removal with respect to initial concentration 20 ppm

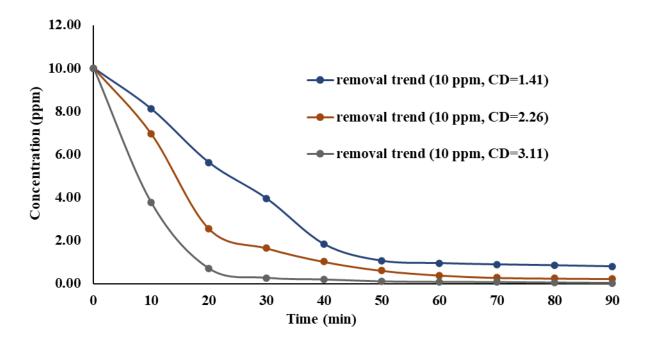


Fig. 6.38: Comparison of dye removal with respect to initial concentration 10 ppm

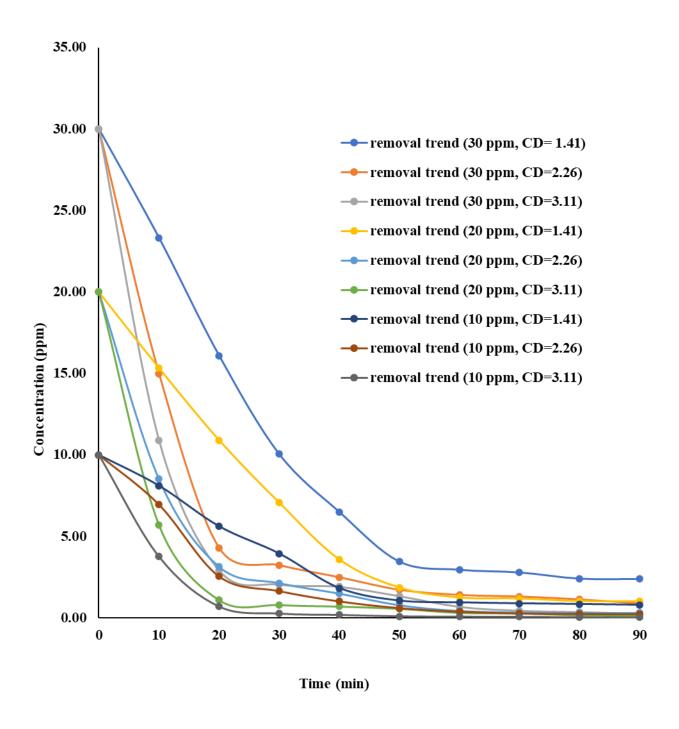


Fig. 6.39: Overall Comparison of dye removal on different current density

# 6.4 Variation of removal percentage with Inter Electrode Distance (IED)

One of the major process parameters of EC process is Inter Electrode Distance (IED). Removal efficiency varies significantly with changes in this parameter. In the batch reactor, 3 Inter Electrode Distance (IED) were taken for testing purposes.

- i) IED = 2 cm
- ii) IED = 4 cm
- iii) IED = 6 cm

During testing Current density was 2.26 mA/cm<sup>2</sup>. Electrolyte concentration was 2 g/L. pH value was 7. Among the 3 mentioned IED, the results when IED = 2 cm is discussed already.

#### 6.4.1 IED = 4 cm, Initial Concentration = 30 ppm

The following table shows the variation of removal when initial concentration is 30 ppm. From the below table we can see that after 70 min almost 90.53 % dye was removed and after that rate of removal percentage was very low. After 90 min of batch timing, final removal percentage was almost 93.61 %. Figure 6.40 shows the variation of remaining dye concentration with time at and figure 6.41 shows the variation of percentage dye removal with time.

Table 6.31: Operating conditions of batch reactor [Initial Dye Concentration = 30 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	30
2	pН	7
3	Inter Electrode Distance (cm)	4
4	Current Density (mA/cm2)	2.26
5	Electrolyte concentration (g/L)	2

Table 6.32: Variation in Dye concentration and percentage of dye reduction with time [Initial Dye Concentration = 30 ppm]

Time	Dye Concentration remaining	Percentage Removal
(min)	(ppm)	(%)
0	30.00	0.00
10	24.32	18.93
20	17.64	41.20
30	12.92	56.93
40	7.98	73.40
50	5.85	80.50
60	3.98	86.73
70	2.84	90.53
80	2.08	93.08
90	1.92	93.61

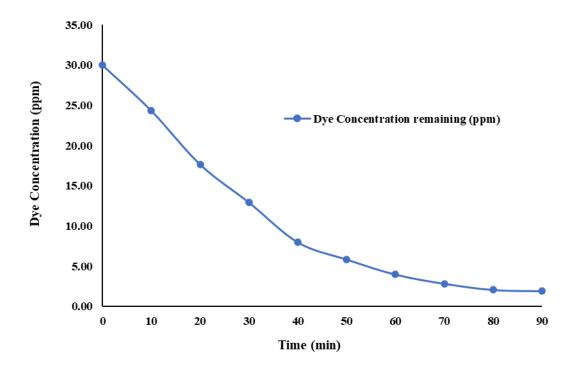


Fig. 6.40: Concentration of remaining Dye with time (IED = 4 cm, Concentration = 30 ppm)

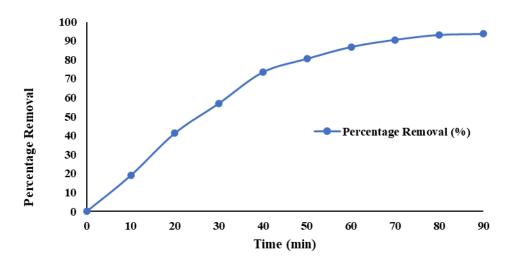


Fig. 6.41: Dye removal percentage with time (IED = 4 cm, Concentration = 30 ppm)

#### **6.4.2 IED = 4 cm, Initial Concentration = 20 ppm**

The following table shows the variation of removal when initial concentration is 20 ppm. From the below table we can see that after 70 min almost 90.98 % dye was removed and after that rate of removal percentage was very low. After 90 min of batch timing, final removal percentage was almost 91.95 %. Figure 6.42 shows the variation of remaining dye concentration with time at and figure 6.43 shows the variation of percentage dye removal with time.

Table 6.33: Operating conditions of batch reactor [Initial Dye Concentration = 20 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	20
2	рН	7
3	Inter Electrode Distance (cm)	4
4	Current Density (mA/cm <sup>2</sup> )	2.26
5	Electrolyte concentration (g/L)	2

Table 6.34: Variation in Dye concentration and percentage of dye reduction with time [Initial Dye Concentration = 20 ppm]

Time	Dye Concentration remaining	Percentage Removal
(min)	(ppm)	(%)
0	20.00	0.00
10	16.90	15.50
20	12.32	38.40
30	8.42	57.92
40	4.43	77.87
50	3.26	83.70
60	2.13	89.34
70	1.80	90.98
80	1.69	91.55
90	1.61	91.95

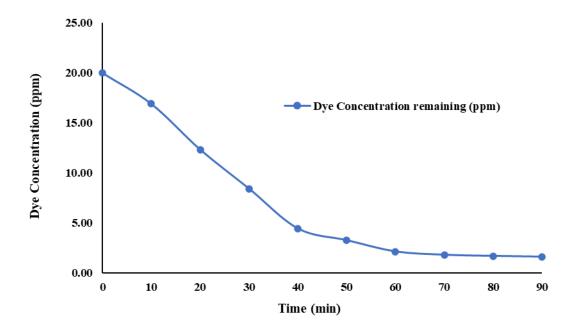


Fig. 6.42: Concentration of remaining Dye with time (IED = 4 cm, Concentration = 20 ppm)

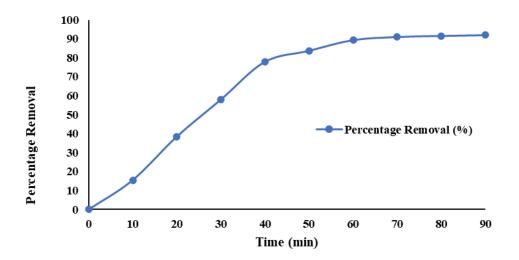


Fig. 6.43: Dye removal percentage with time

(IED = 4 cm, Concentration = 20 ppm)

#### 6.4.3 IED = 4 cm, Initial Concentration = 10 ppm

The following table shows the variation of removal when initial concentration is 10 ppm. From the below table we can see that after 70 min almost 87.43 % dye was removed and after that rate of removal percentage was very low. After 90 min of batch timing, final removal percentage was almost 89.07 %. Figure 6.44 shows the variation of remaining dye concentration with time at and figure 6.45 shows the variation of percentage dye removal with time.

Table 6.35: Operating conditions of batch reactor [Initial Dye Concentration = 10 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	10
2	рН	7
3	Inter Electrode Distance (cm)	4
4	Current Density (mA/cm <sup>2</sup> )	2.26
5	Electrolyte concentration (g/L)	2

Table 6.36: Variation in Dye concentration and percentage of dye reduction with time [Initial Dye Concentration = 10 ppm]

Time	Dye Concentration remaining	Percentage Removal
(min)	(ppm)	(%)
0	10.00	0.00
10	8.22	17.80
20	6.15	38.50
30	4.12	58.80
40	2.19	78.14
50	1.64	83.61
60	1.42	85.79
70	1.26	87.43
80	1.20	87.98
90	1.09	89.07

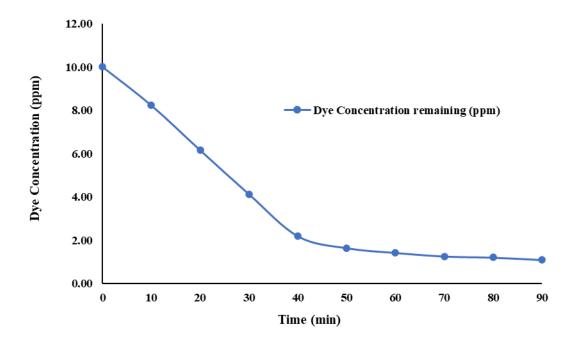


Fig. 6.44: Concentration of remaining Dye with time

(IED = 4 cm, Concentration = 10 ppm)

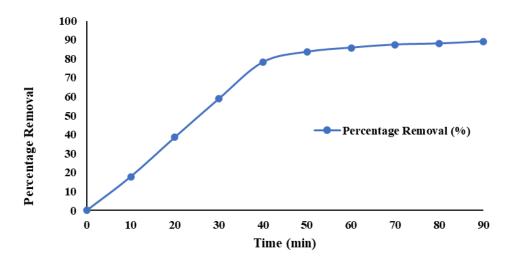


Fig. 6.45: Dye removal percentage with time (IED = 4 cm, Concentration = 10 ppm)

#### **6.4.4 IED = 6 cm, Initial Concentration = 30 ppm**

The following table shows the variation of removal when initial concentration is 30 ppm. From the below table we can see that after 90 min of batch timing, final removal percentage was almost 50.07 %. Figure 6.46 shows the variation of remaining dye concentration with time at and figure 6.47 shows the variation of percentage dye removal with time.

Table 6.37: Operating conditions of batch reactor [Initial Dye Concentration = 30 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	30
2	рН	7
3	Inter Electrode Distance (cm)	6
4	Current Density (mA/cm <sup>2</sup> )	2.26
5	Electrolyte concentration (g/L)	2

Table 6.38: Variation in Dye concentration and percentage of dye reduction with time [Initial Dye Concentration = 30 ppm]

Time	Dye Concentration remaining	Percentage Removal
(min)	(ppm)	(%)
0	30.00	0.00
10	29.23	2.57
20	28.79	4.03
30	27.65	7.83
40	26.22	12.60
50	23.55	21.50
60	18.64	37.87
70	16.87	43.77
80	15.39	48.70
90	14.98	50.07

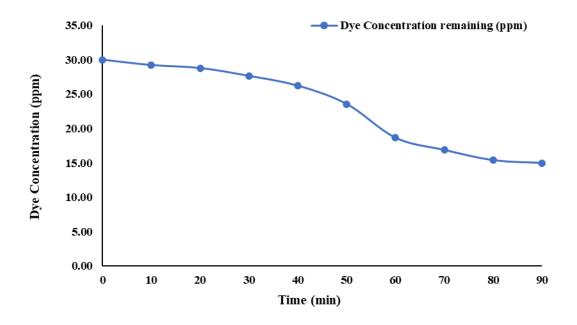


Fig. 6.46: Concentration of remaining Dye with time (IED = 6 cm, Concentration = 30 ppm)

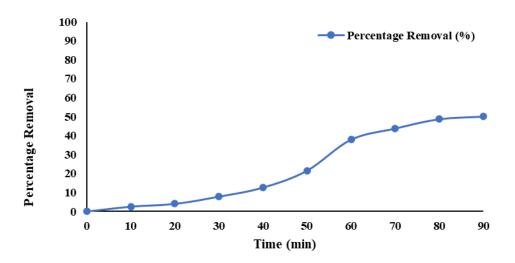


Fig. 6.47: Dye removal percentage with time (IED = 6 cm, Concentration = 30 ppm)

## 6.4.5 IED = 6.0 cm, Initial Concentration = 20 ppm

The following table shows the variation of removal when initial concentration is 20 ppm. From the below table we can see that after 90 min of batch timing, final removal percentage was almost 54.95 %. Figure 6.48 shows the variation of remaining dye concentration with time at and figure 6.49 shows the variation of percentage dye removal with time.

Table 6.39: Operating conditions of batch reactor [Initial Dye Concentration = 20 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	20
2	pН	7
3	Inter Electrode Distance (cm)	6
4	Current Density (mA/cm <sup>2</sup> )	2.26
5	Electrolyte concentration (g/L)	2

Table 6.40: Variation in Dye concentration and percentage of dye reduction with time [Initial Dye Concentration = 20 ppm]

Time	Dye Concentration remaining	Percentage Removal
(min)	(ppm)	(%)
0	20.00	0.00
10	18.80	6.01
20	18.47	7.65
30	17.81	10.93
40	17.27	13.66
50	15.85	20.77
60	12.39	38.05
70	10.84	45.80
80	9.81	50.95
90	9.01	54.95

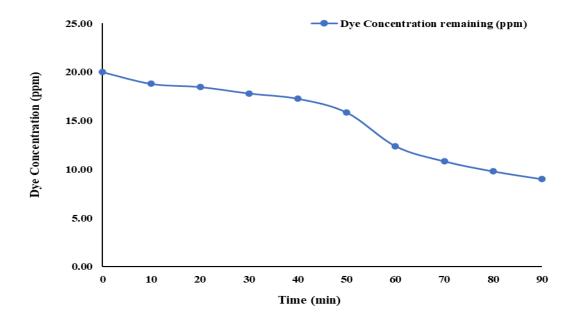


Fig. 6.48: Concentration of remaining Dye with time (IED = 6 cm, Concentration = 20 ppm)

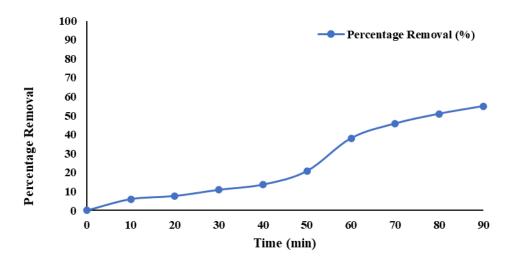


Fig. 6.49: Dye removal percentage with time

(IED = 6 cm, Concentration = 20 ppm)

#### 6.4.6 IED = 6 cm, Initial Concentration = 10 ppm

The following table shows the variation of removal when initial concentration is 10 ppm. From the below table we can see that after 90 min of batch timing, final removal percentage was almost 40.90 %. Figure 6.50 shows the variation of remaining dye concentration with time at and figure 6.51 shows the variation of percentage dye removal with time.

Table 6.41: Operating conditions of batch reactor [Initial Dye Concentration = 10 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	10
2	рН	7
3	Inter Electrode Distance (cm)	6
4	Current Density (mA/cm²)	2.26
5	Electrolyte concentration (g/L)	2

Table 6.42: Variation in Dye concentration and percentage of dye reduction with time [Initial Dye Concentration = 10 ppm]

Time	Dye Concentration remaining	Percentage Removal
(min)	(ppm)	(%)
0	10.00	0.00
10	9.91	0.87
20	9.84	1.64
30	9.56	4.37
40	8.85	11.48
50	8.36	16.39
60	7.81	21.86
70	6.67	33.33
80	6.56	34.43
90	5.91	40.90

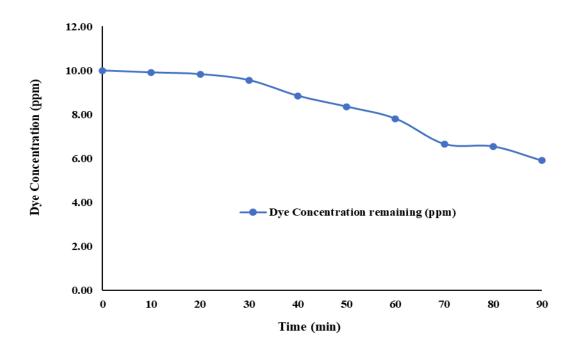


Fig. 6.50: Concentration of remaining Dye with time (IED = 6 cm, Concentration = 10 ppm)

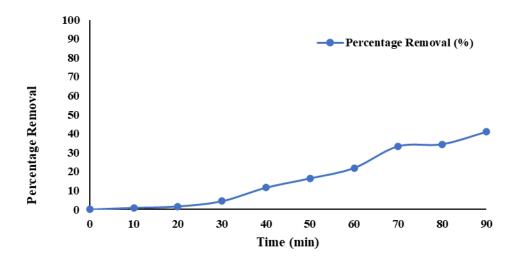


Fig. 6.51: Dye removal percentage with time (IED = 6 cm, Concentration = 10 ppm)

**Outcome:** From the above figures we can see that removal efficiency decreases significantly when IED increases. For initial concentration of 10 ppm, removal efficiency was 40.90 % when IED was 6 cm. For same initial concentration, removal efficiency was 97.70 % when IED was 2cm. In higher IED, less number of electrodes were there in the reactor which leads to production of less coagulant. A comparative graphical representation of removal efficiency is shown below.

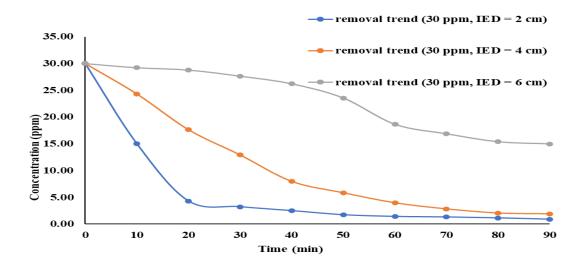


Fig. 6.52: Comparison of dye removal with respect to initial concentration 30 ppm

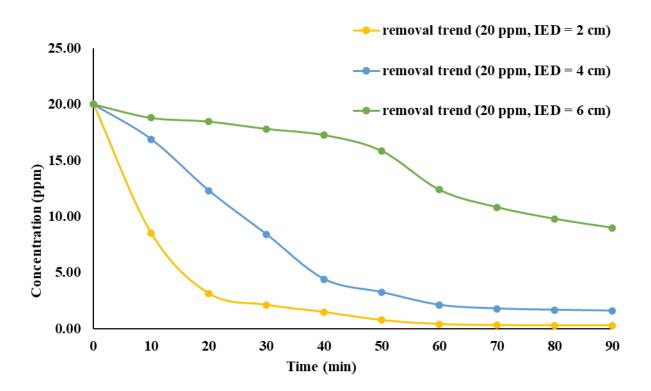


Fig. 6.53: Comparison of dye removal with respect to initial concentration 20 ppm

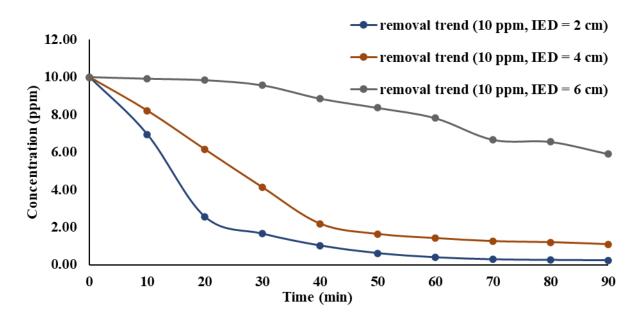


Fig. 6.54: Comparison of dye removal with respect to initial concentration 10 ppm

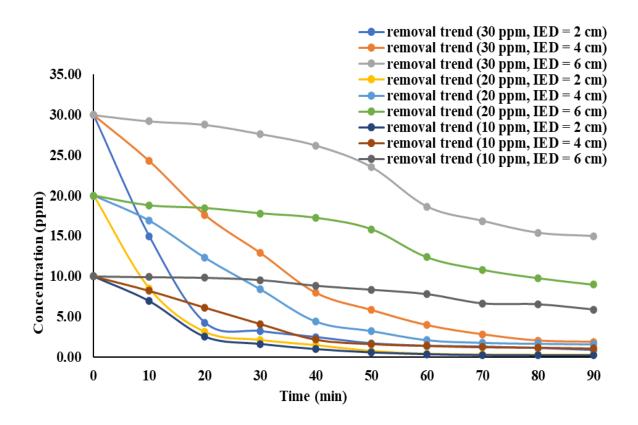


Fig. 6.55: Overall Comparison of dye removal on different IED

# 6.5 Variation of removal percentage with respect to electrolyte concentration

Electrolyte concentration plays a significant role in the process of electrocoagulation. To investigate the effect of electrolyte concentration on the electrocoagulation removal efficiencies of RhB, experiments were performed on solutions containing a constant loading of RhB 30 ppm, at current density of 2.26 mA/cm², inter electrode distance 2 cm, pH 7, while the concentrations of NaCl were varied. 3 concentrations were taken for experimental purpose.

- i) Nacl concentration = 1 g/l
- ii) Nacl concentration = 2 g/l
- iii) Nacl concentration = 3 g/l

#### 6.5.1 Electrolyte concentration 1 gm/l, Initial Dye Concentration = 30 ppm

The following table shows the variation of removal when initial concentration is 30 ppm and electrolyte concentration is 1 g/l. From the below table we can see that after 90 min of batch timing, final removal percentage was almost 90.53 %. Figure 6.56 shows the variation of remaining dye concentration

with time at and figure 6.57 shows the variation of percentage dye removal with time.

Table 6.43: Operating conditions of batch reactor [Initial Dye Concentration = 30 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	30
2	рН	7
3	Inter Electrode Distance (cm)	2
4	Current Density (mA/cm <sup>2</sup> )	2.26
5	Electrolyte concentration (g/L)	1

Table 6.44: Variation in Dye concentration and percentage of dye reduction with time [Initial Dye Concentration = 30 ppm]

Time (min)	Dye Concentration remaining (ppm)	Percentage Removal (%)
0	30.00	0.00
10	26.60	11.33
20	18.40	38.66
30	12.86	57.13
40	6.89	77.05
50	5.31	82.30
60	4.26	85.79
70	3.46	88.47
80	2.30	92.35
90	1.84	93.87

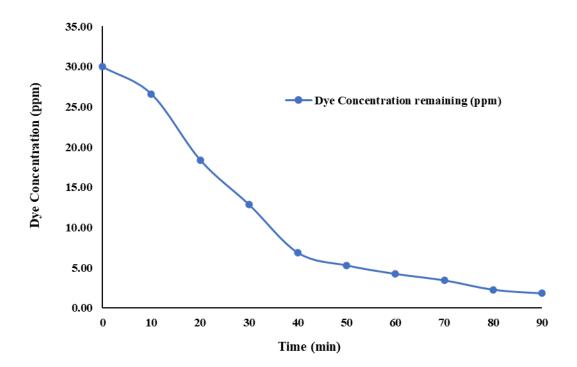


Fig. 6.56: Concentration of remaining Dye with time (Electrolyte concentration = 1 g/L, dye concentration = 30 ppm)

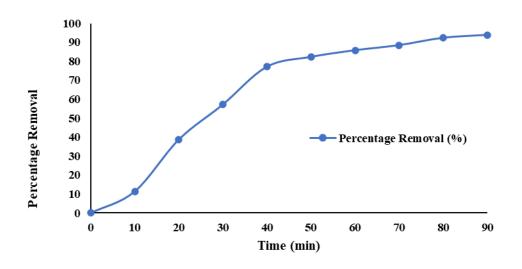


Fig. 6.57: Dye removal percentage with time (Electrolyte concentration = 1 g/L, dye concentration = 30 ppm)

# 6.5.2 Electrolyte concentration 2 gm/l, Initial Dye Concentration = 30 ppm

The following table shows the variation of removal when initial concentration is 30 ppm and electrolyte concentration is 2 g/l. From the below table we can see that after 90 min of batch timing, final removal percentage was almost 90.53 %. Figure 6.58 shows the variation of remaining dye concentration with time at and figure 6.59 shows the variation of percentage dye removal with time.

Table 6.45: Operating conditions of batch reactor [Initial Dye Concentration = 30 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	30
2	рН	7
3	Inter Electrode Distance (cm)	2
4	Current Density (mA/cm <sup>2</sup> )	2.26
5	Electrolyte concentration (g/L)	2

Table 6.46: Variation in Dye concentration and percentage of dye reduction with time [Initial Dye Concentration = 30 ppm]

Time (min)	Dye Concentration remaining (ppm)	Percentage Removal (%)
0	30.00	0.00
10	15.00	50.00
20	4.30	85.67
30	3.24	89.20
40	2.50	91.67
50	1.73	94.23
60	1.41	95.29
70	1.31	95.62
80	1.14	96.20
90	0.89	97.03

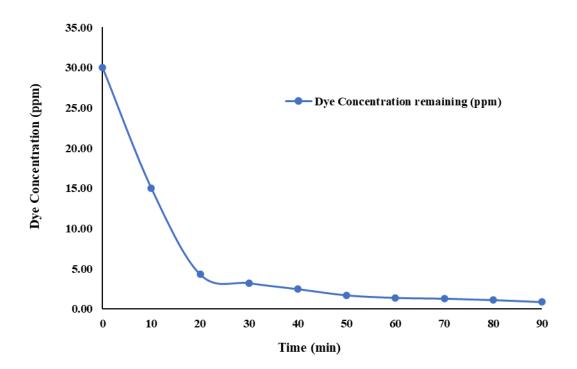


Fig. 6.58: Concentration of remaining Dye with time (Electrolyte concentration = 2 g/L, dye concentration = 30 ppm)

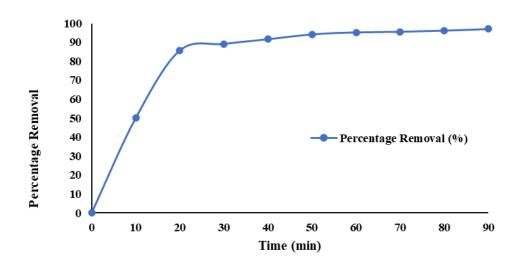


Fig. 6.59: Dye removal percentage with time (Electrolyte concentration = 2 g/L, dye concentration = 30 ppm)

#### 6.5.3 Electrolyte concentration 3 gm/l, Initial Dye Concentration = 30 ppm

The following table shows the variation of removal when initial concentration is 30 ppm and electrolyte concentration is 3 g/l. From the below table we can see that after 90 min of batch timing, final removal percentage was almost 97.20 %. Figure 6.60 shows the variation of remaining dye concentration with time at and figure 6.61 shows the variation of percentage dye removal with time.

Table 6.47: Operating conditions of batch reactor [Initial Dye Concentration = 30 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	30
2	рН	7
3	Inter Electrode Distance (cm)	2
4	Current Density (mA/cm <sup>2</sup> )	2.26
5	Electrolyte concentration (g/L)	2

Table 6.48: Variation in Dye concentration and percentage of dye reduction with time [Initial Dye Concentration = 30 ppm]

Time	Dye Concentration remaining	Percentage Removal
(min)	(ppm)	(%)
0	30.00	0.00
10	13.98	53.40
20	4.12	86.27
30	3.09	89.70
40	2.15	92.83
50	1.64	94.53
60	1.32	95.60
70	1.25	95.83
80	1.09	96.37
90	0.84	97.20

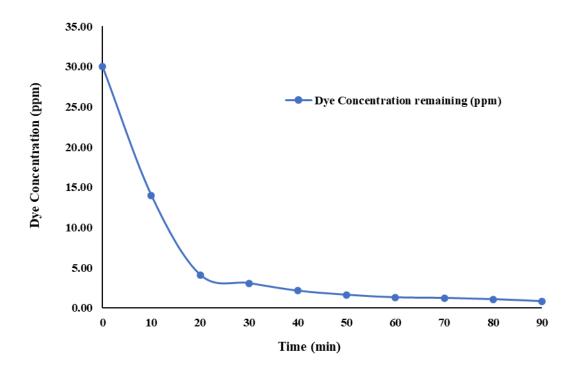


Fig. 6.60: Concentration of remaining Dye with time (Electrolyte concentration = 3 g/L, dye concentration = 30 ppm)

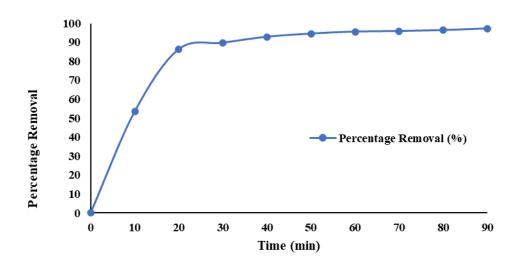


Fig. 6.61: Dye removal percentage with time (Electrolyte concentration = 3 g/L, dye concentration = 30 ppm)

Outcome: Solution conductivity influences the current efficiency, cell voltage and consumption of electrical energy in electrolytic cells. The use of NaCl to increase solution conductivity is also accompanied by the production of chloride ions that reduce the effects of other anions leading to ohmic resistance of the electrochemical cell (Daneshvar et al. 2006). Above data shows that the colour removal efficiency also increases from 90% to 97 % as the electrolyte concentration changes to 2 g/L from 1 g/L, and a further increase in electrolyte concentration beyond these values does not significantly affect the removal efficiency of the dye from the solution. A comparative graphical representation of removal efficiency is shown below.

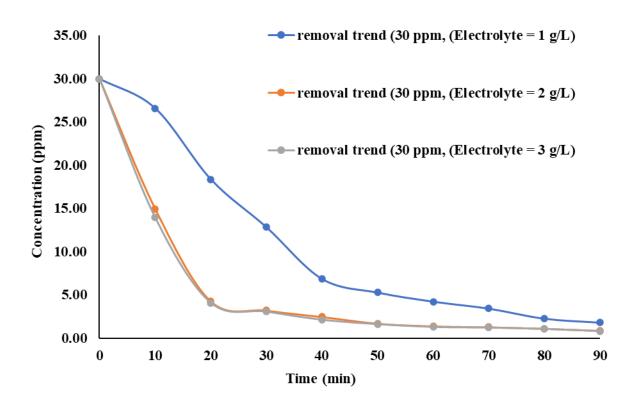


Fig. 6.62: Overall Comparison of dye removal on different electrolyte concentrations

#### 6.6 Variation of removal percentage with respect to Turbulence

Turbulence in the reactor effects seriously in the removal of pollutant. The removal of Rhodamine B was conducted both in stagnant phase and turbulent

phase. Aeration was used to create the turbulence. A pump was used for aeration purposes. Below result shows the removal trend when aeration is on.

#### **6.6.1** Initial Dye Concentration = 30 ppm, with aeration

The following table shows the variation of removal when initial concentration is 30 ppm, electrolyte concentration is 2 g/l and aeration is on. From the below table we can see that after 90 min of batch timing, final removal percentage was almost 90.53 %. Figure 6.63 shows the variation of remaining dye concentration with time at and figure 6.64 shows the variation of percentage dye removal with time.

Table 6.49: Operating conditions of batch reactor [Initial Dye Concentration = 30 ppm]

Sl. No.	Parameter	Range
1	Initial concentration (ppm)	30
2	pН	7
3	Inter Electrode Distance (cm)	2
4	Current Density (mA/cm <sup>2</sup> )	2.26
5	Electrolyte concentration (g/L)	2
6	Aeration	On

Table 6.50: Variation in Dye concentration and percentage of dye reduction with time [Initial Dye Concentration = 30 ppm]

Time (min)	Dye Concentration remaining (ppm)	Percentage Removal
0	30.00	0.00
10	25.92	13.60
20	23.84	20.53
30	20.76	30.80
40	17.39	42.03
50	16.15	46.17
60	15.03	49.90
70	13.95	53.50
80	13.84	53.87
90	13.29	55.70

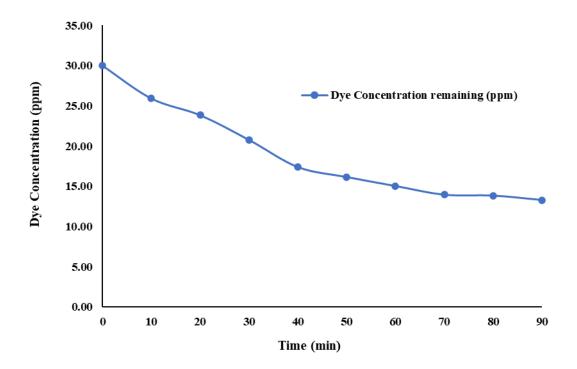


Fig. 6.63: Concentration of remaining Dye with time
(With aeration, concentration = 30 ppm)

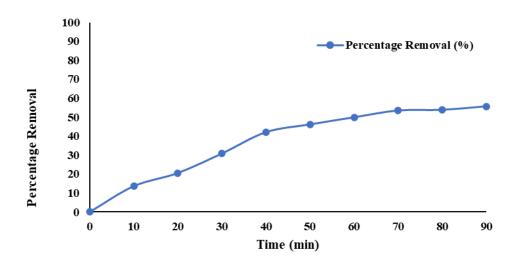


Fig. 6.64: Dye removal percentage with time (With aeration, concentration = 30 ppm)

If we compare the result with removal in stagnation condition, it is observed that without aeration condition gives better result. The comparison is shown in the below figure.

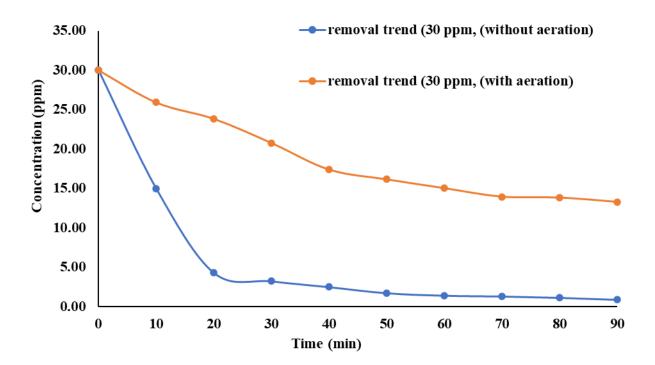


Fig. 6.65: Overall Comparison of dye removal with and without aeration

## **Chapter-7**

### **Conclusion**

The following conclusions are drawn from the present experimental investigation work.

- 1) Rhodamine B can be removed from the aquas solution by Electro Coagulation technique in a feasible way.
- 2) From the result obtained we can conclude that best operating parameter for the reactor is
  - I) pH = 7
  - II) Inter Electrode Distance = 2 cm
  - III) Current density =  $2.23 \text{ mA/cm}^2$
  - IV) Electrolyte concentration = 2 g/L
- 3) Higher current density can produce a better-quality effluent in less time but that will not be cost benefit.
- 4) Electrolyte concentration more than 2 g/L will not produce a significant better-quality effluent.
- 5) Aeration will not help effectively in removal pf Rhodamine B.

### **Chapter-8**

## <u>Limitation of the present study and future</u> <u>scopes</u>

A serious effort has been endeavored to achieve the target objectives of this study, yet this present study is not devoid of certain limitations.

- The present study is conducted in a batch mode. Efficiency of the reactor is not obtained in continuous mode.
- pH of the solution was measured in the initial stage. There was no such arrangement for measurement of pH continuously.
- Due to less available time and fixed reactor configuration aeration was
  used for creating the turbulence. A magnetic stirrer with rpm control
  equipment can produce a controlled turbulence. In that condition change
  in efficiency of the reactor was not conducted.
- Different combination of anode and cathode material (Al-Fe, Fe-Al, Al-Al) can be used in future and obtained result can be studied for better efficiency.

### **Chapter-9**

### References

- Abu Ghalwa, N. M., & Abdel-Latif, M. S. (2005). Electrochemical degradation of acid green dye in aqueous wastewater dyestuff solutions using a lead oxide coated titanium electrode. *Journal of the Iranian* Chemical Society, 2, 238-243.
- Ahangarnokolaei, M. A., Ganjidoust, H., & Ayati, B. (2018). Optimization
  of parameters of electrocoagulation/flotation process for removal of Acid
  Red 14 with mesh stainless steel electrodes. *Journal of Water Reuse and Desalination*, 8(2), 278-292.
- Akar, S. T., Özcan, A. S., Akar, T., Özcan, A., & Kaynak, Z. (2009).
   Biosorption of a reactive textile dye from aqueous solutions utilizing an agro-waste. *Desalination*, 249(2), 757-761.
- Aleboyeh, A., Daneshvar, N., & Kasiri, M. B. (2008). Optimization of CI Acid Red 14 azo dye removal by electrocoagulation batch process with response surface methodology. *Chemical Engineering and Processing: Process Intensification*, 47(5), 827-832.
- Al-Raad, A. A., Hanafiah, M. M., Naje, A. S., & Ajeel, M. A. (2020).
   Optimized parameters of the electrocoagulation process using a novel reactor with rotating anode for saline water treatment. *Environmental Pollution*, 265, 115049.
- Anwer, H., Mahmood, A., Lee, J., Kim, K. H., Park, J. W., & Yip, A. C. (2019). Photocatalysts for degradation of dyes in industrial effluents: Opportunities and challenges. *Nano Research*, 12, 955-972.
- Avlonitis, S. A., Poulios, I., Sotiriou, D., Pappas, M., & Moutesidis, K. (2008). Simulated cotton dye effluents treatment and reuse by nanofiltration. *Desalination*, 221(1–3), 259–267. <a href="https://doi.org/10.1016/j.desal.2007.01.082">https://doi.org/10.1016/j.desal.2007.01.082</a>

- Burkinshaw, S. M., & Son, Y.-A. (2010). The dyeing of supermicrofibre nylon with acid and vat dyes. *Dyes and Pigments*, 87(2), 132–138. https://doi.org/10.1016/j.dyepig.2010.03.009
- Chiu, Y. H., Chang, T. F. M., Chen, C. Y., Sone, M., & Hsu, Y. J. (2019).
   Mechanistic insights into photodegradation of organic dyes using heterostructure photocatalysts. *Catalysts*, 9(5), 430.
- Clark, M. (2011). *Handbook of textile and industrial dyeing*. Woodhead Publishing Limited. https://doi.org/10.1533/9780857094919
- CRINI, G. (2006). Non-conventional low-cost adsorbents for dye removal:
   A review. Bioresource Technology, 97(9), 1061–1085.
   <a href="https://doi.org/10.1016/j.biortech.2005.05.001">https://doi.org/10.1016/j.biortech.2005.05.001</a>
- Daneshvar, N., Ayazloo, M., Khataee, A. R., & Pourhassan, M. (2007).
   Biological decolorization of dye solution containing Malachite Green by microalgae Cosmarium sp. *Bioresource Technology*, 98(6), 1176–1182.
   <a href="https://doi.org/10.1016/j.biortech.2006.05.025">https://doi.org/10.1016/j.biortech.2006.05.025</a>
- Do, J. S., & Chen, M. L. (1994). Decolourization of dye-containing solutions by electrocoagulation. *Journal of applied electrochemistry*, 24(8), 785-790.
- El Qada, E. N., Allen, S. J., & Walker, G. M. (2008). Adsorption of basic dyes from aqueous solution onto activated carbons. *Chemical Engineering Journal*, 135(3), 174–184. <a href="https://doi.org/10.1016/j.cej.2007.02.023">https://doi.org/10.1016/j.cej.2007.02.023</a>
- Farias, R. S. de, Buarque, H. L. de B., Cruz, M. R. da, Cardoso, L. M. F., Gondim, T. de A., & Paulo, V. R. de. (2018). Adsorption of congo red dye from aqueous solution onto amino-functionalized silica gel. *Engenharia Sanitaria e Ambiental*, 23(6), 1053–1060. <a href="https://doi.org/10.1590/s1413-41522018172982">https://doi.org/10.1590/s1413-41522018172982</a>
- García-Martinez, J., Becerra Moreno, D., ARTEAGA CELIS, B. L., OCHOA RANGEL, Y. E. & Ramirez, L. (2020). Coupling of heterogeneous photocatalysis and aerobic biological process of activated sludge to treat

- wastewater containing Chlorpyrifos. Revista Ingeniería Y Competitividad, 22(1 (2020)), 1-12.
- Gozálvez-Zafrilla, J. M., Sanz-Escribano, D., Lora-García, J., & León Hidalgo, M. C. (2008). Nanofiltration of secondary effluent for wastewater reuse in the textile industry. *Desalination*, 222(1–3), 272–279. https://doi.org/10.1016/j.desal.2007.01.173
- Hernández-Zamora, M., Martínez-Jerónimo, F., Cristiani-Urbina, E., & Cañizares-Villanueva, R. O. (2016). Congo red dye affects survival and reproduction in the cladoceran Ceriodaphnia dubia. Effects of direct and dietary exposure. *Ecotoxicology*, 25(10), 1832–1840. <a href="https://doi.org/10.1007/s10646-016-1731-x">https://doi.org/10.1007/s10646-016-1731-x</a>
- Kobya, M., Demirbas, E., Can, O. T., & Bayramoglu, M. (2006). Treatment
  of levafix orange textile dye solution by electrocoagulation. *Journal of*hazardous materials, 132(2-3), 183-188.
- Kos, L. (2016). Use of Chitosan for Textile Wastewater Decolourization. Fibres and Textiles in Eastern Europe, 24(3(117)), 130–135. https://doi.org/10.5604/12303666.1196623
- Kumar, V. (2017). A review on the feasibility of electrolytic treatment of wastewater: Prospective and constraints. *constraints*, 2(1), 52-62.
- Li, Y., Yan, X., Hu, X., Feng, R., and Zhou, M., 2019, Trace pyrolyzed ZIF-67 loaded activated carbon pellets for enhanced adsorption and catalytic degradation of Rhodamine B in water, Chem. Eng. J., 375, 122003.
- McCall, I. C., Betanzos, A., Weber, D. A., Nava, P., Miller, G. W., & Parkos, C. A. (2009). Effects of phenol on barrier function of a human intestinal epithelial cell line correlate with altered tight junction protein localization. *Toxicology and Applied Pharmacology*, 241(1), 61–70. https://doi.org/10.1016/j.taap.2009.08.002
- Mishra, G., Tripathy, M., 1993. A critical review of the treatments for decolourization of textile effluent. Colourage 40, 35-38.

- Modirshahla, N., Behnajady, M. A., & Kooshaiian, S. (2007). Investigation
  of the effect of different electrode connections on the removal efficiency of
  Tartrazine from aqueous solutions by electrocoagulation. Dyes and
  pigments, 74(2), 249-257.
- Mohan, D., Singh, K. P., Singh, G., & Kumar, K. (2002). Removal of dyes from wastewater using flyash, a low-cost adsorbent. *Industrial & engineering chemistry research*, 41(15), 3688-3695.
- Mollah, M. Y., Morkovsky, P., Gomes, J. A., Kesmez, M., Parga, J., & Cocke, D. L. (2004). Fundamentals, present and future perspectives of electrocoagulation. *Journal of hazardous materials*, 114(1-3), 199-210.
- Mollah, Mohammad YA, Saurabh R. Pathak, Prashanth K. Patil, Madhavi Vayuvegula, Tejas S. Agrawal, Jewel AG Gomes, Mehmet Kesmez, and David L. Cocke. "Treatment of orange II azo-dye by electrocoagulation (EC) technique in a continuous flow cell using sacrificial iron electrodes." Journal of hazardous materials 109, no. 1-3 (2004): 165-171.
- Nekouei Marnani, N., and Shahbazi, A., 2019, A novel environmental-friendly nanobiocomposite synthesis by EDTA and chitosan functionalized magnetic graphene oxide for high removal of Rhodamine B: Adsorption mechanism and separation property, Chemosphere, 218, 715–725.
- Nidheesh, P. V., Oladipo, A. A., Yasri, N. G., Laiju, A. R., Cheela, V. S., Thiam, A., ... & Roberts, E. T. P. (2022). Emerging applications, reactor design and recent advances of electrocoagulation process. *Process Safety* and Environmental Protection.
- Nigam, P., Banat, I. M., Singh, D., & Marchant, R. (1996). Microbial process for the decolorization of textile effluent containing azo, diazo and reactive dyes. *Process biochemistry*, 31(5), 435-442.
- Ögütveren, Ü. B., Gönen, N., & Koparal, S. (1992). Removal of dye stuffs from waste water: Electrocoagulation of Acilan Blau using soluble anode. *Journal of Environmental Science & Health Part A*, 27(5), 1237-

1247.

- Pascariu, P., Airinei, A., Olaru, N., Olaru, L., and Nica, V., 2016,
   Photocatalytic degradation of Rhodamine B dye using ZnO-SnO2 electrospun ceramic nanofibers, Ceram. Int., 42 (6), 6775–6781.
- Rajeshkannan, R., M. Rajasimman, and N. Rajamohan. "Removal of malachite green from aqueous solution using hydrilla verticillataoptimization, equilibrium and kinetic studies." *International Journal of Environmental and Ecological Engineering* 4.1 (2010): 23-30.
- Sadik, M. A. (2019). A review of promising electrocoagulation technology for the treatment of wastewater. *Advances in Chemical Engineering and Science*, 9(01), 109.
- Sanroman, M. A., Pazos, M., Ricart, M. T., & Cameselle, C. (2004).
   Electrochemical decolourisation of structurally different dyes. Chemosphere, 57(3), 233-239.
- Sarkar Phyllis, A. K., Tortora, G., & Johnson, I. (2022). Photodegradation.
   The Fairchild Books Dictionary of Textiles.
   https://doi.org/10.5040/9781501365072.12105
- Selvarani K (2000), Studies on Low cost Adsorbents for the removal of Organic and Inorganics from Water, Ph D., Thesis, Regional Engineering College, Tiruchirapalli.
- Sharma, J., Sharma, S., & Soni, V. (2021). Classification and impact of synthetic textile dyes on Aquatic Flora: A review. Regional Studies in Marine Science, 45, 101802. <a href="https://doi.org/10.1016/j.rsma.2021.101802">https://doi.org/10.1016/j.rsma.2021.101802</a>
- Sharma, J., Sharma, S., Bhatt, U., & Soni, V. (2022). Toxic effects of Rhodamine B on antioxidant system and photosynthesis of Hydrilla verticillata. *Journal of Hazardous Materials Letters*, 3(August), 100069. <a href="https://doi.org/10.1016/j.hazl.2022.100069">https://doi.org/10.1016/j.hazl.2022.100069</a>
- Silveira, E., Marques, P. P., Silva, S. S., Lima-Filho, J. L., Porto, A. L. F.,
   & Tambourgi, E. B. (2009). Selection of Pseudomonas for industrial textile

- dyes decolourization. International Biodeterioration & Biodegradation, 63(2), 230-235.
- Szpyrkowicz, L., Juzzolino, C., & Kaul, S. N. (2001). A comparative study on oxidation of disperse dyes by electrochemical process, ozone, hypochlorite and Fenton reagent. Water research, 35(9), 2129-2136.
- Taheri, M., Moghaddam, M. R. A., & Arami, M. (2014). A comparative study on removal of four types of acid azo dyes using electrocoagulation process. *Environmental Engineering & Management Journal* (EEMJ), 13(3).
- Yu, Y., Wang, C., Luo, L., Wang, J., and Meng, J., 2018, An environment-friendly route to synthesize pyramid-like g-C3N4 arrays for efficient degradation of Rhodamine B under visible-light irradiation, Chem. Eng. J., 334, 1869–1877.
- Zazou, H., Afanga, H., Akhouairi, S., Ouchtak, H., Addi, A. A., Akbour, R.
   A., & Hamdani, M. (2019). Treatment of textile industry wastewater by electrocoagulation coupled with electrochemical advanced oxidation process. *Journal of Water Process Engineering*, 28, 214-221.