SEPARATION OF RHODAMINE B USING NANO-FILTRATION PROCESS

A DISSERTATION

SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE AWARD OF THE DEGREE

MASTER OF SCIENCE

IN

CHEMISTRY

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CANDIDATE'S DECLARATION

We, (Atul Patel, 2k21/Mscche/08; Abhishek Mishra 2k21/Mscche/01) students of M.Sc. (Applied Chemistry), hereby declare that the project Dissertation titled "Nano-Filtration of Rhodamine B" which is submitted by us to the Department of Applied Chemistry, Delhi Technological University, Delhi in partial fulfilment of the requirement for the award of the degree of Master of Science, is original and not copied from any source without proper citation. This work has not previously formed the basis for the award of any Degree, Diploma Associateship, Fellowship or other similar title or recognition.

Place: Delhi **Atul Patel**

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CERTIFICATE

I hereby certify that the Project Dissertation titled "Nano-Filtration of Rhodamine B" which is submitted by [Atul Patel, 2k21/Mscche/08; Abhishek Mishra 2k21/Mscche/01)] students of M.Sc. (Applied Chemistry) Delhi Technological University, Delhi in partial fulfilment of the requirement for the award of the degree of Master of Science, is a record of the project work carried out by the students under my supervision. To the best of my knowledge this work has not been submitted in part or full for any Degree or Diploma to this University or elsewhere.

Place: Delhi **DR. MANISH JAIN**

Date: 23rd May 2023 **SUPERV ISOR**

ABSTRACT

With increasing industrialisation, dyes have become one of the most promising colourants used worldwide on a large scale. It has been estimated that around 12-15 % of these dyes are discharged as effluents into the water bodies. One of such dyes is Rhodamine B which is used widely in textile and food colouring. These dyes are harmful, toxic and carcinogenic in nature. They cause pollution of the water bodies, death of the aquatic organisms, reduce the light penetration into the water hence lesser under water photosynthesis and many more severe problems. Therefore, it becomes important to filter them before discharging.

In the present investigation, nano-filtration of Rhodamine-B dye was done using membrane HF-150. Effect of different parameters on the rejection rate of the dye such as the amount of Rhodamine B, flow rate of the feed solution, pressure of the system was studied. Furthermore, volume and flux of the permeate solution was calculated and its relation with above mentioned parameters was also investigated. Dye removal efficiency of the membrane was measured in terms of rejection percentage. The rejection percentage was found to be around 90%. The findings of the work can be of promising application in dye removal and waste water purification.

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CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1

INTRODUCTION

Various dyes are used on industrial level for coloring textiles, papers, food stuffs, plastic etc. These industries after processing the dye release these effluents into the water bodies. Currently, water contamination because of failure of multiple industries to dispose of their waste water having significant content of dye is one of the major challenges that effects the whole world. About 60,000 tons of dyes are discharged into the environment in the form of waste each year worldwide[1], of which 80% are azo dyes. Among all dyes manufactured across the world, 11% goes out as waste in water discharge, 2% from production and as much as 9% from coloring[2] . Every year, 64,000 tons of dyes is manufactured by India of which 7,040 tones are directly discharged into the environment. These colored dye effluents contain compounds which aren't easy to treat biologically due to their non-biodegradable nature. The dye effluents may contain some components or moieties that could be toxic, carcinogenic or mutagenic to aquatic life[3] . These dyes also degrade the quality of water bodies by increasing biochemical and chemical oxygen demand, thereby disturbing photosynthesis, stopping plant growth, entering the food chain, providing recalcitrance and bioaccumulation, and potentially inducing toxicity, mutagenicity, and carcinogenicity . Even very small of such dyes is easily noticeable and are unacceptable. Therefore, treatment of these effluents prior to discharging into water becomes extremely crucial.

Figure 1 Untreated Discharge of Dye effluents into streams

1.1: - <u>TYPES OF DYES</u>

Dyes can be actually classified into various types depending on their chemical structure, application and method of application:

Acid Dyes: Acid dyes are water-soluble dyes that are applied to fibers containing protein, such as silk, wool, and nylon. They are used extensively in the textile industry for dyeing and printing of these fibers. Acid dyes are known for their vibrant colors and good lightfastness.

01: - Basic Dyes: - Basic dyes are water-soluble cationic dyes that are primarily used for dyeing acrylic fibers. They have a positive charge and are attracted to the negatively charged acrylic fibers. Basic dyes are also used for dyeing paper, leather, and some types of plastics.

02: - Direct Dyes: - Direct dyes are water-soluble dyes that can be directly applied to cellulosic fibers, such as cotton, rayon, and linen, without the need for a mordant. They are commonly used in the textile industry due to their ease of application and cost-effectiveness. Direct dyes are also used in the coloration of paper and leather.

03: - Disperse Dyes: - Disperse dyes are water-insoluble dyes that are primarily used for dyeing synthetic fibers, particularly polyester and acetate. They are finely dispersed in a liquid medium before application and require high temperatures to achieve dye fixation. Disperse dyes are commonly used in the textile industry for dyeing synthetic fabrics, including polyester clothing and polyester blends.

03: - Reactive Dyes: - Reactive dyes are a class of dyes that chemically react with the fiber molecules to form covalent bonds. They are primarily used for dyeing cellulosic fibers, such as cotton, rayon, and linen. Reactive dyes offer excellent colorfastness and are widely used in the textile industry.

04: - Vat Dyes: - Vat dyes are insoluble dyes that require a reducing agent to make them soluble and dye the fibers. They are used for dyeing cellulosic fibers, such as cotton, as well as silk and wool. Vat dyes are known for their excellent colorfastness and are commonly used in the production of denim fabrics.

05: - Solvent Dyes: Solvent dyes are dyes that are soluble in organic solvents but insoluble in water. They are primarily used for coloring plastics, hydrocarbon fuels, waxes, and various industrial products. Solvent dyes are also used in the production of printing inks and marking pens.

06: - Natural Dyes: - Natural dyes are dyes derived from natural sources, such as plants, insects, and minerals. They have been used for centuries for coloring textiles and other materials. Natural dyes offer a wide range of colors, but their application is often limited by factors such as colorfastness and availability.

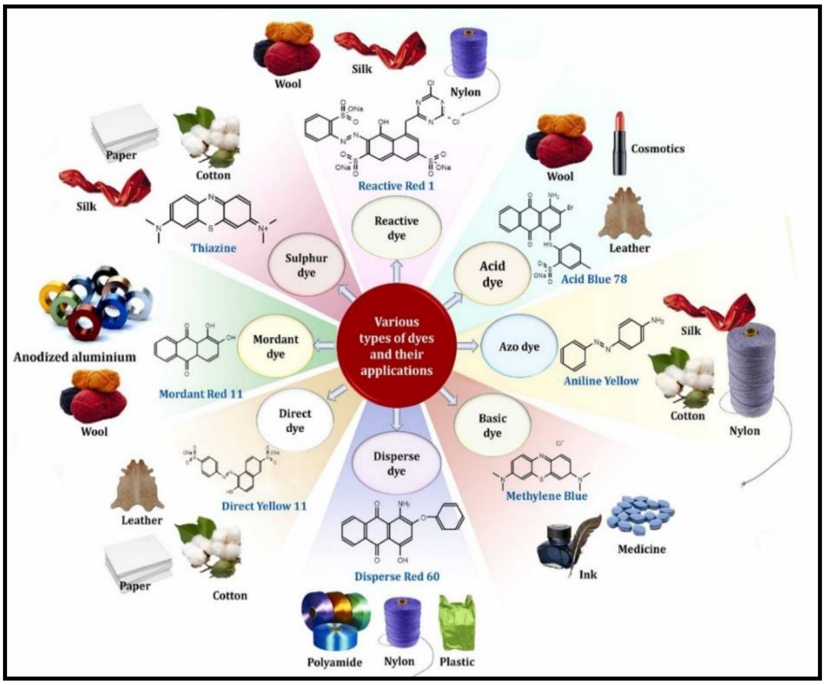

Figure 2 Various Types of Dyes

There are numerous other specialized dyes used in various industries, including optical brightening agents, fluorescent dyes, disperse fluorescent dyes, and more, each with their unique properties and applications.

1.2: - IMPACTS OF WATER CONTAMINATION FROM DYE EFFLUENTS

These dyes play a huge role as coloring agents but their production and usage in multiple industries leads to discharging of waste water into environment which has multiple implications. Water contamination due to dye can occur when dyes or pigments are released into water sources, leading to a change in water color and potentially causing harm to aquatic life and human health. Dye contamination can happen in various ways, such as industrial discharges, accidental spills, improper disposal, or even through the use of dyes in consumer products that end up in wastewater. Here are some key points about water contamination due to dye:

01: - Industrial discharges: Industries that use dyes, such as textile, paper, leather, or dye manufacturing plants, can release dye-laden wastewater into rivers, lakes, or other water bodies. If not properly treated, these discharges can cause significant water pollution.

02: - Accidental spills and improper disposal: Spills during transportation or mishandling of dyes can result in direct releases into water sources. Similarly, improper disposal practices like dumping unused dyes or dye-containing products down the drain can contribute to contamination.

03: - Wastewater from consumer products: Some consumer products, such as detergents, cosmetics, and hair dyes, may contain dyes. When these products are used and washed off, the dyes can enter the wastewater system and ultimately contaminate water bodies if not adequately treated.

04: - Environmental impact: Dyes in water bodies can have detrimental effects on aquatic ecosystems. They can disrupt the balance of underwater flora and fauna, reduce light penetration affecting photosynthesis, and cause oxygen depletion, leading to fish kills and other ecological imbalances.

05: - Health concerns: Certain dyes, especially synthetic ones, may contain harmful chemicals or heavy metals that can pose risks to human health. Ingesting or using contaminated water for drinking, cooking, or bathing can result in various health problems, including skin irritation, allergies, gastrointestinal issues, or even long-term chronic effects.

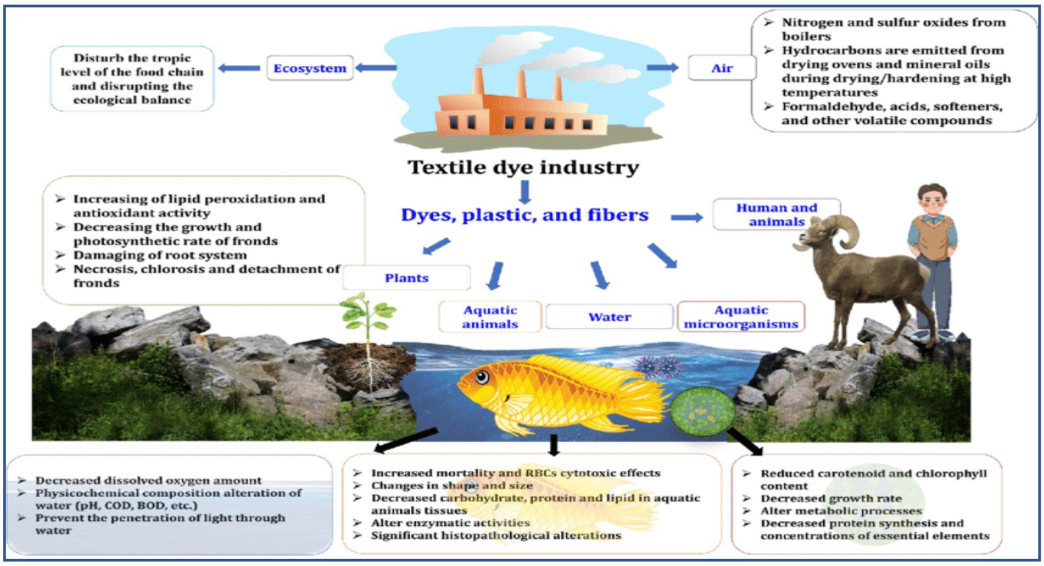

Figure 3 Ecological Impact of Textile Dyes

1.3: - IMPACT OF DYE EFFLUENTS ON ENVIRONMENT

One of the most essential resources on planet Earth for human development and life is water. The discharge of dye effluents into water bodies is one of the human activities which is responsible for water pollution and contamination. These dye effluents significantly adversely impact quality of water bodies, increase the biochemical as well as chemical oxygen demand, impacts photosynthesis in marine plants adversely, promotes toxicity in water and makes water carcinogenic. The color associated with effluent discharges may not impact the quality of water but it reduces the penetration of sunlight into water bodies which eventually reduces rate of photosynthesis in plants[4] . These effluents also act a toxic substance and may be carcinogenic as well. The contaminated water may also be used in agriculture which degrades the quality of soil even makes these toxic effluents enter into food chains impacting Human lives as well. The use of these azo compounds is very negative to soil microbial communities[4] and to the plant's germination.

1.3.1: - Water Pollution

Water is the basic need for the survival of living organisms. Humans in the name of cultivation and globalization have been exploiting water and multiple Earth's natural resources. This rapid pace of industrialization has led us to this severe stage of water contamination impacting both human as well as aquatic life. For instance, the dye effluents released into the streams of water bodies comprises of chemicals and heavy metals which directly or indirectly impacts our lives. These effluents alter the chemical and physical aspect of the water bodies making it unsuitable to use for human and aquatic life. The chemicals being discharged are so much in amount that it even becomes a tough task to quantify the exact quantity. It even impacts the aesthetic appearance of our water bodies.

1.3.2: - Soil Contamination

One of the primary reasons for the contamination of soil are azo dyes. These dyes enter into our land eco-system via irrigation and cultivation of soil using the contaminated water from the streams. This eventually degrades the quality and fertility of soil making it unsuitable for farming and even makes these dyes enter into human food-chain via Plants and food that are being consumed which eventually grow on this contaminated soil. The chemicals in the effluents can accumulate in the soil over time, affecting soil quality, nutrient balance, and plant growth. This contamination can also seep into groundwater, further polluting water resources.

1.3.3: - Disruption of Ecosystems

Even though water covers around 70% of our Earth's surface, one of the most important problems that living population is confronting today is inadequate availability of pure water. The need for proper drinkable water is rising at a rapid pace, with estimates indicating that agriculture, industry, and households use 70%, 19%, and 11% of available water, respectively[5]. Moreover, thanks to the contribution of these harmful azo dyes and chemical compounds being discharged into streams untreated are leading to further disrupting the ecological balance. These untreated effluent discharges are impacting both terrestrial habitats as well as aquatic ecosystem. The effluents inhibit the capability of aquatic flora to perform photosynthesis, thereby reducing the oxygen content for marine organisms which leads to the threat to aquatic ecosystem. Moreover, the marine organisms under the impact of these chemical dyes even impacts the gills of fishes and marine animals to uptake oxygen threating their existence. These chemicals even lead to causing genotoxic impacts on aquatic life which in turn effects them physiologically impacting negatively their cells and liver. These chemicals also disturb the presence of multiple micro-organisms present in aquatic ecosystem disturbing the chain of biodegradation events.

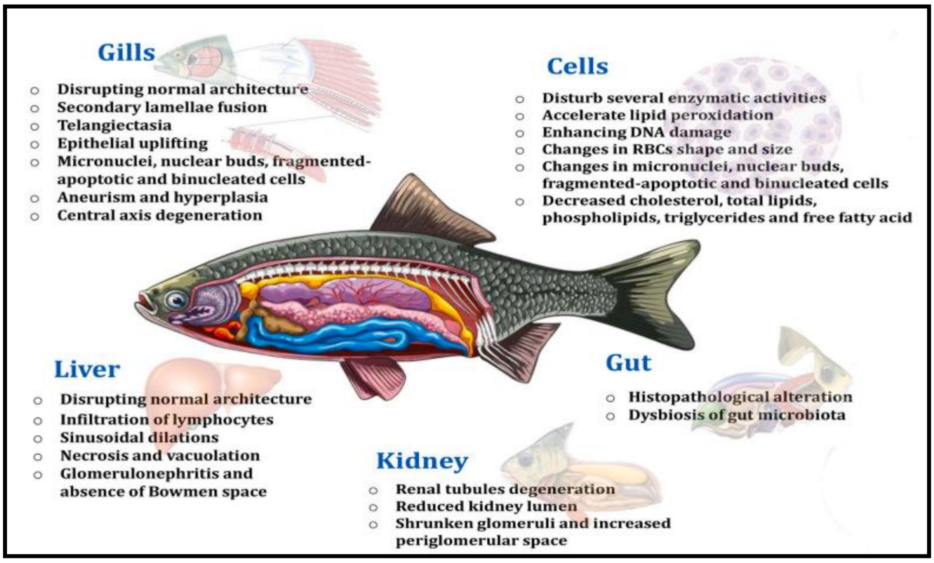

Figure 4 Harmful Impact of Dye Effluent on Marine Fishes

When such contaminated water is used on land then in turn it again leads to disturbing the balance of soil microbial flora. From the soil these toxic, mutagenic and carcinogenic compounds find its way to crops which indirectly enters into human as well as animal bodies impacting the terrestrial ecosystem. These chemicals can cause multiple biological and physiological changes at micro levels which in a long run can lead to development of multiple diseases[6].

1.3.4: - Human Health Risks

Exposure to the harmful dyes or consumption of contaminated water and food items can bring cause disruption in various bodily functions of humans and can even cause damages to human organs. These effluents can severely impact humans physiology and can even lead to development of cancer. These dye effluents can cause mild skin and hair related issues to severe damages to Central Nervous System (CNS). This dye effluent can disrupt functions of various enzymes in body which act as co-factor in multiple biological process[7]. Often people who work in industries producing or utilizing dyes are reported of suffering from multiple ailments like skin and eye irritation, rhinitis and acquired asthma from workplace[8]. Azo dyes derived from benzidine and its derivatives have been thoroughly investigated for their toxicity, which has been linked to human bladder cancer[9]. Textile dyes such as Reactive Green 19, Disperse Red 1, and Reactive Blue 2 all have a long-term genotoxic effect on human health. Reactive Green 19 was found to be genotoxic in a dose-dependent manner[9].

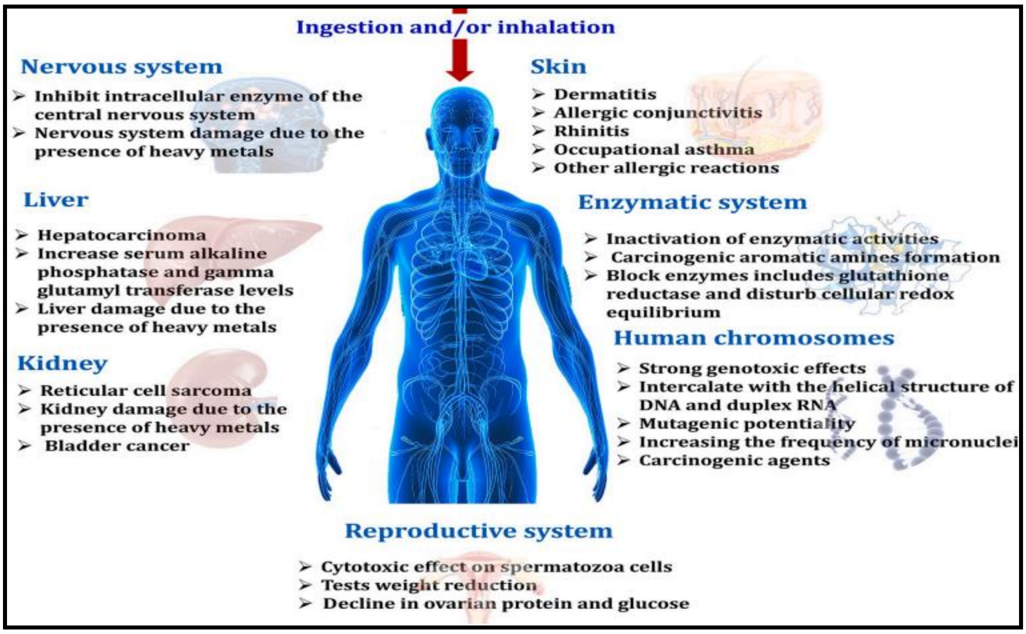

Figure 5 Harmful Impact of Dye Exposure on Humans

Due to the above-mentioned multiple reason, the separation of dyes and proper treatment of dye effluents before discharging into streams becomes a matter of serious concern. The proper treatment of effluents will help in bringing down multiple consequences faced from untreated discharges and will also help in bringing up the quality of water bodies thereby uplifting both aquatic and terrestrial ecosystems.

1.4: - SEPARATION OF DYE EFFLUENTS

The process of removing dyes from dye effluents or wastewater is known as separation of dye effluents. The separation of dye can help us in reducing the environmental impacts of dye effluent discharges. Basically, there are three methods of dye removal viz. Chemical, Biological and Physical[10].

1.4.1: - Chemical separation

Chemical Separation of dyes can be done using various methods to remove dyes from the wastewater. Here are some commonly used separations technique;

01: - Coagulation-flocculation: In this method coagulants are used for proper treatment of wastewater before disposal so that the water becomes toxin free and safer to be disposed. In the treatment technique, polymers and certain metal salts are used as coagulants, while polymers which increase the aggregation for easy separation of dyes are used as flocculant[11]. At first coagulants are added at the vigorous mixing stage and once it neutralizes or reduces the charge of finely dispersed particles, we add flocculant to the same. Flocculant with fine particles form large particles which can be separated easily by sedimentation[12].

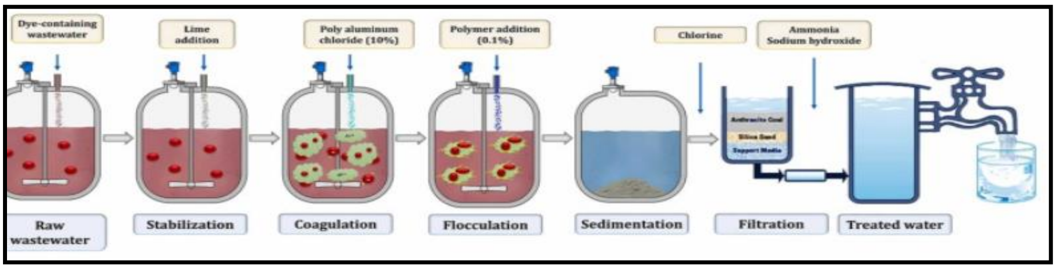

Figure 6 Mechanism of Coagulation-flocculation

02: - Electrochemical Methods: Electrochemical processes involves high cost of electricity and are considered less effective compared to other methods but the advantage that it offers is that it doesn't require us to add any additional chemicals to the wastewater. Electrocoagulation is one of the electrochemical approach which involves two electrodes made up of metal for direct power supply and also for the collection of coagulant mass which is later separated from wastewater[13]. One other time of electrochemical approach is Anodic oxidation in which organic compounds are absorbed at anode and then degraded by anode electrode transfer mechanism[14].

03: - Oxidation/Reduction Methods: Oxidation and Reduction reactions are used in order to break molecules of dye into simpler or less harmful compounds. Oxidizing agents like chlorine, hydrogen peroxide, ozone, or advanced oxidation processes (AOPs) generate reactive species that chemically degrade dyes. Reduction processes may involve the use of reducing agents like sodium bisulphite or sodium metabisulphite to convert dyes into colourless or less toxic forms.

1.4.2: - BIOLOGICAL SEPARATION

Biological techniques for separating dye effluents from wastewater are quite more promising than chemical techniques due to the fact that they require less chemicals, is less expensive, energy saving and easily performable. The biggest advantage that biological treatments offer is that they use microorganisms which have high biodegradable ability[15]. Here are several biological treatments used for separation of dye from wastewater;

01: - Enzyme Assisted Degradation: It is a slightly costlier method since it uses pure Enzymes and it is not usually the very first method for treatment of wastewater. Although some Industrial enzymes which are cost effective and have high efficiency are often used as part of Enzyme Assisted Degradation. Laccases and azo reductases, are one of the examples of effective enzymes that degrades azo dye-containing wastewater, converting complex organic pollutants into simple products, and removing them from wastewater *via* flocculation[16]. Specific enzymes produced by microorganisms can catalyze the degradation of dyes. Enzymatic treatments can be used alone or in combination with other biological methods to enhance dye removal efficiency.

02: - Bacteria Assisted Degradation: Certain Bacterial Species have proven to be more efficient when it's about degradation of complex compounds of dyes than microorganisms[17]. Bacterial Species like *Bacillus, Rhodococcus, Shigella and Pseudomonas* have shown promising results towards degradation of azo dye[18][19]. Using Bacterias offer an advantage that they are easier to cultivate and have higher specific growth rate in comparison to microorganisms[20].

03: - Fungal Assisted Degradation: Fungal species play an important role in the degradation of a wide range of dyes and fungi have the ability to produce various enzymes that can effectively degrade a wide range of dyes. Fungal strains are capable of degrading certain dyes via producing enzymes having dye degrading capabilities. Commonly used fungi include *white-rot fungi* (such *as Phanerochaete chrysosporium*) and *Aspergillus species*[21]. Fungi secrete extracellular enzymes, including ligninolytic enzymes (such as *laccases, lignin peroxidases, and manganese peroxidases*), cellulases, and proteases. These enzymes play a crucial role in the degradation of dye molecules.

04: - Yeast-Assisted Degradation : Yeast-Assisted degradation offers advantages over both fungi and bacterial degradation since they Yeast carry an ability of rapid growth and are quite resistant to environmental and pH factors[22]. Certain yeasts, like *Scheffersomyces spartinae*, *Pichia occidentalis*, and *Sterigmatomyces halophilus*, have the ability to degrade multiple dyes, including azo dyes[23].

1.4.3: - PHYSICAL SEPARATION

Physical separation of dye effluents refers to the process of isolating and removing dye-containing wastewater from industrial or domestic sources. The process has an efficiency of around 85-99% concerning the dye removal. The advantages that it offers over chemical and biological approaches if that it is cost-effective, have easy setup and uses minimal chemical compounds. Various Physical approaches can be used in order to separate dye from wastewater.

01: - Adsorption: In this method absorbent materials like Zeolites, clay, activated carbon etc. are used in order to attach and bind the dye particles which are then removed from the wastewater. This approach offers certain advantages like reusability of absorbent, high efficiency and short time duration for the removal of dye[24].

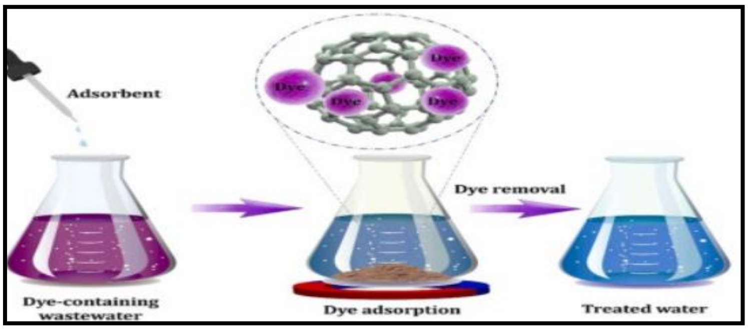

Figure 7 Mechanism of Adsorption Process of Dye removal

02: - Ion Exchange Method: This method has gained popularity recently since it offers advantages like cost-effectiveness, regeneration, simplicity, flexibility, and high efficiency. This method is based on the strong interactions between functional group of the resin used in the packed bed and the ions of the dye molecule. The mechanism for same in demonstrated in figure below.

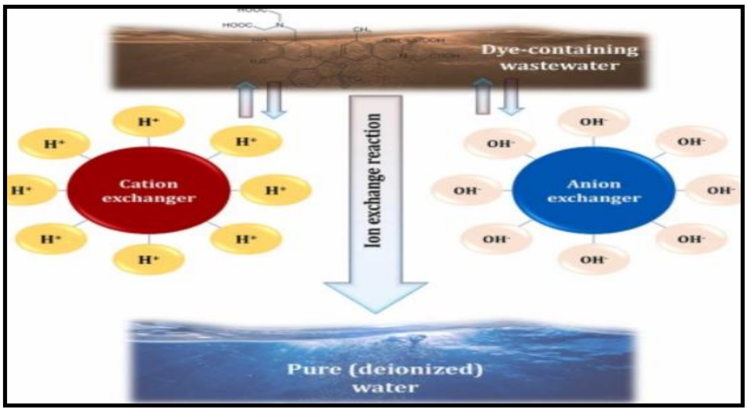

Figure 8 Mechanism of Ion-exchange method of Dye removal

03: - Membrane-Separation: In this method we employ techniques like nano-filtration, reverse osmosis and ultrafiltration in which we use suitable semi-permeable membrane to separate out the dye particles from the wastewater. It is one of the best approaches that are used for dye separation and works on the principle that it allows particles having small size to pass through and inhibits or stops dye particles having large size.

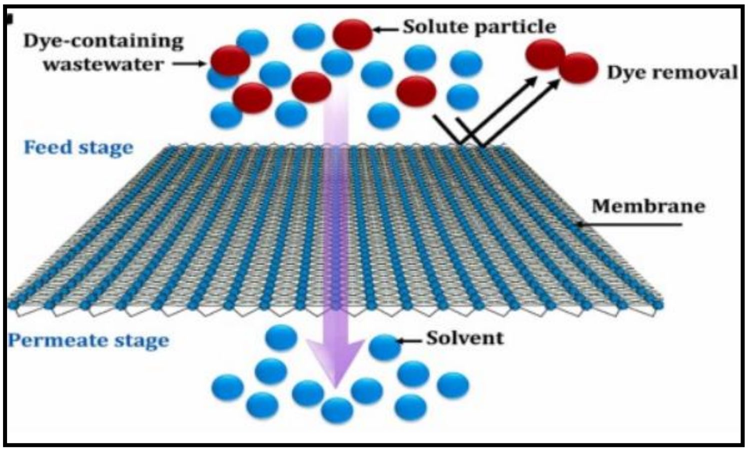

Figure 9 Dye Separation via Membrane separation

1.5: - NANOFILTRATION

Water pollution resulting from heavy metals is widely recognized for its toxicity and adverse effects on human health and the environment. With the expanding human population, water pollution becomes inevitable due to activities in sectors such as industry, agriculture, and services. To address this issue, have investigated various treatment technologies, including reverse osmosis, disinfection, granular filtration, gravity separation, coagulation-flocculation, air stripping and aeration, ion exchange, adsorption, and membrane filtration. Nano-filtration is one of the membrane filtration techniques which is used for treatment of dye effluents. This technique utilizes semi-permeable membrane with pore size 0.2-2 nm with molecular weight cut-off (MWCO) from 200 to 1000 Da which is somewhere in between that of reverse osmosis and ultrafiltration. The Mechanism of membrane separation is represented down below in the figure.

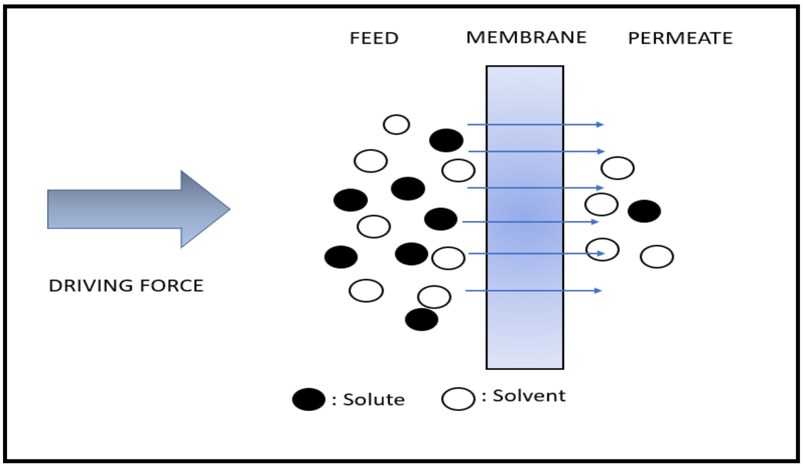

Figure 10 Mechanism of Membrane separation method of Dye removal

When we talk about membrane separation then it can actually broadly be classified into three groups namely Electrically-driven Membrane process, Concentration-driven Membrane process and Pressure-driven Membrane process. Microfiltration, ultrafiltration, nanofiltration, and reverse osmosis comes under Pressure-driven membrane process where varying pressure is the driving force of the process as depicted in figure below.

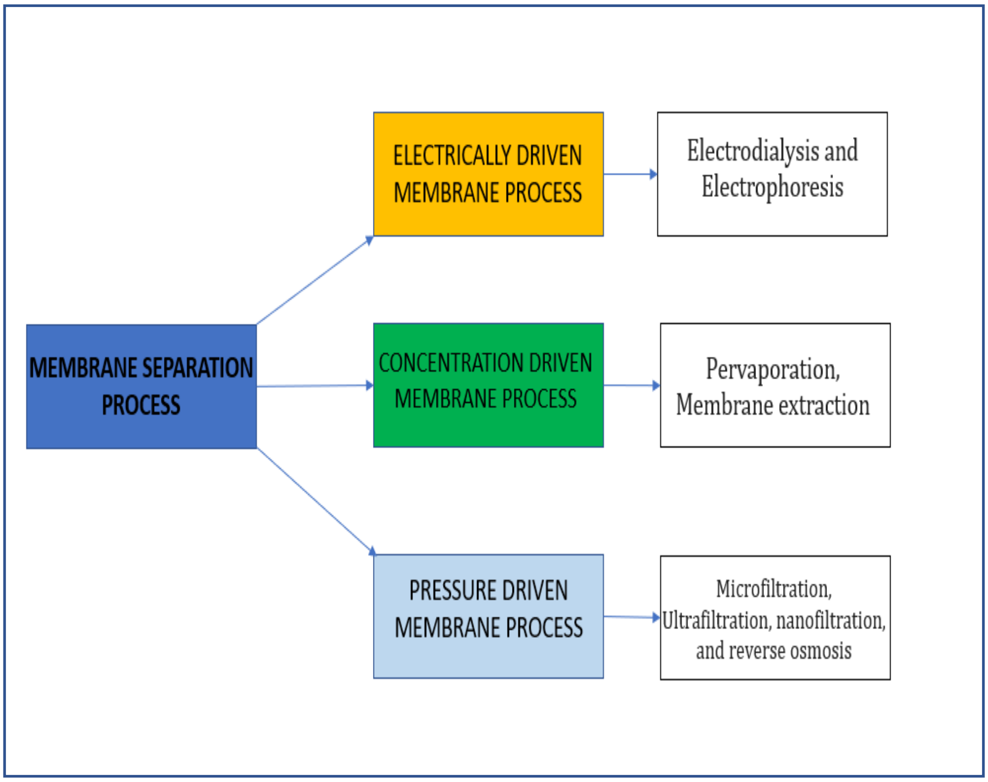

Figure 11 Classification of Membrane Separation methods

1.6: - DIFFERENCE BETWEEN NANO-FILTRATION, REVERSE OSMOSIS AND ULTRAFILTRATION

Nanofiltration (NF) is a membrane filtration process that falls between reverse osmosis and ultrafiltration. It has a pore size larger than reverse osmosis but smaller than ultrafiltration. NF membranes are capable of removing divalent ions, larger organic molecules, and certain multivalent salts while permitting smaller ions and molecules to pass through. NF is commonly employed for water softening, color removal, reduction of specific contaminants, and selective separation of components.

Reverse osmosis (RO) is a filtration process that employs a semi-permeable membrane with small pores. The membrane selectively allows solvent molecules, usually water, to pass through while rejecting dissolved solutes such as salts, minerals, organic compounds, and microorganisms. RO is widely used for desalinating seawater or brackish water, purifying drinking water, and treating industrial process water. It produces highly purified water, but it necessitates higher pressure and energy consumption compared to other membrane processes.

Ultrafiltration involves the use of membranes with larger pore sizes compared to nanofiltration and reverse osmosis. UF membranes effectively remove suspended solids, colloids, macromolecules, bacteria, and some viruses while allowing smaller molecules and ions to pass through. UF is often used for pretreatment in water and wastewater treatment systems, as well as in the food and beverage industry for clarification and concentration processes.

METHODS	PORE SIZE	COMPONENTS REMOVED
Ultrafiltration	0.02-0.2µm	High molecular weight compounds and suspended particles
Nanofiltration	0.002-0.2µm	Polyvalent anions, cations, uncharged compounds, suspended compounds
Reverse Osmosis	0.0002-0.2µm	Essentially all dissolved compounds and suspended particles

1.7: - ADVANTAGES OF NANOFILTRATION

Nanofiltration offers certain advantages for the treatment of dye effluents:

Effective dye removal: Nanofiltration membranes with small pore sizes can selectively remove dyes from the effluent, resulting in efficient reduction of color and elimination of various types of dyes, both organic and inorganic.

Versatility: Nanofiltration is capable of handling a wide range of dye concentrations and types, making it suitable for different industrial applications. It is effective in treating both high and low concentration dye effluents.

Reduced energy consumption: Nanofiltration operates at lower pressures compared to other membrane processes like reverse osmosis. This leads to lower energy requirements and potentially reduced operational costs.

Retention of essential components: Nanofiltration selectively removes dyes while allowing smaller ions and water molecules to pass through. This means that essential components in the effluent, such as beneficial salts or dissolved minerals, can be retained in the treated water.

Environmental benefits: Nanofiltration effectively removes dyes and reduces the color of the effluent, helping meet regulatory requirements for wastewater discharge. This contributes to the protection of water bodies and reduces the environmental impact of dye effluents.

Compact system design: Nanofiltration systems can be designed in a compact manner, requiring less space compared to conventional treatment methods. This makes them suitable for installations in areas with limited space availability.

Flexibility in process integration: Nanofiltration can be integrated with other treatment processes, such as coagulation or activated carbon adsorption, to enhance the overall efficiency of dye effluent treatment. This flexibility allows for customized treatment solutions based on specific requirements.

However, Nanofiltration membranes are susceptible to a common issue known as membrane fouling. Membrane fouling refers to the build-up and deposition of undesired substances on both the surface and within the pores of the membrane.

1.8: - RHODAMINE B

Rhodamine B is a synthetic organic compound classified as a cationic dye. It belongs to the family of xanthene dyes and has a bright pink to reddish color. The dye is soluble in water and other polar solvents, and its chemical formula is C28H31ClN2O3.

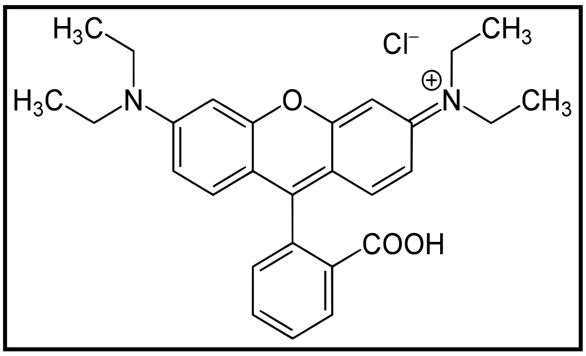

Figure 12 Structure of Rhodamine B

Rhodamine B is dye which finds its application in multiple industries like in textile industry for dyeing of clothes, as a fluorescent dye in various applications, including fluorescence microscopy, flow cytometry, and fluorescence-based assays, as a sensitizing dye in the production of optical brighteners, which are additives that enhance the appearance of materials by absorbing ultraviolet light and re-emitting it as visible light etc.

Rhodamine B also has multiple harmful impacts on human as well as environment. Rhodamine B, can potentially enter the body and cause oxidative stress on cells and tissues. Prolonged consumption of food containing Rhodamine B can lead to liver dysfunction or even cancer. Additionally, exposure to high amounts of Rhodamine B over a short period can result in acute poisoning[25]. Rhodamine B has high affinity for Chlorine and when it binds with it forming halogen compounds which interacts with various proteins in body and acts as carcinogenic toxin[26]. Contact with rhodamine B can cause irritation and allergic reactions on the skin and eyes. It may lead to redness, itching, burning sensations, and swelling. Rhodamine B is not readily biodegradable and can persist in the environment for a long time. Disposal of products containing rhodamine B or its release into water bodies can have detrimental effects on aquatic ecosystems. Hence it is generally discouraged or strictly regulated to protect human health and the environment and it becomes really crucial to separate this dye from wastewater prior to releasing into streams.

CHAPTER 2: EXPERIMENTAL

2.1: - MATERIALS USED

The various materials used throughout experimentation are as follows: -

01: - Dye Rhodamine-B. Purchased from Sisco Research Laboratories Pvt. Ltd. Maharashtra, India.

02: - Membrane HF-150. A polyamide membrane. Purchased from Permionics Membranes Pvt. Ltd. Gujrat, India.

03: - Photo-spectrometer with 10 mm quartz crystal was used for the determination of the absorbance values.

04: - Distilled water was used for preparation of Dye Solution.

05: - All the other chemicals used were of analytical grade and were used in the original form as received from the supplier.

2.2: - <u>EXPERIMENTAL SETUP</u>

The typical system used for the nano-filtration of Rhodamine-B dye is shown below in figure 2 below. The figure shown is the schematic of the lab scale apparatus that was build and used for the experiment. The lab scale apparatus is shown in Figure 3

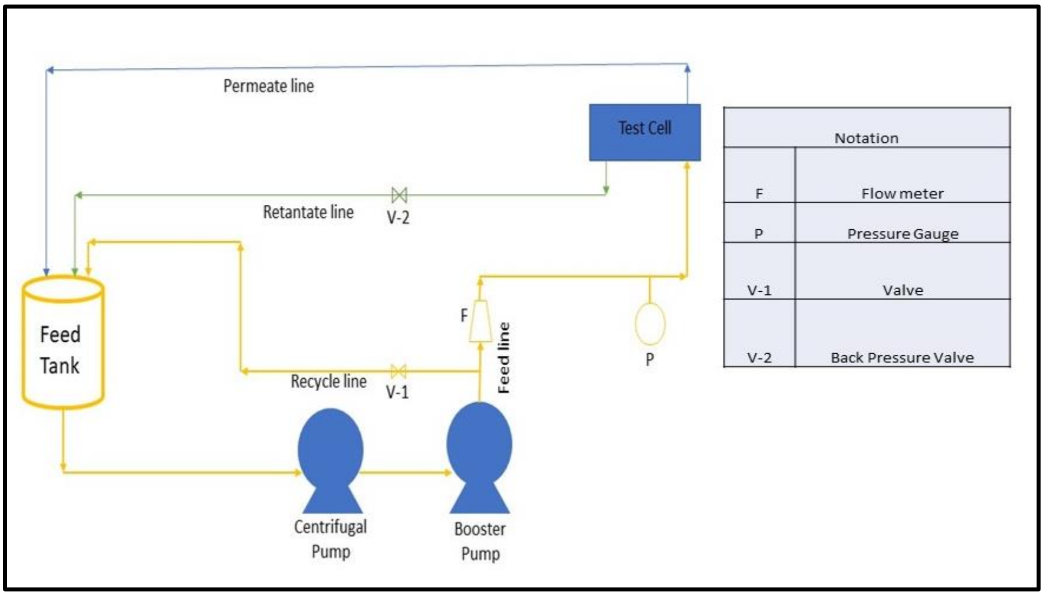

Figure 13 Block diagram of Filtration apparatus

As shown in the figure, the setup consists of a 5 L feed tank in which the feed solution is kept. The feed solution was prepared by dissolving the dye of different concentrations in distilled water. The 2 pumps i.e., centrifugal and booster pumps are kept in the feed tank which pumps the feed solution into the filtration half of the apparatus. The details of the membrane and the channel is listed below in table.

The total area of the membrane	0.00785 m^2
Diameter of the membrane	0.1 m
Thickness of the channel	10 mm

Across the channel, a flow meter is connected which regulates the flow of the solution. The flow meter measures the flow in Litres per min (Lpm). The range of measurement of flow meter was 0 to 1000 lpm with the least count being 50 lpm. For our study it was varied from 200 to 600 lpm with an interval of 100 lpm. Moving ahead across the channel a pressure gauge P is connected. Its job is to regulate the pressure of the system. Maximum attainable pressure of the system was 12 bars. For this study the pressure was varied between 2 to 6 bar. It should be noted that the reason behind taking a moderate pressure was, since the filtration apparatus was a lab-scale apparatus therefore it could hardly give good results at high pressure. At very higher pressures either the system started leaking or in worst case the membrane itself was getting torn. Therefore, a moderate pressure of 2 to 6 bar was taken for the experiment. Through the channel comprising flow meter and pressure gauge the feed solution reaches the test cell i.e., the region where the membrane is fitted. The solution gets filtered here. The filtered solution is divided into two parts i.e., the permeate is taken through one channel and collected and the retentate through another channel is again discharged into the feed solution.

The lab scale setup of the nanofiltration apparatus used for the experiment is shown below: -

Figure 14 Lab scale Nano filtration apparatus

2.3: - PREPARATION OF DYE SOLUTION

Dye solutions was prepared for the nanofiltration. The feed solution was prepared by dissolving the dye Rhodamine B in distilled water. Physical parameters like temperature and pressure were kept constant while the dissolution was taking place. Five different concentrations of solution were prepared namely 50,100,150,200,250 ppm. The dye was dissolved properly into 5 L of distilled water each time. Since the interdependence of various parameters on filtration of dye solution were to be studied therefore concentrations ranging from 50 to 250 ppm was selected.

2.4: - MEMBRANE PERMEABILITY

Before beginning the nanofiltration the membrane was soaked into water for some time and then the readings were taken at high pressure until a constant flux was obtained. This was done so as to avoid any ambiguity in the readings during the real run of the apparatus. After soaking the membrane for some time, the membrane was fitted into the membrane case of the apparatus as shown in figure __. Raw run of the apparatus was done using water so as to ensure no leakage in the apparatus.

During dry run of the apparatus, water was allowed to pass through the membrane for a time interval of 3 minutes (an arbitrary no.) and the amount of water filtered also known as permeate of filtration was noted. This value was used to calculate the flux of the filtration. Flux of filtration is defined as the volume of solution that passes through per unit area of the membrane. It is basically the rate at which permeate is obtained; or in general terms it is the rate of filtration. It was calculated using the following formula:

$$\boldsymbol{Flux} = \frac{\boldsymbol{Volume}}{\boldsymbol{Area} \times \boldsymbol{Time}} \quad (\times \mathbf{10^{-6}}\, \boldsymbol{m/s}) \qquad [\mathbf{1}]$$

Where Volume: Vol of permeate obtained in (m^3).

Area: Effective area of the membrane in (m^2).

Time: Time across which the volume of permeate was recorded in (s).

It should be noted that the effective area of the membrane, as specified previously was 0.007856 m^2 and the time interval of permeate collection was 3 min.

2.5: - NANOFILTRATION OF THE DYE SOLUTION

Nanofiltration of the dye solution prepared was done using the lab scale filtration apparatus as shown in figure __ and the permeate solution were collected. The apparatus was run on different set of conditions. During the filtration three sets of parameters were changed viz., feed concentration, pressure of the system, and flow rate of the feed solution into the test cell. For e.g., for a particular concentration, keeping the pressure constant, permeates were obtained for 5 different flow rates. Similarly, this was repeated for five different pressure values. For each set of reading the permeate was collected after running the apparatus for 3 min. Hence, a set of 25 values were obtained each having a unique set of concentration, pressure, and flow rate i.e., the concentration was varied from 50 to 200 ppm at an interval of 50 ppm; pressure was varied from 2 to 6 bar at an interval of 1 bar; flow rate was varied from 200 to 600 lpm at an interval of 100 lpm. After each filtration cycle the membrane was cleaned with distilled water at a high pressure. To ensure that there was no fouling/plugging of the membrane, after each cycle of separation water flux was noted and checked.

Permeates of all these 25 sets of readings were obtained and their absorbance values were checked using UV Spectrophotometer. For most of the readings it was obtained around 547 nm wavelength of light. The absorbance values across the peaks for each set of reading was noted and with the help of these absorbance values the concentration of the permeate obtained was calculated. The concentrations of the permeate and retentate is used to find the rejection factor using the formula mentioned below:

$$\boldsymbol{Rejection}\ (\%) = \mathbf{1} - \left(\frac{\boldsymbol{C_P}}{\boldsymbol{C_F}}\right) \times \mathbf{100} \qquad [2]$$

Where, C_P stands for concentration of the permeate obtained.

C_F stands for concentration of the feed solution or bulk solution.

The rejection % thus obtained was plotted against the three constraints viz., feed concentration, pressure, flow rate and their inter-dependence was studied. The same was done with the permeate flux calculated previously.

CHAPTER 3: RESULT & DISCUSSION

3.1: - MEMBRANE PERMEABILITY

During the dry run of the apparatus water was made to run through the apparatus. The permeate was collected and its volume was measured. Using the calculation mentioned in section 2.4 flux of the water permeate was calculate hence experimental data for permeability of water using the membrane was obtained. The plot for flux calculated vs the pressure applied is shown below in graph 01. It can be seen that the flux varies linearly with the pressure in accordance with the Darcy's law. i.e., as we increase the amount of pressure on the system the filtration was also found to increase. From the slope of the plot, the pure water permeability was found to be 3.26 x 10^{-6} $ms^{-1}bar^{-1}$

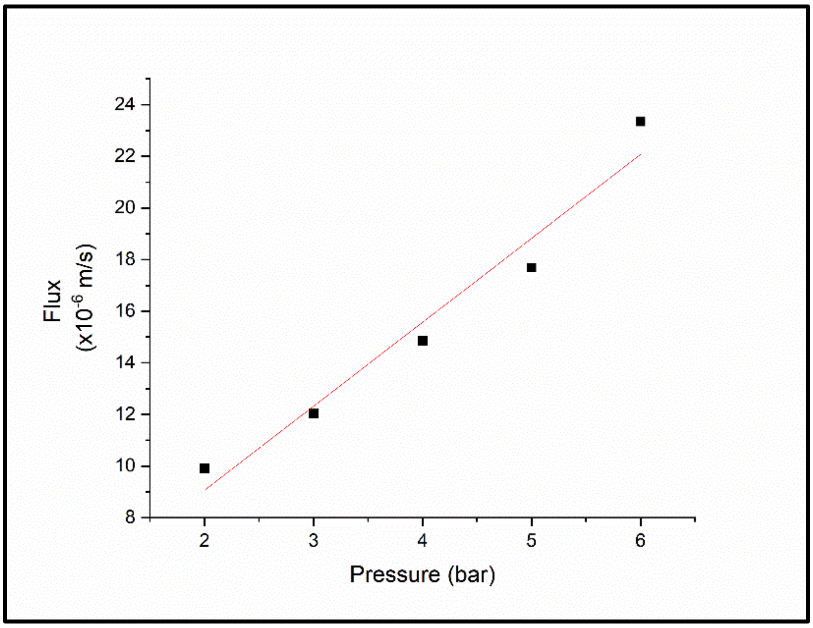

Figure 15 Graph for membrane permeability calculation

3.2: - EFFECT OF PRESSURE ON DYE REMOVAL

EFFECT OF PRESSURE ON FLUX OF FILTRATION

The flux performance of filtration on increasing pressure is shown below in graph 2. For the current study the pressure of the system was varied with respect to the feed concentration and the flow rate. The pressure was varied between 2 to 6 bars at an interval of 1 bar. The different coloured shapes in the graph represents different concentrations.

As it can be seen from the graph that as we increase the pressure applied the flux of the permeate solution is also increasing consequently. This may be attributed to the fact that the increase in pressure induced may also increase the thrust with which the dye solution is being acted upon the membrane. Therefore, the solution passes rapidly through the pores of the membrane hence filtration pace increases therefore the permeate volume and flux. However, some deviations from the expected values can be seen in the plot. This may happen due to fouling of the membrane. Fouling is the process by which the solute/dye particles get deposited on the pores of the membrane thus decreasing the filtration rate. Hence fouling decreases the porosity. To avoid fouling, the membrane, after each cycle of filtration was washed with water under high pressure.

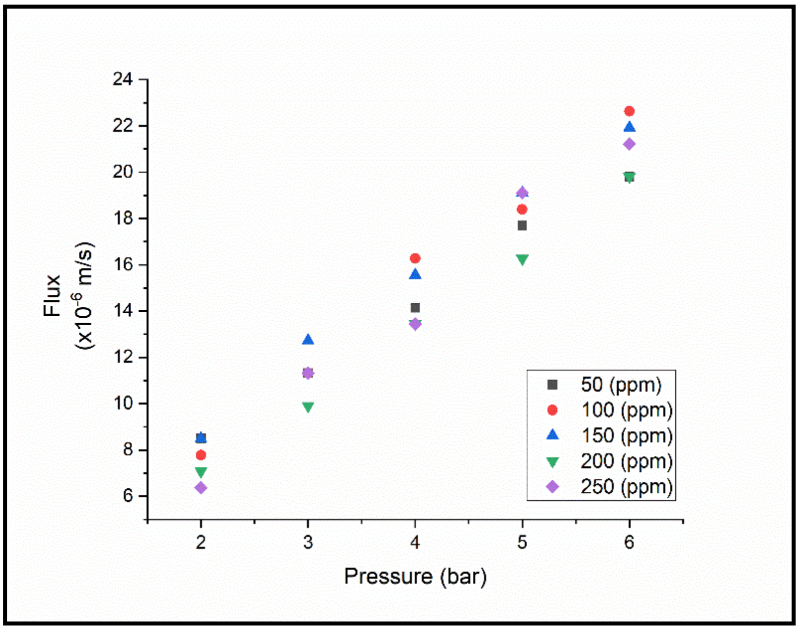

Figure 16 Graph for Flux vs Pressure

EFFECT OF PRESSURE ON REJECTION % OF THE DYE

The change in rejection of dye (in percentage) on changing the pressure of the system is plotted below in graph 3. For the present study, the pressure of the system was changed with respect to the bulk concentration as well as the flow rate. The graph plotted below comprises of the readings recorded at 300 lpm flow rate. The pressure was changed from 2 bar to 6 bar at interval of 1 bar.

The rejection percentage varied was averagely found above 90%. As visible from the graph, in totality, the rejection percentage is increasing with the increase in the applied pressure. For e.g., the 100-ppm data (shown in red circle) is found to increase from around 97% to 98.5% on increasing the pressure from 2 bar to 6 bar. This may be explained on the basis of the fact mentioned previously i.e., the increasing thrust on the surface of the membrane, hence faster rejection. However, here there are major deviations from the expected behaviour because fouling plays a major role here. Due to fouling, decrease in porosity happens hence very less amount of dye particles could cross the pores therefore major deviations.

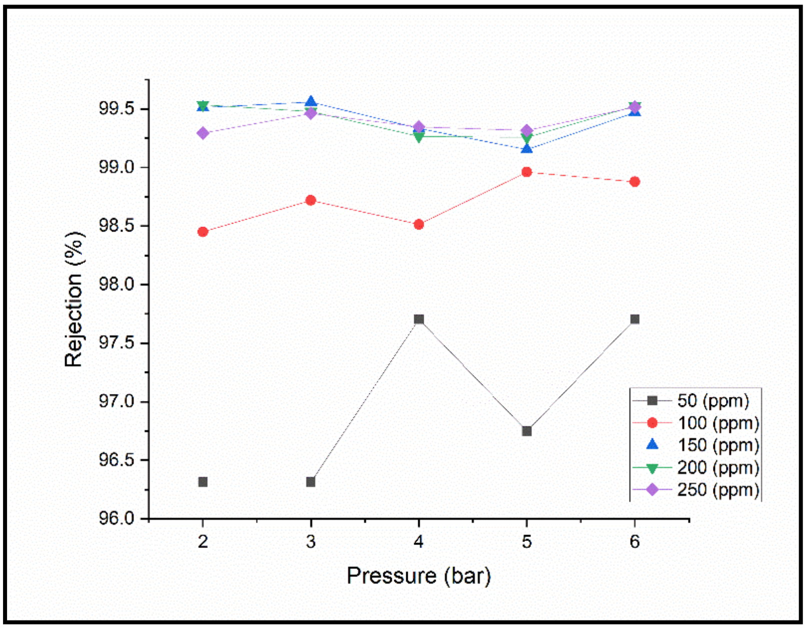

Figure 17 Graph for Rejection % vs Pressure

3.3: - EFFECT OF FEED CONCENTRATION ON DYE REMOVAL

EFFECT OF FEED CONCENTRATION ON FLUX OF FILTRATION

The relation between the feed/bulk concentration with respect to flux of filtration was studied. The findings of the same are presented below in graph 4. Five different concentrations of the dye solution were used viz., 50-250 ppm at an interval of 50 ppm each. Furthermore, the pressure and the flow rate were also changed. For the graph, plotted values corresponding to 400 lpm was kept constant.

The result of the findings is visible from the graph below. As the concentration of the dye solution was increased, the flux of the permeate filtered was found to decrease. (Say) The readings taken at 4 bar pressure (shown in blue rectangle) was found to decrease from around 17 m/s to 14 m/s on increasing the concentration from 50 ppm to 250 ppm. This may be explained on the basis of the viscosity. As the concentration increased the solution became thicker i.e., no of dye particles present in a given amount of dye solution was increased, hence, the fouling of the membrane happened faster causing decrease in the porosity of the membrane. Also, it becomes tough for the solution with higher viscosity to pass from the pores of the membrane hence the result.

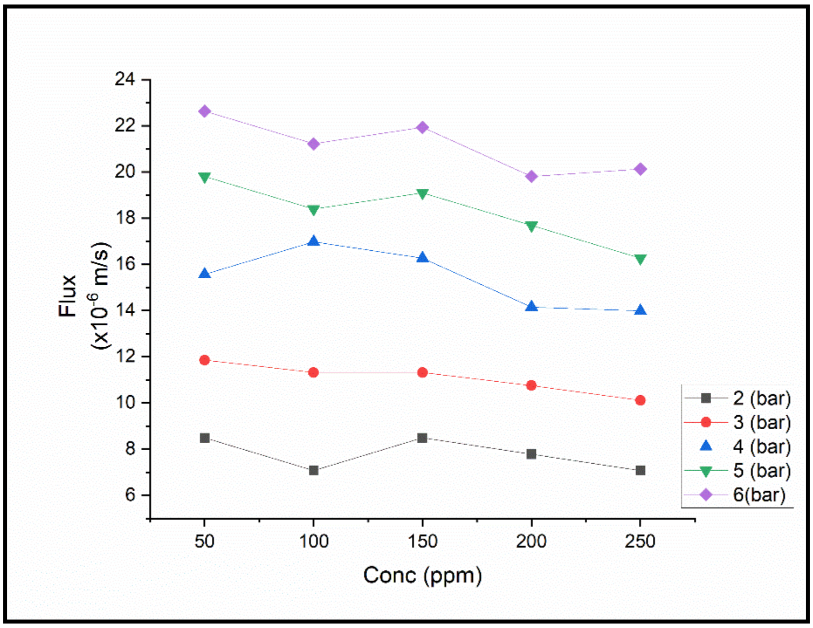

Figure 18 Graph for Flux vs Conc.

EFFECT OF FEED CONCENTRATION ON REJECTION % OF FILTRATION

The relation between feed concentration and rejection % was observed by changing the concentration of the bulk solution with respect to other different parameters like pressure and feed solution. In the present case the concentration was varied between 50-250 ppm at an interval of 50 ppm each. For the current plot of the findings flow rate was taken constant at 300 lpm.

As visible from the graph the relation between the two i.e., the feed concentration and rejection % is found to be of inverse nature. That is, with increase in the concentration of the dye solution the rejection % was found to decrease. For e.g., (Say) the readings taken at 6 bar (shown in violet rhombus) at 50 ppm gave around 94% rejection which on increasing the concentration to 250 ppm gave around 89% rejection of the dye. This again, is due to the fact that with increased conc, the no of dissolved particles are increasing causing fouling of the membrane thus dcreased rejection.

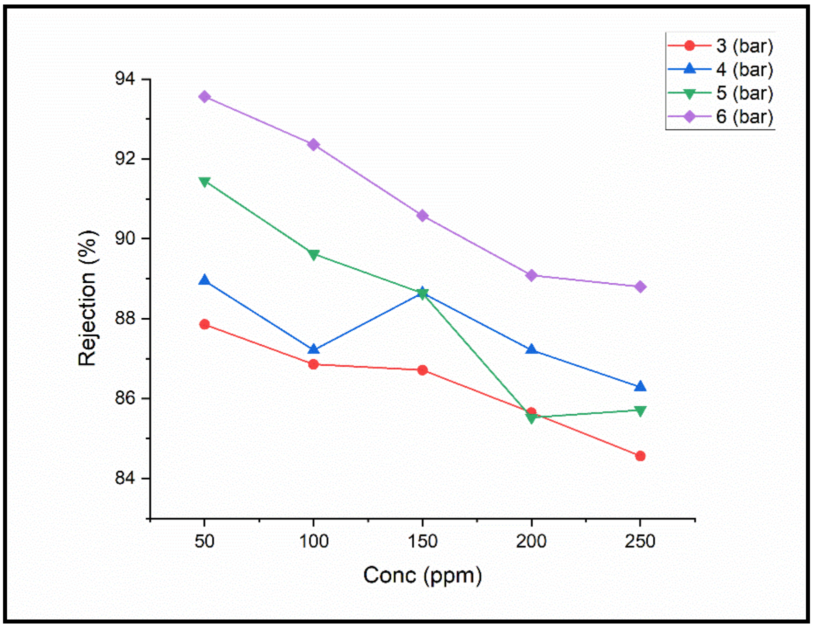

Figure 19 Graph for Rejection % vs Conc

3.4: - EFFECT OF FLOW RATE ON DYE FILTARTION

EFFECT OF FLOW RATE ON FLUX OF FILTRATION

The relation between the rate of filtration or flux and the flow of the solution was studied. For the present study the flow of the solution was changed with respect to concentration of the solution and the pressure. The flow of the solution was varied between 200 lpm to 600 lpm at an interval of 100 lpm., The readings at 3 bar pressure was taken for the plot shown below.

The result can be seen from the graph itself. The nature of the graph is increasing. i.e., on increasing the flow of the solution, the rate of filtration also increases. For e.g., the readings taken at 100 ppm (shown in red circle) the flux of filtration at 200 lpm is around 14.5 m/s which on increasing the flow too 600 becomes around 17.5 m/s. This may be attributed to the fact that due to increase in the flow rate of the solution the volume of feed solution reaching the membrane in a given amount of time increased i.e., the dispersal of bulk solution on the membrane surface increased which pushed the filtration thrust therefore the rate of filtration may also have increased.

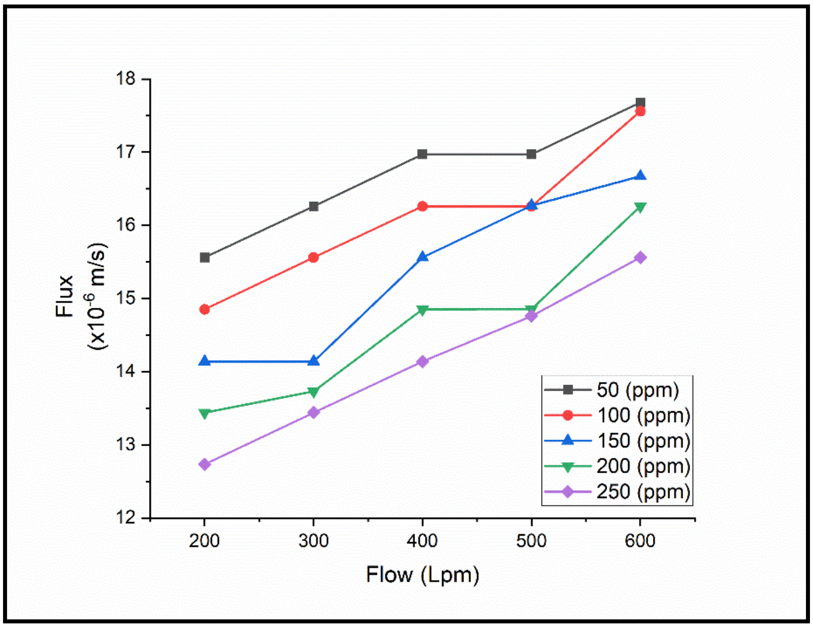

Figure 20 Graph for Flux vs Flow rate

EFFECT OF FLOW RATE ON REJECTION % OF FILTRATION

The relation between the rejection % of the dye and the flow of the solution is presented below. For this study the rejection of the dye solution at different flows were recorded. Other factors like concentration and pressure were also changed during the process. The readings taken at 200 lpm are used for the graph presented below. The findings are as follows:

It can be predicted from the increasing slope of the graph that the relation between the two was found to be directly related. i.e., on increasing the flow rate of the solution, the rejection % was found to increase. For e.g., the reading taken at 200 ppm (shown in inverted green triangle), the rejection obtained at 200 lpm was around 83%, which on increasing to 600 lpm was 93%. This maybe due to the fact that greater flow rate increased the dispersal of the dye particles on the membrane surface therefore higher rejection happened.

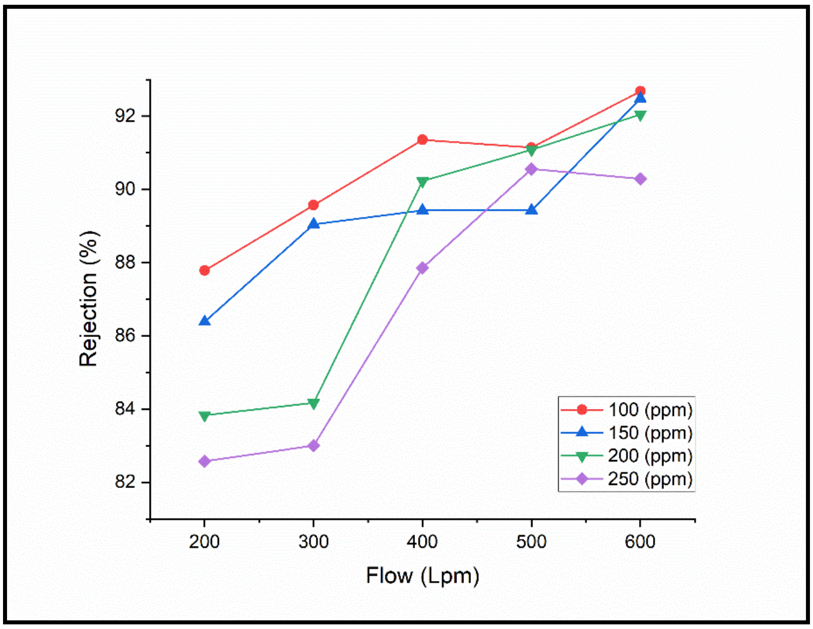

Figure 21 Graph for Rejection % vs Flow rate

CHAPTER 4: CONCLUSION

To summarize, Nano-filtration which employs a nano-filter membrane is a simple and economic yet efficient setup to remove dye effluents from the wastewater. Nanofiltration has smaller pore sizes compared to microfiltration and ultrafiltration. This characteristic enables Nanofiltration to achieve small molecular sieving, organic solvent recovery, and drug purification. Additionally, Nanofiltration has larger pore sizes and higher permeability compared to reverse osmosis, resulting in lower transmembrane pressure and reduced energy consumption during the Nanofiltration process. Therefore, in recent years Nanofiltration has emerged as one of the competitive techniques for the purpose of dye removal. It even finds its application in multiple industries like Food processing, textile industry, ink processing etc. It even has shown high efficacy of about 85-99% in terms of dye removal.

In the study presented above Physical separation of Rhodamine B from its dye solution was studied using the HF-150 membrane (polyamide chemistry based nanofiltration membrane). During the study, the variation of permeate Flux and Rejection with Pressure, Flow Rate and Feed concentration was observed. It was found that with increasing pressure and flow, the permeate flux as well as the Rejection was found to increase whereas, with increase in the feed concentration both these parameters were found to decrease. In the separation process, Rejection was found to be in the range 90%. Hence, employing HF-150 membrane for the purpose of removal of dye Rhodamine B which on exposure causes multiple health hazards and impacts our environment can be one of the solutions for wastewater treatment. One drawback which the proposed mechanism poses is fouling of membrane overtime but more investigation is needed to conclude the extent of fouling of the membrane. The proposed method of nano-filtration is easy, safe as well an economical process for separation of Rhodamine B solution.

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ATUL PATEL

Department of Applied Chemistry Delhi Technological University, New Delhi

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