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## THE REMOVAL OF COLOUR FROM SYNTHETIC TEXTILE DYE EFFLUENT USING CELLULOSE ACETATE REVERSE OSMOSIS MEMBRANE

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**Abstract:** *The main objective of this work is to remove the colour from synthetic textile dye effluent by membrane technology. The delivery of colour in the form of dyes onto textile fibres is not an efficient process. The degree of efficiency varies, depending on the method of delivery. As a result, most of the wastewater produced by the textile industry is coloured. It is likely that coloured wastewater was a feature of the first practices of textile dyeing. To make this research effective we suggest reverse osmosis process. The membrane used for reverse osmosis is cellulose acetate membrane and gravitational force is used as a major driving force. Many of the systems of separation use pressure gradient as the main driving force, but here we are keeping gravitational force as the main driving force with less amount of inlet pressure. The two synthetic dyes which are commonly present in the effluent of textile waste water were considered, namely Torquise blue dye and Remozol yellow dye. Then these two different types of dyes solution are treated in the reverse osmosis column which contains cellulose acetate membrane to check the percentage of color removal. This four stage setup is very much effective for effluent treatment also. This research work focused on the reverse osmosis part only for the colour removal of the synthetic effluent. The result shows that it is very much effective for the colour removal.*

**Keywords:** *Colour removal, Textile dye effluent, Reverse osmosis, Membrane, Cellulose acetate, Dyes, Effluent treatment.*

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## 1. INTRODUCTION

### 1.1. Reverse Osmosis (RO) process

Reverse Osmosis, initially termed as hyper filtration, is another pressure driven process in which a solvent or a predominating component of the solution is transferred through a membrane in such a way that the concentration of the solution is transferred through a membrane in such a way that the concentration of some solute with low molecular weights (up to 500 Daltons) or other solvents is decreased. This process applies for removal of particles sizes of  $5 \times 10^{-3}$  &  $5 \times 10^{-4}$   $\mu\text{m}$ . The reverse osmosis or hyper filtration, which is characterized by a membrane pore size in the range of 0.0005 microns. Some researchers consider the RO membrane as with out have pores. Transport of the solvent is accomplished through the free volume between the segments of the polymer of which the membrane is constituted. The operating pressures in RO are generally between 7 and 100 bar and this technique is mainly used to remove water. The importance of these membrane processes can be judged from the membrane area installed in the various industrial sectors. Membrane applications can be seen in below table:

| Process          | Applications   |
|------------------|--|
| Micro filtration | Sterile solution, water purification, beverage filtration effluents.                                   |
| Ultra filtration | Protein concentration (enzyme), oily wastewaters effluent, blood fractionation, antibiotic separation. |
| Nano filtration  | Potable water, desalination of brackish water, polyvalent ions stream cleaning, whey fractionation.    |
| Reverse osmosis  | Food concentration, water purification, desalination (monovalent ions stream), biomedical application. |
| Electro dialysis | Desalination, water purification, deacidification of citrus juice.                                     |
| Gas permeation   | Separation of He from natural gas, He recovery, CO <sub>2</sub> removal, NG dehydration                |
| Pervaporation    | Dehydration of organic solvents  |



**Table 1: Typical membrane applications**

### 1.2. Types of membranes

The different types of Polymers used for membrane manufacturing includes:

- Cellulose Acetate (**CA**) and Blends with di - and tri – Acetates
- Polyamide (**PA**)
- Polysulfone (**PS**)
- Sulfonated Polysulfone (**SPS**)
- Thin film composite and
- Hydrophilic and Hydrophobic **PTFE** membrane.

The above membranes differ in their properties such as surface pore size, pore distribution, rejection, flux, temperature stability, solvent resistance and pressure resistance. The choice of membranes for a particular application constitutes the most important step toward its process development. The factors affecting retentivity of any membrane include size and shape of molecules, presence of other solutes, type of membrane material, membrane configuration, and concentration of retained species and adsorption of solute into the membrane.

### 1.3. Configurations of membrane modules

Membranes are used in specific configurations to optimize design criteria like a high membrane surface to volume ratio, low pressure drop on the feed side, turbulent flow on the feed side, ease of membrane cleaning, long membrane life and low replacement cost.

The most common configurations available commercially are:

- Spiral Wound
- Tubular
- Hollow fiber &
- Plate and Frame

Industrial scale membrane systems are usually constructed with multiple modules to increase performance in terms of recovery, capacity, etc. Important multiple modular arrangements are as follows:

- Parallel mode
- Series mode
- Tapered mode



- Recycle mode

#### **1.4. Dyes**

There are more than 100,000 commercially available dyes with over million tones of dyestuff produced annually. Due to their chemical structure, dyes are resistant to fading on exposure to light, water, and many chemicals. Also many dyes are difficult to be decolorized and decomposed biologically. There are many structure varieties such as acidic, basic, disperse, azo, diazo, anthroquinone based, and metal complex dyes. These dyes are very stable and can be decomposed only at temperatures higher than 200<sup>0</sup>C. For this reason synthetic dyes often receive considerable attention from researchers in textile waste water treatment processes.

#### **1.5. Methods of treating dye containing wastewater**

Dye released to the waste stream, if without proper treatment, could exert great impact to the environment. Many textile manufacturers use dyes that release aromatic amines (eg., benzidine, toluidine). Dye bath effluents may contain heavy metals, ammonia, alkali salts, toxic solids and large amounts of pigments, many of which are toxic. About 40 percent of globally used colorants contain organically bound chlorine, a known carcinogen. Natural dyes are rarely low impact, depending on the specific dye and mordant used. Mordents (the substance used to "fix" the color onto the fabric) such as chromium are very toxic and has a high impact. Hence, to protecting the environment and to meet the stringent government laws, many researchers try to find an effective and economical way of treating dye containing wastewater. Several studies have been performed, that can be classified into on three categories depending on the method used. They are chemical, physical, and biological methods. Currently, the main methods of textile dye-containing wastewater treatment are physical and chemical methods.

## **2. LITERATURE REVIEW**

The common dyes are difficult to be degraded biologically; many researchers were interested in treatment of dye-containing wastewater. The studies were conducted on various kinds of dye wastewater using the biological process by Ahmad et al. They found that COD and the color of dye wastewater could not be efficiently removed.

The objective of this research <sup>[7]</sup> is to investigate the performance of blend cellulose acetate (CA)-polyethersulphone (PES) membranes prepared using microwave heating (MWH)



techniques and then compare it with blend CA-PES membranes prepared using conventional heating (CH) methods using bovine serum albumin solution. The superior membranes were then used in the treatment of palm oil mill effluent (POME). Various blends of CA-PES have been blended with PES in the range of 1-5 wt%. This distinctive series of dope formulations of blend CA/PES and pure CA was prepared using N, N-dimethylformamide (DMF) as solvent. The dope solution was prepared by MW heating for 5 min at a high pulse and the membranes were prepared by phase inversion method. The performances of these membranes were evaluated in terms of pure water and permeate flux, percentage removal of total suspended solids (TSS), chemical oxygen demand (COD) and biochemical oxygen demand (BOD). The results indicate that blend membranes prepared using the microwave technique is far more superior compared to that prepared using CH. Blend membranes with 19% CA, 1-3% PES and 80% of DMF solvent were found to be the best membrane formulation.

In chromium tanning process, about 20–40% of applied chromium is usually discharged into sewerage system causing serious environmental impact. The spent chromium effluents collected from two local small-size tanneries indicated that chromium concentration ranged from 1300–2500 mg Cr<sup>6+</sup>/l, while NaCl concentration varies from 40000 to 50000 mg/l. The study<sup>[5]</sup> of chromium removal efficiency by using of a pilot-scale setup of 7 and 16 bar RO membrane units at different working pressures and under variable salt concentrations were carried out to remove the hazardous chromium from the spent tanning effluent and recover it for further recycling. The study proved that the membrane technique is able to separate chromium efficiently from the pretreated tanning wastewater. The low cost RO membrane units, i.e. the medium and low pressure, could be used economically for separation and recovery of chromium from wastewater of small-size tanning shops.

The delivery of colour in the form of dyes onto textile fibres is not an efficient process. The degree of efficiency varies, depending on the method of delivery. As a result, most of the wastewater produced by the textile industry is coloured. It is likely that coloured wastewater was a feature of the first practices of textile dyeing. However, treatment to remove this colour was not considered until the early natural dyestuffs were replaced by synthetic dyes, and the persistence of such synthetic dyes in the environment was recognized. Colour pollution in aquatic environments is an escalating problem, despite the



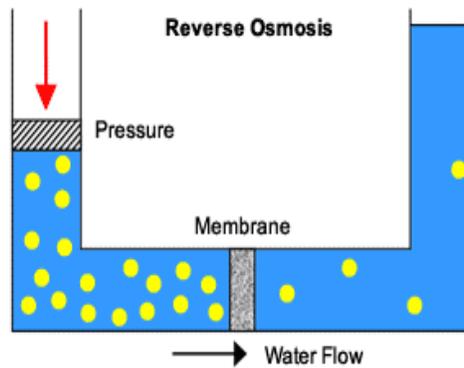
fact that there has been substantial research into the modification of the dyeing process to improve the level of affinity/fixation of the dyestuffs onto the substrate. The recalcitrant nature of modern synthetic dyes has led to the imposition of strict environmental regulations. The need for a cost effective process to remove the colour from wastewater produced by the textile industry has been recognized. Several strategies have been investigated <sup>[13]</sup>. However, the review presented here concerns the use of whole bacterial cells for the reduction of water-soluble dyes present in textile dyeing wastewater.

A modified microfiltration membrane has been prepared by blending a matrix polymer with a functional polymer. Cellulose acetate (CA) was blended with polyethyleneimine (PEI), which was then crosslinked by polyisocyanate, in a mixture of solvents. In the membrane, PEI can supply coupling sites for ligands in affinity separation or be used as ligands for metal chelating, removal of endotoxin or ion exchange. The effects of the time of phase inversion induced by water vapor, blended amount of PEI and amount of cross linking agent on membrane performance were investigated <sup>[22]</sup>. The prepared blend membranes have specific surface area of 12.04 – 24.11 m<sup>2</sup> /g and pure water flux (PWF) of 10–50 ml/cm<sup>2</sup> min with porosity of 63–75%. The membranes, made of 0.1550 wt% PEI/CA ratio and 0.5 cross linking agent/PEI ratio, were applied to adsorbing Cu<sup>2+</sup> and bovine serum albumin (BSA) individually. The maximum adsorption capacity of Cu ion on the blend membrane is 7.42 mg/g dry membrane. The maximum adsorption capacities of BSA on the membranes with and without chelating Cu ion are 86.6 and 43.8 mg/g dry membrane, respectively.

### 3. PROCESS DESCRIPTION

Osmosis is a natural phenomenon in which a solvent (usually water) passes through a semi permeable barrier from the side with lower solute concentration to the higher solute concentration side. Water flow continues until chemical potential equilibrium of the solvent is established. At equilibrium, the pressure difference between the two sides of the membrane is equal to the osmotic pressure of the solution. To reverse the flow of water (solvent), a pressure difference greater than the osmotic pressure difference is applied; as a result, separation of water from the solution occurs as pure water flows from the high concentration side to the low concentration side. This phenomenon is termed reverse osmosis. The phenomenon of reverse osmosis is illustrated in figure. A semi-permeable membrane is placed between two compartments. In this configuration, the direction of

solvent flow is determined by its chemical potential, which is a function of pressure, temperature and concentration of dissolved solids. Pure water in contact with both sides of an ideal semi-permeable membrane at equal pressure and temperature has no net flow across the membrane because the chemical potential is equal on both sides.



**Figure 1: Schematic representation of Reverse Osmosis**

Application of an external high pressure to the salt solution side will raise the chemical potential of the water in the salt solution and cause a solvent flow to the pure water side. These RO systems mainly remove a variety of ions and metals as well as certain organic, inorganic and bacterial contaminants by using pressure difference as a driving force. Reverse osmosis has always been noted for its low energy consumption. The principal uses of reverse osmosis are for the reduction of high levels of nitrate, sulfate, sodium and total dissolved solids. But a good pre-treatment is necessary for a reverse osmosis system and there is a possibility of a bacterial contamination.

### 3.1. Common uses of RO process

Reverse Osmosis is a technology that is found virtually anywhere pure water is needed; common uses include:

- Drinking Water
- Humidification
- Ice-Making
- Car Wash Water Reclamation
- Rinse Waters and
- Water used in chemical processes Biomedical, Laboratory & Pharmaceutical Applications

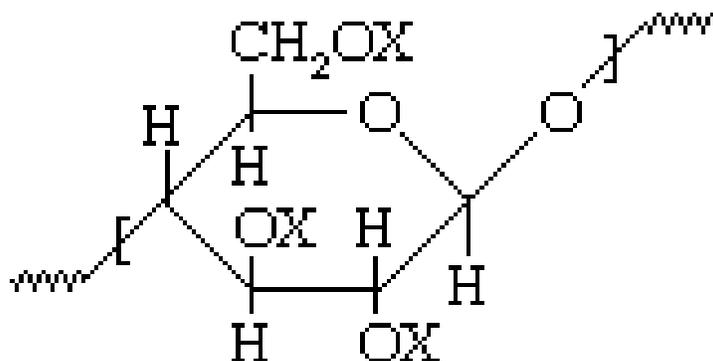
### 3.2. Cellulose acetate membrane



Cellulose acetate (CA) membranes were the first commercialized RO membranes developed in the late 1960's. CA membranes offered a good combination of rejection, fouling resistance, and the ability to tolerate continuous chlorine up to 1.0 ppm. Some of the reasons they lost favor to the new polyamide membranes was the requirement for acidification (CA membranes should be operated at a pH of between 4-6 to minimize hydrolysis), lower rejection (98% versus 99.5%) and higher net drive pressure requirements (300 psi/20 bar versus 150 psi/10 bar). But in many applications, CA membranes operate very well and give long and useful service lives. They have an advantage over the newer polyamide membranes for applications with high organic fouling, such as wastewaters, and waters where biological growth is an issue (easily addressed using chlorine). The commercial membrane elements are available in 2.5", 4", 8", 8.3", and 8.5" diameters. There are three different membranes offered, the SB20, SB50, and SB90. The SB20 offers the highest rejection at 98%, while the SB50 offers 20% higher flow at 95% salt rejection. The SB90 is a nanofiltration (NF) membrane operating at about twice the flow of the SB20 at 85-90% rejection.

### **3.3. Structure of cellulose acetate**

Cellulose Acetate, synthetic compound derived from the acetylation of the plant substance cellulose. Cellulose acetate is spun into textile fibers known variously as acetate rayon, acetate, or triacetate. It can also be molded into solid plastic parts such as tool handles or cast into film for photography or food wrapping, though its use in these applications has diminished. Cellulose is a naturally occurring polymer obtained from wood fibers or the short fibers (linters) adhering to cotton seeds. It is made up of repeating glucose units that have the chemical formula  $C_6H_7O_2(OH)_3$  and the following molecular structure:



**Figure 2: Structure of Cellulose acetate.**

In unaltered cellulose, the X in the molecular structure represents hydrogen (H), indicating the presence in the molecule of three hydroxyl (OH) groups. The OH groups form strong hydrogen bonds between cellulose molecules, with the result that cellulose structures cannot be loosened by heat or solvents without causing chemical decomposition. However, upon acetylation, the hydrogen in the hydroxyl groups is replaced by acetyl groups (CH<sub>3</sub>-CO). The resultant cellulose acetate compound can be dissolved in certain solvents or softened or melted under heat, allowing the material to be spun into fibers, molded into solid objects, or cast as a film. Cellulose acetate is most commonly prepared by treating cellulose with acetic acid and then with acetic anhydride in the presence of a catalyst such as sulfuric acid. When the resultant reactions are allowed to proceed to completion, the product is a fully acetylated compound known as primary cellulose acetate, or, more properly, cellulose triacetate. Triacetate is a high melting (300<sup>o</sup>C), highly crystalline substance that is soluble only in a limited range of solvents (usually methylene chloride). From solution, triacetate can be dry spun into fibers or, with the aid of plasticizers, cast as a film. If the primary acetate is treated with water, a hydrolyzation reaction can occur in which the acetylation reaction is partially reversed, producing a secondary cellulose acetate, or cellulose diacetate. Diacetate can be dissolved by cheaper solvents such as acetone for dry spinning into fibers. With a lower melting temperature (230<sup>o</sup>C) than triacetate, diacetate in flake form can be mixed with appropriate plasticizers into powders for molding solid objects, and it can also be cast as a film.

### 3.4. Properties of cellulose acetate membranes

- Hydrophobic membrane is ideal for use in venting applications.



- Resists water while simultaneously venting gases.
- 'Hydrophobic' means 'fear for water'. Hence the water does not wet the membrane surface. Strong and durable for easy handling.
- Lot-to-lot consistency with quality checks which ensure lot-to-lot consistency, both down and across the polyester web, for dependable results every time.
- Hydrophobic membrane is a pure polymer internally supported with an inert polyester web.

### 3.5. Experimental procedure

The actual project deals with the treatment of sea water to get the drinking water and treatment of effluent water from textile industries to remove the dyes. For this the pH, total hardness, chemical oxygen demand and biological oxygen demand are the parameters should be measured to prove that water is suitable for drinking.

In the series of steps involved in the sea water treatment, we focus our research to the reverse osmosis process. For that purpose the synthetic effluent is treated in the column which contains cellulose acetate membrane. Reverse osmosis is a liquid filtration method which removes many types of large atomic molecules from smaller molecules, by forcing the liquid at high pressure through a semi permeable membrane with pores (holes) just big enough to allow the small molecules to pass through. The semi permeable membrane in different stages used here is cellulose acetate.



Figure 3: The reverse osmosis column



The effluent samples (Dye samples) taken for this process are

1. Torquise blue dye solution - Sample A
2. Remozol golden yellow dye solution - Sample B

These dyes are normally present in the textile effluent. Colourimeter is concerned with the determination of concentration of substance by measurement of the relative absorption of light with respect to a known concentration of the substance. The light passed is absorbed by the colour present in the dye solution. This becomes the principle to find the concentration as per Beer-Lambert's law.

In order to estimate the concentration of these synthetic dye solutions, a series of standard solutions are prepared. Then, colorimeter is set to zero absorbance using a blank solution (distilled water) with a proper filter. Absorbance of each standard solution is then measured using the same filter. The absorbance value obtained is plotted versus concentration. This plot is called calibration curve. When a species under test is colourless, then a suitable complexig agent is added to the solution so that coloured complex (which can absorb light in the visible region) is obtained. The absorbance concentration data and calibration curve for the two dye solutions are given below.

| Concentration (ppm) | Absorbance |
|---------------------|------------|
| 0                   | 0          |
| 10                  | 0.11       |
| 20                  | 0.17       |
| 30                  | 0.29       |
| 40                  | 0.37       |
| 50                  | 0.45       |
| 60                  | 0.57       |
| 70                  | 0.68       |
| 80                  | 0.76       |
| 90                  | 0.89       |
| 100                 | 0.98       |
| 110                 | 1.06       |
| 120                 | 1.18       |



|     |      |
|-----|------|
| 130 | 1.25 |
| 140 | 1.38 |
| 150 | 1.45 |
| 160 | 1.56 |
| 170 | 1.68 |
| 180 | 1.77 |
| 190 | 1.83 |
| 200 | 1.85 |

Table 2: Absorbance for various concentrations - Torquise blue dye solution

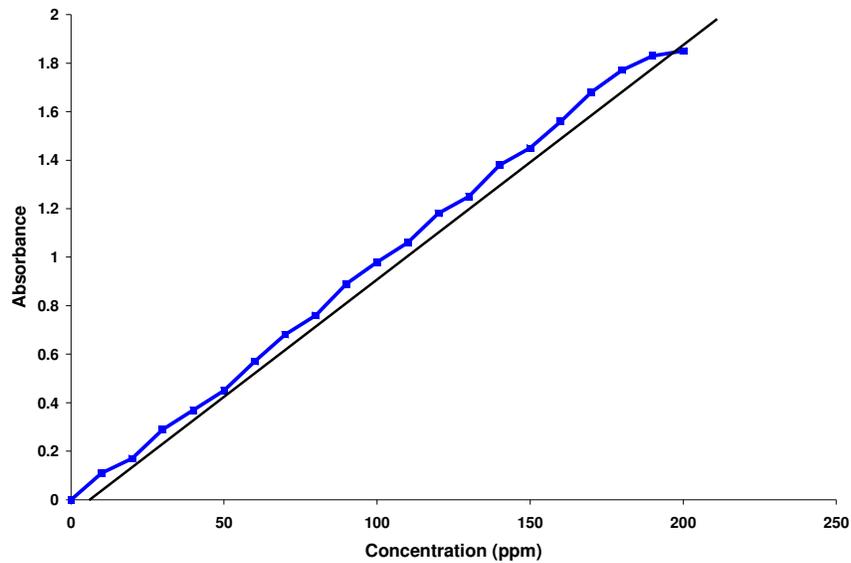


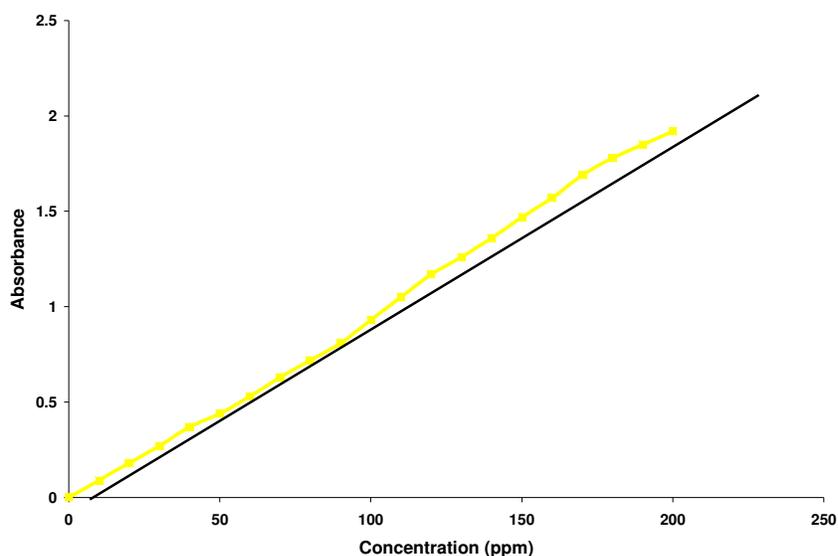
Figure 4: Calibration curve for Torquise blue dye solution

| Concentration (ppm) | Absorbance |
|---------------------|------------|
| 0                   | 0          |
| 10                  | 0.09       |
| 20                  | 0.18       |
| 30                  | 0.27       |
| 40                  | 0.37       |
| 50                  | 0.44       |
| 60                  | 0.53       |
| 70                  | 0.63       |
| 80                  | 0.72       |
| 90                  | 0.81       |
| 100                 | 0.93       |
| 110                 | 1.05       |
| 120                 | 1.17       |



|     |      |
|-----|------|
| 130 | 1.26 |
| 140 | 1.36 |
| 150 | 1.47 |
| 160 | 1.57 |
| 170 | 1.69 |
| 180 | 1.78 |
| 190 | 1.85 |
| 200 | 1.92 |

**Table 3: Absorbance for various concentrations – Remazol golden yellow dye solution**



**Figure 5: Calibration curve for Remazol golden yellow dye solution**

### 3.6. Effluent treatment (Dye solution) – Torquise blue dye

| S.No | Sample  | Absorbance | C   | C/C0 |
|------|---------|------------|-----|------|
| 1.   | Inlet   | 1.85       | 187 | 0.94 |
| 2.   | Stage 1 | 1.52       | 154 | 0.77 |
| 3.   | Stage 2 | 0.74       | 76  | 0.38 |
| 4.   | Stage 3 | 0.32       | 34  | 0.17 |
| 5.   | Stage 4 | 0.24       | 27  | 0.14 |



**Table 4: The absorbance at different stages determined by colorimeter**

Inlet:-

|   |   |         |
|---|---|---------|
| Initial concentration, C <sub>0</sub>               | = | 200 ppm |
| Volume of feed, V                                   | = | 500 ml  |
| Absorbance of initial concentration, A <sub>i</sub> | = | 1.85    |

Stage 1:-

|  |   |                                  |
|--|---|----------------------------------|
| Absorbance after stage 1 is              | = | 1.52                             |
| From calibration curve,<br>Concentration | = | 154 ppm                          |
| Percentage removal for stage 1 is        | = | $((200 - 154) / 200) \times 100$ |
|  | = | 23%                              |

Stage 2:-

|  |   |                                 |
|--|---|---------------------------------|
| Absorbance after stage 2 is              | = | 0.74                            |
| From calibration curve,<br>Concentration | = | 72 ppm                          |
| Percentage removal for stage 2 is        | = | $((200 - 72) / 200) \times 100$ |
|  | = | 64%                             |

Stage 3:-

|  |   |                                 |
|--|---|---------------------------------|
| Absorbance after stage 3 is              | = | 0.32                            |
| From calibration curve,<br>Concentration | = | 34 ppm                          |
| Percentage removal for stage 3 is        | = | $((200 - 34) / 200) \times 100$ |
|  | = | 83%                             |

Stage 4:-

|  |   |                                 |
|--|---|---------------------------------|
| Absorbance after stage 4 is              | = | 0.24                            |
| From calibration curve,<br>Concentration | = | 27 ppm                          |
| Percentage removal for stage 4 is        | = | $((200 - 27) / 200) \times 100$ |
|  | = | 86.5%                           |



### 3.7. Effluent treatment (Dye solution) – Remozol golden yellow dye

| S.No | Sample  | Absorbance | C   | C/C0 |
|------|---------|------------|-----|------|
| 1.   | Inlet   | 1.92       | 194 | 0.97 |
| 2.   | Stage 1 | 1.68       | 170 | 0.85 |
| 3.   | Stage 2 | 0.81       | 83  | 0.42 |
| 4.   | Stage 3 | 0.39       | 41  | 0.21 |
| 5.   | Stage 4 | 0.27       | 29  | 0.15 |

**Table 5: The absorbance at different stages determined by colorimeter**

#### Inlet:-

Initial concentration, C<sub>0</sub> = 200 ppm

Volume of feed, V = 500 ml

Absorbance of initial concentration, A<sub>i</sub> = 1.92

#### Stage 1:-

Absorbance after stage 1 is = 1.68

From calibration curve,

Concentration = 164 ppm

Percentage removal for stage 1 is =  $((200 - 164) / 200) \times 100$   
= 18%

#### Stage 2:-

Absorbance after stage 2 is = 0.81

From calibration curve,

Concentration = 79 ppm

Percentage removal for stage 2 is =  $((200 - 79) / 200) \times 100$   
= 60.5%

#### Stage 3:-

Absorbance after stage 3 is = 0.39

From calibration curve,

Concentration = 37 ppm

Percentage removal for stage 3 is =  $((200 - 37) / 200) \times 100$   
= 81.5%



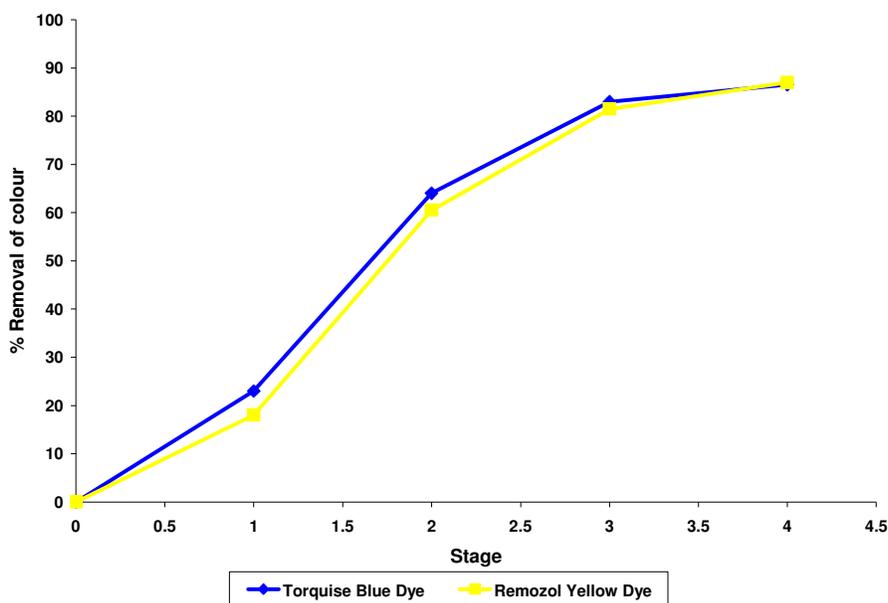
Stage 4:-

Absorbance after stage 4 is = 0.27  
From calibration curve,  
Concentration = 26 ppm  
Percentage removal for stage 4 is =  $((200 - 26) / 200) \times 100$   
= 87%

**3.8. Comparison of percentage removal for sample A & sample B**

| S.No | Stage   | % removal in sample A | % removal in sample B |
|------|---------|-----------------------|-----------------------|
| 1.   | Inlet   | --                    | --                    |
| 2.   | Stage 1 | 23                    | 18                    |
| 3.   | Stage 2 | 64                    | 60.5                  |
| 4.   | Stage 3 | 83                    | 81.5                  |
| 5.   | Stage 4 | 86.5                  | 87                    |

**Table 6: percentage of colour removal for both dye samples**



**Figure 6: Comparison of colour removal for the samples**

**CONCLUSION**

The main objective of this research is to remove the colour from synthetic textile dye effluent by membrane technology. The two dyes which are commonly available in the textile effluent are Torquise blue dye and Remozol golden yellow dye solutions. The membrane used for reverse osmosis is cellulose acetate membrane and gravitational force



is used as a major driving force. Many of the systems of separation use pressure gradient as the main driving force, but here we are keeping gravitational force as the main driving force with less amount of inlet pressure. These two different dye samples are passed through the column which contains the cellulose acetate membrane and the outlet concentrations are measured at different stages using calorimeter. The result shows that around 90% of the colour is removed by using cellulose acetate membrane in reverse osmosis process.

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