TREATMENT OF WOOD AND AGRO BASED PAPER MILL WASTEWATER IN INDIA

Thesis submitted
in fulfillment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

In

ENVIRONMENTAL ENGINEERING

By

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Roll No. 2K19/PHDEN/004

Under the Supervision of
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September, 2024

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DECLARATION

I hereby declare that the research work presented in this thesis entitled "Treatment of wood and agro based paper mill wastewater in India" is original and carried out by me under the supervision of Prof. S. K. Singh, Professor and Head, Department of Environmental Engineering, Delhi Technological University, Delhi, and under the co-supervision of Dr. Manoj Kumar Gupta, Director, CPPRI, Saharanpur, UP, and being submitted for the award of Ph. D degree from Delhi Technological University, Delhi, India. The content of this thesis has not been submitted either in part or whole to any other university or institute for the award of any degree or diploma.

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CERTIFICATE

This is to certify that the Ph.D. thesis entitled "Treatment of wood and agro based paper mill wastewater in India" being submitted by Mr. Sumit Dagar for the award of the degree of Doctor of Philosophy in Environmental Engineering, to Delhi Technological University, Delhi, India, is a bonafide record of original research work carried out by her under our guidance and supervision. The results embodied in this thesis have not been submitted to any other university or institution for the award of any degree or diploma.

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ACKNOWLEDGEMENT

With a deep sense of gratitude, I thank the almighty who blessed my life throughout and has added another beautiful and successful chapter in my life with this thesis. Without his grace it was never possible for me to reach this point. I am grateful to "GOD", for bestowing me with affectionate parents who encouraged me to undergo higher studies. As I reach another cornerstone in my academic career, I wish to express my eternal feeling to those, whose support and motivation made possible to present my research work in the form of a thesis.

I express my profound sense of gratitude and sincere thanks to my respectable supervisor **Prof. S.K Singh, Vice Chancellor, Rajasthan Technical University** for his words of wisdom, scholastic guidance, knowledge and constant encouragement throughout the progress of my doctorate programme. I am thankful to him for his valuable suggestions, prudent guidance and insightful suggestions during the course of this work and until the completion of the present investigation.

I wish to express my sincere thanks to my co-supervisor **Dr. Manoj Kumar Gupta, Director, Central Pulp and Paper Research Institute, Saharanpur, U.P** for support throughout this period and for their instinctive help, guidance, suggestions and encouragement at each step of my research.

I offer my sincere gratitude to **Dr. A.K. Haritash, Head, Department of Environmental Engineering,** for his valuable suggestions and constant encouragement at every step of my research and providing me with all the necessary facilities for the research. I am thankful to the **Vice Chancellor, Delhi Technological University** for regularly upgrading and providing optimum lab facilities and sophisticated instruments needed to carry out the in-depth analysis.

My special thanks to **Mohd. Salim, Scientist B, Central Pulp and Paper Research Institute, Saharanpur, U.P**, who generously offered his time and resources during my research. I would also like to thank **Mr. Abhishek Tyagi, Research Fellow at CPPRI** for his constant support

I can hardly overlook the immense cooperation, timely help and moral support provided to me by my lab mates **Daan Kaur** and **Riki**.

I convey my heartiest thanks to non-teaching staff **Mr. Sandeep** and **Mr. Deepak** for their help during the entire span of study.

I am indebted to my respected and beloved Mother **Smt. Santosh Devi** and Father **Sh. Jagdish Dagar,** the human form of God, for their support, encouragement and unfathomable faith in my abilities. They have guided me and kept their hand on me in all my smooth and rough phases. Their selfless persuasion and sacrifice, heartfelt blessings and firm faith has made this manuscript a reward to translate their dreams into reality.

I would be selfish if I do not appreciate the constant inspiration, encouragement, patience and ever available help of elder Brother **Mr. Amit Dagar** for their unconditional support and encouragement. I would also like to express my appreciation to my other family members who boosted me up morally.

My nephew **Dhawan** also deserves thanks as his cute face had always taken my pains and stress away and for their selfless love and affection that always kept me in a delightful mood.

Sumit Dagar

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LIST OF ABBREVIATIONS

CPPRI	Central Pulp & Paper Research Institute
ETP	Effluent Treatment Plant
ANOVA	Analysis of Variance
TDS	Total dissolved solids
TSS	Total suspended solids
COD	Chemical oxygen demand
BOD	Biochemical oxygen demand
DO	Dissolved Oxygen
RO	Reverse Osmosis
DAF	Dissolved Air Floater
UF	Ultrafilteration
MoEF	Ministry of Environment and Forests
CPCB	Central Pollution Control Board
IPPC	Intergovernmental Panel on Climate Change
AKD	Alkyl Ketene Dimer
ATC	Anioinic Trash Catcher
RDA	Redundancy analysis
FAOSTAT	Food and Agriculture Organization Corporate Statistical Database
RMP	Refiner Mechanical Pulp
TMP	Thermo-mechanical Pulp
CTMP	Chemi-thermo-mechanical Pulp
EC	Electrical Conductivity
AOX	Adsorbable Organic Hallides
ECF	Elemental Chlorine Free
PPT	Parts per thousand
EPA	Environmental Protection Agency
HMW	High Molecular Weight
LMW	Low Molecular Weight
PAC	Powdered Activated Charcoal
AOP	Advanced Oxidation Process
UV	Ultra-Violet
MS	Mass Spectrometry

GC	Gas Chromatography
TOC	Total Organic Carbon
PTFE	Polytetrafluoroethylene
PE	Polyethylene
MF	Microfiltration
NF	Nanofiltration
MWCO	Molecular Weight Cutoff
RSM	Response Surface Methodology
SEM	Scanning Electron Microscopy
IMS	Integrated Membrane system
ABS	Absorbance per path length
ZIF	Zinc Imidazole Framework
TP	Total phosphorus
VRF	Volume Reduction Factor
FRI	Fluorescence Regional Integration
APHA	American Public Health Association
IS	Indian Standards
FAS	Ferrous Ammonium Sulfate
FD	Flux Decline
OA	Orthogonal arrays
DF	Degree of Freedom
GRG	Grey Relational Grade
SPSS	Statistical Package for the Social Sciences
PCA	Principal Component Analysis
TPD	Tons per day
NTU	Nephelometric Turbidity unit
PCU	Platinum Cobalt Units
DMF	Direct Membrane Filter
SDB	Sludge Bed Drier
OCEMS	Online Continuous Effluent Monitoring System
SWD	Salt Water Disposal
PPM	Parts per million
SOR	Surface Overflow Rate
VLR	Vertical Loop Reactor

COD_R	Chemical Oxygen Demand Rejection
BIS	Bureau of Indian Standards
CAPEX	Capital expenditure
OPEX	Operational expenditure
TOPEX	Total operational expenditure
LPH	Liters per hour
KLD	Kilo liters per day
MLD	Mega liters per day
NaCl	Sodium Chloride
CO_2	Carbon dioxide
Cl_2	Chlorine
Na_2CO_3	Sodium Carbonate
Na_2S	Sodium sulfide
$NaHSO_3$	Sodium bisulfite
NaOH	Sodium hydroxide
$CaCl_2$	Calcium Chloride
H_2O_2	Hydrogen Peroxide
HNO_3	Nitric acid
H_2SO_4	Sulfuric acid
S_2O_8	Peroxydisulfate
$K_2Cr_2O_7$	Potassium dichromate

CHAPTER 1
INTRODUCTION

1.1 Background

Water is the most pivotal and elementary resource for the survival of human beings on the planet. Due to its necessity for residential, industrial, and agricultural purposes and its limited availability it has to be considered as the most important resource. Inadequate availability, supply, changing climatic patterns, and the deterioration of the quality of water are a few of the major issues concerning most parts of the World. Because of the uneven distribution of water resources, ever-increasing human population, urbanization, pollution and contamination of both surface and groundwater are the major issues pertaining to water resources (Ahmed et al., 2021). According to Musie and Gonfa, by 2023, more than 2.5 billion people in developing nations will be under severe water scarcity by 2025. Unplanned and injudicious use by the industries is the main reason for this present issue of water crisis. Increased use of chemicals, incorrect applications, and inappropriate regulations by the authorities are putting huge pressure on the natural resource. Discharge of untreated effluent depending on the amount of water they consume and the concentration of different pollutants negatively affects the water quality as well as the aquatic life. The pulp and paper processing industries, egg processing industries, and rice mills are considered the most water-intensive industries utilizing huge quantities of chemicals in their process. The Ministry of Environment and Forest (MoEF) has categorized industries based on the pollution they discharge into Red, Orange, Green, and White Industries. Red Category industries are typically considered to be highly polluting in nature followed by orange, green, and white. Red category industries have 17 industries on the list, and the paper and pulp industries are sixth among the same (CPCB, 2001). The pulp and paper manufacturing industries are traditionally important, economically crucial, and decisive industries for both developed and developing nations. It has renowned and esteemed value in commercial as well as financial expansion of the nation. And because of the crucial role of paper since ages as a prime medium of record, its production day by day is increasing.

Table 1.1: Typical Water consumption ton of the product by different industries.

Industry	**Water Requirement (in Gallons)**
Paper	**21,000-528,000**
Sugar	**792-105,668**
Steel	**528-92,460**
Soap	**264-9,246**
Gasoline	**26-10,566**

Source: World's water assessment program of United Nations, Report: water in a Changing World, 2009.

1.2 Indian paper industry

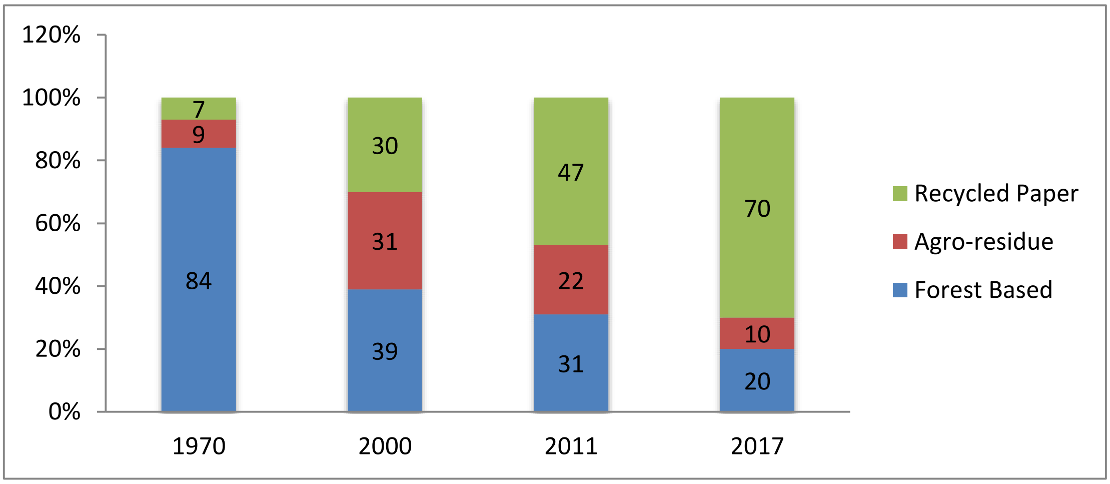
India ranks fifteenth in the World in terms of paper production as about 884 small and large-scale paper manufacturing and pulp processing units are operational in India. It provides large-scale employment to nearly 1.3 million people and is very pivotal for the technology-based economic growth of India, with a net worth of around INR. 25 billion (Jain et al., 2009). The industries depending on their production capacity have been categorized into large, medium, and small-scale industries. Industries having a production capacity of more than 24,000T/annum are considered large scale and the rest are small-scale. Among 884 mills 15% are large, 40% are medium and 45% are small scale industries. Out of total paper production come 60% and 40% from small and large mills respectively. The raw materials used for paper production are cellulose fibers usually coming from wood, agricultural waste, and recycled paper. Kulkarni, 2013, depending on the raw material used in various production units categorized the mill into three groups:

a) **Wood and Forest-based Mill:** These industries use wood and bamboo and account for around 31% of the total paper production.

b) **Agro-based Mill:** These units utilize the agro-residues like cereal straw, jute, rags, bagasse, reeds, bamboo, etc to produce 22% of the gross paper production (Raaz et al., 2012)

c) **Recycled paper Mill:** These utilize waste paper and contribute to the maximum paper production i.e. 47%.

Over a while, the percentage of forest-derived raw materials to the net raw material consumption has diminished due to the fast depletion of forest resources, gradual price rise, and increased prohibitory regulation by the Government (Figure 1.1). The government encouraged the use of agricultural residue to reduce the stress on the forest resources and it also leads to sustainability. Moreover, the pulp formation from agricultural waste is less time-consuming as compared to wood pulping, hence lesser energy and water requirement. However, after the year 2000, there has been a tremendous decrease in demand for agricultural residues due to the constraint of its shorter fiber length and width, seasonal availability, high transportation cost, and emphasis on environmental protection by recycling wastes.



(Source- Annual Report, CPPRI, Saharanpur, 2018-19)

Figure 1.1: Raw material Consumption pattern In Indian Pulp processing and paper Industries

1.3 Pulp processing and paper-manufacturing process

The steps utilized for paper formation include pulping, bleaching, and paper-making. The pulp processing starts with the preparation of raw material. For this, the wood is debarked and chopped into chips and the bagasse is depicted. The bark and pith present lead to scaling and affect the quality of the pulp. Also, the presence of bark/pith increases the consumption of alkali and reduces pulp yield and pulp strength. Moreover, its removal decreases the organic content in the effluent.

After debarking/depithing, the raw material is converted into small chips for further processing. The conversion of wooden chips into pulp includes chemical cooking, wherein the single cellulose fibers are produced by the reduction of wood or other fibrous materials. For the purpose of chemical pulping various chemicals like Caustic soda, Sulfuric acid, Sodium sulfide, hydrogen peroxide, etc. are used to split the carbohydrate and lignin bonds, to depolymerize the lignin and finally convert it into a soluble form (Li et al., 2021 and Ragauskas et al., 2014). In the process of pulping, the wooden chips and other non-wood-containing raw materials are baked in an aqueous solution. While cooking the lignin gets dissolved and leaves most of the cellulose and hemicelluloses as intact fiber. After this, the cooked pulp is processed for washing to remove the impurities and recycle the remaining cooking liquor. These steps help in the chemical recovery of cooking liquor and also significantly reduce its entry into bleaching plants. The pulp after washing is subjected to bleaching to increase the brightness of the paper. This process also removes the residual lignin. The pulping can either be done mechanically or chemically. Mechanical pulp bleaching is considered lignin-preserved bleaching since chemical bleaching attacks the carbohydrates and also decreases the polymerization of cellulose hence affecting weaker fibers with lesser strength.

The transformation process from pulp to paper is known as stock preparation. The varieties of pulp stores are drawn to the receiving chest of the paper-making machine. The pulp is then passed through the refiner to develop strength and blended in the blend chest to the required proportions. In the blend chest, different synthetic sizes like Alkyl Ketene dimer (AKD), size fixing agents like Anionic trash Catcher (ATC), and filler material like ash are mixed into the pulp. Then this blended chest is transferred to a Machine chest for control refining, dilution with backwater, deaerated, and finally cleaned in a deculator and cleaning system of paper paper-making machine. This cleaned stock is screened through pressure screens and after the addition of RDA chemicals finally sent to paper machines. The sheet formation takes place on one of the two wire formers present in the paper machine, where large-scale dewatering takes place. The paper sheets are then passed through dryers that are steam heated so that the required level of dryness can be attained and then passed through a calendar stack that consists of tow rolls to control the surface properties and finally rolled into jumbo rolls which are then cut as per the market demands. The schematic flow chart of paper production in the mill is shown in Figure 1.2.

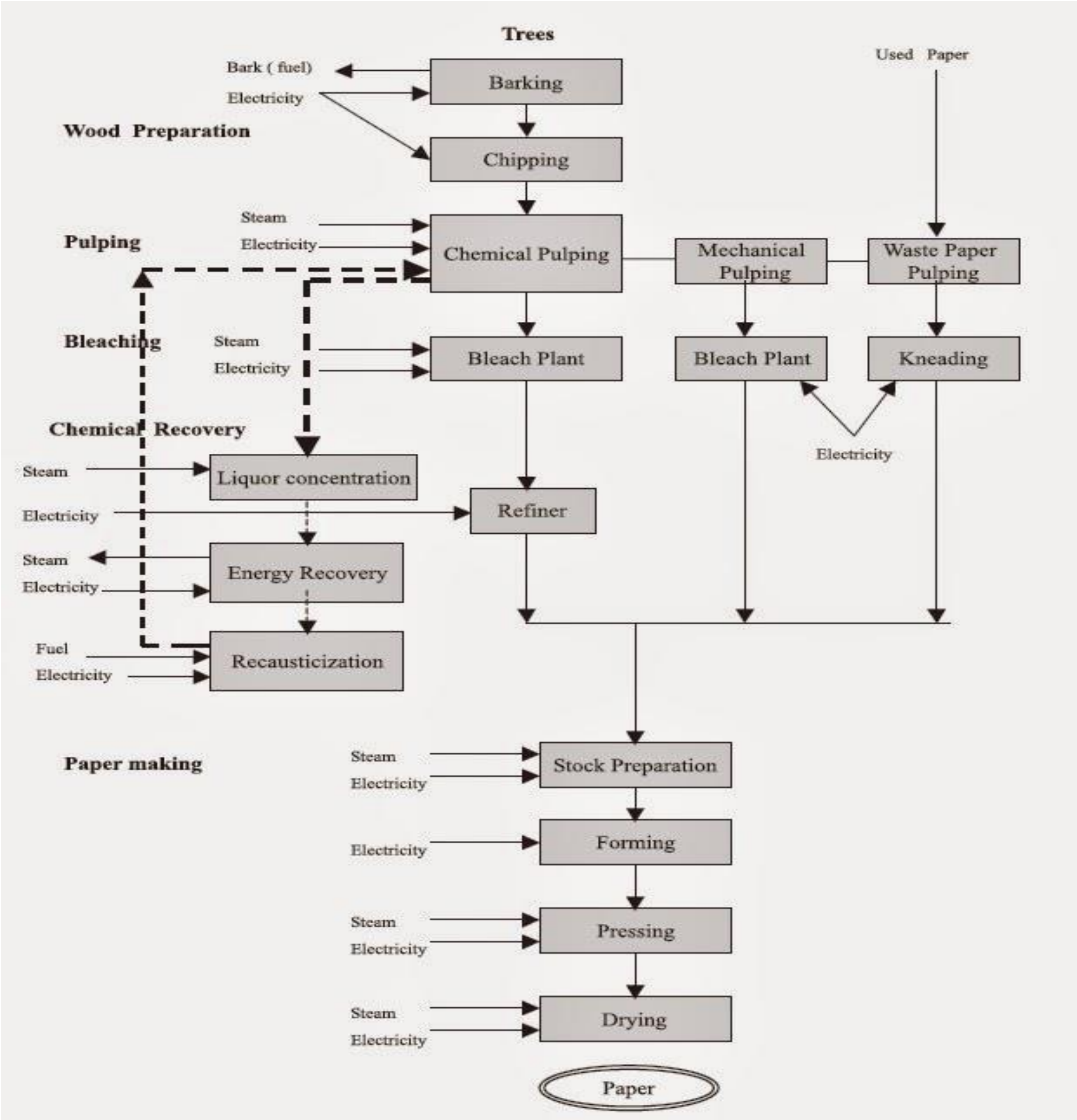


Figure 1.2: Paper-making Process

These paper and pulp processing mills utilize huge amounts of money. The average water consumption at various manufacturing stages in the paper-making process is shown in Figure 2. For every tonnage of paper production, 220-380m^3 of highly polluted, colored, and potentially toxic water is released by the pulp processing and paper manufacturing units. Along with the huge deteriorated quality of water, massive amounts of lignocellulosic material, chlorinated resin acids, chlorinated lignosulfonic acids, and hydrocarbons are also released as effluents (Sharma and Singh, 2021). Around 500 Different Chlorinated and organic compounds including resins, furans, catechols, dioxins, etc. have been identified in the effluent of pulp processing industries. The process of pulping and its bleaching produces most of the waste generation in all three phases viz. solid, liquid, and gas.

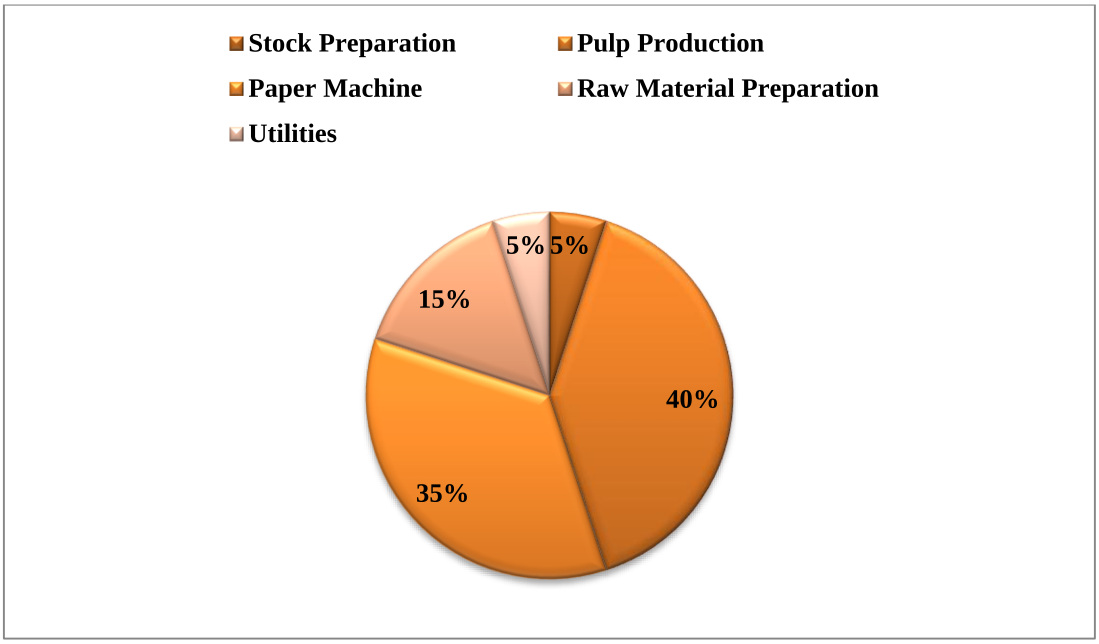


Figure 1.3: Water Usage in Different Steps of Paper Making

1.4 Environmental damage caused due to the pulp and paper processing industries

The parameters corresponding to generated effluent depend upon the process of pulping used and the raw material used. The higher the amount of oxygen-demanding substances increases, the more the BOD and COD of the water. Major oxygen-demanding waste comprises the residual lignin and other molecular fractions. The color imparted by the wood extractives and the degradation of lignin in the wastewater generated are also major concerns. These entire fractions are not easily degradable and, therefore need an efficient treatment system that can effectively reduce the pollutants.

The chemical used in pulp and paper processing and manufacturing causes a variety of carcinogenic, mutagenic, and clastogenic effects on both aquatic and terrestrial life (Kumar et al., 2022). The brown color of lignin once released in aquatic bodies inhibits the penetration of sunlight and hence inhibits photosynthetic processes in primary producers. It also reduces the dissolved oxygen content in the water body which affects the producers and consumers of aquatic water bodies (Xiao et al., 2007). The suspended particles and the loss of nutrients can also lead to eutrophication of the water bodies that receive waste water discharge from industries (Thompson et al., 2001). With the effluent release from such industries detrimental impacts on the respiratory system, damage to the liver, inhibition of enzymes and genotoxicity have been observed in the fish (Thomson et al., 2001). When it comes to terrestrial ecosystems, this wastewater reduces the soil fertility and productivity of crops. Some of the toxic chemicals can also bioaccumulate in cereal crops to enter the human food chain, impacting the health of humans by causing severe metabolic aberration (Tiku et al., 2010). Toxic compounds like dioxins cause severe health hazards like hormonal imbalance, deceased reproductively and fetus development, immune system deterioration, etc. (Kulkarni et al., 2013; Sun et al, 2015). The untreated or partially treated water when discharged may become a major source of groundwater contamination surging the level of toxic compounds including xenobiotics, heavy metals, etc. (Chou et al., 2014; Richardson and Kimura 2017).

Various conventional technologies are used for the elimination of toxic compounds from industrial effluents which include coagulation, flocculation, sedimentation, precipitation, filtration, adsorption, absorption, ozonation, reverse and forward osmosis, and oxidation-reduction processes (Greenlee et al., 2010).

The process of sedimentation focuses on the TSS removal by settling from the effluent under the influence of gravity. But the rate as well as the degree of separation varies according to the size of the particles, hence only particles of large size can settle down easily.

The process of floatation allows the small particles to float on the surface of water. These techniques have been used in mining and mineral separation since ages. The air present in water collects on the surface of the water and forms tiny bubbles. These bubbles may get attached to the particle surface by combining and trapping the air and forming agglomeration of air and particles to form the apparent density less than water and make them float on the surface of the water. In Pulp processing units the holding tanks are designed to provide an upward flow of water allowing the fiber floc to float on the surface of water. The compressed air is pumped into the clarifiers to promote the formation of suspended solid flocs. Coagulation is the process of adding chemicals to increase the capacity of floc formation by agglomeration. The floc forms larger and more massive floc and then settles at the bottom of the clarifier as sludge.

Chemical oxidizing agents like Chlorine dioxide, hydrogen peroxide, hypochlorite chlorine, etc. are also utilized to achieve the destruction of both toxic and chromophoric compounds. However, the addition of these oxidants makes it an expensive technique. And also, these oxidizing agents generated unwanted organochlorine byproducts like chloroform making it not a promising one to treat the effluent.

Various ion species can also be eliminated from the wastewater effluent by the use of ion exchange technique or by adoration on the adsorbents like alumina. In ion exchange, the effluent passes through a packed bed comprising ion exchange bead resins or alumina. However, this technique has been observed to be limited because of the bed exhaustion which needs to be regenerated which makes it time-consuming as well as costly.

The electrochemical treatment technique uses the process of electrolysis to convert the chlorine from the bleaching process into chlorite and hypochlorite. This is a Redox process driven by electrochemical energy. However, its high operating cost makes it inefficient to be used in paper production mills.

Biological treatment involves the use of microorganisms that survive on the present organic matter residue in the wastewater and carry out the conversion of the various organic pollutants into simpler and less toxic products. For this, a biological system is maintained which provides conditions to promote and maintain microbial growth. But the rate of removal depends upon the species and it may vary from a few hours to days depending upon the microbial activity. Also, when this technique is applied in pulp processing and paper manufacturing mills, the color of water usually increases because of the formation of smaller chromophoric compounds by the breakdown of organic materials, making it inappropriate for further use.

Membrane filtration has come up as one such technique that has been used for many years for the desalination of water. However, its application in effluent treatment generated by paper and pulp mills is still debatable. Since not many databases/case studies are available based on the applicability of membrane filtration technology for the recycle, reuse, and restoration of effluent generated from wastewater generated by pulp and paper mills in India perspectives. Also, there is no comprehensive comparative evaluation of the efficacy of different treatment technologies for wastewater in India and Techno-economic feasibility needs to be established regarding the mills that process Pulp and Paper in India. Hence, the following objectives have been selected to fill the gaps in the research.

1.5 Objectives of the Study

1. Study of existing and advanced treatment technologies for wastewater treatment in the pulp and paper industry.
2. To explore the possibilities for reduction of pollution load especially removal of color, lignin, and TDS in the pulp and paper industry with the application of advanced technologies such as membrane filtration.
3. To facilitate increased reuse and recycling of treated water back into the process and reduce the water footprint of pulp and paper mills.
4. To analyze the feasibility of relatively recent technologies for wastewater treatment in the Indian pulp and paper industry.

To achieve the above-mentioned objectives following work plan was formulated and followed

1. Primary data and information about the effluent treatment process and

treatment facilities available at selected wood/agro-based mills were collected.

2. The effluent generated and treated effluent samples collected from selected agro/wood-based mills were characterized.

   Various variables like pH, Temperature, and Volume Reduction factors for the treatment of effluent generated from pulp and paper mill through membrane filtration system were optimized.
3. The performance of membrane filtration technology with respect to various pollution parameters especially TDS, COD, BOD, Color, and Lignin was evaluated.
4. Analysis of the residue obtained after treatment through membrane filtration was done.
5. The techno-economic aspect of the membrane filtration system for the treatment of mill effluent was estimated.

CHAPTER 2
REVIEW OF LITERATURE

2.1 World Paper Consumption Scenario

The rapid population growth has led to the increased establishment of industries to meet their needs. The paper and pulp processing and manufacturing mills are one of the largest establishments which are growing at the same rate as World's the Gross Domestic Product (Gupta and Gupta, 2019). It is one of the core and largest industrial sectors. The world's total production of paper in 2017 has exceeded more than 419.7 million tons (FAOSTAT) and is expected to increase in the future. Several countries like Japan, China, and the U.S. are the major paper producers, and countries like Finland, Brazil, Germany, Sweden, and Canada also contribute to more than half of its total production. The per capita consumption of paper in the US and China is 29 Kg and 74 Kg respectively, which is highest when compared to worldwide average usage (*i.e.*, 57 kg) (http://www.careratings.com). Manufacturing units of the paper and pulp industry are multiplying worldwide because of an increased demand for packaging of paper and tissues.

The major input raw material for the paper and pulp industry is based on the fibers extorted from wood, agro-residues, and recycled paper (Negi and Rajput, 2013). These industries consume huge amounts of freshwater, lignocellulose material like agro-waste (bagasse, straw), jute, bamboo, wood, waste paper, and numerous inorganic chemicals such NaOH, Na_2S, $NaHSO_3$, Na_2CO_3, Cl_2, ClO_2, CaO, etc. as basic raw material for processing and manufacturing of paper (Negi and Suthar, 2018). Hence, the paper processing industry is contemplated as a prime user as well as the natural resources exploiter of materials namely, water, wood, and fossil fuels. The average water consumption in paper and pulp ranges from 5 -100m^3 per liter of paper synthesized based on the quality of parameters associated with the raw material utilized, the various types of paper processed or produced, and the capacity of water reuse (Chaudhary and Paliwal, 2018). And water is one of the most important sources for the survival of human civilization, its constant wastage is not going to lead anywhere (Dagar and Singh, 2021). Proper treatment and recycling could be the only best and most sustainable solution to the constant and ever-growing water demand by industries and to conserve this precious resource (Inamdar and Singh, 2008).

More than 250 chemicals have been characterized in the effluents generated at various stages in the making of paper making this sector the World's sixth most polluting industry for wastewater generation (Ugurlu et al., 2008, Kumar et al., 2018). Approximately 75%-95% of the water used is released as effluent in the natural water resources characterized by undesired features like high COD, BOD, pH, Dark brown color, odor, and other toxic pollutants that deteriorate the quality and properties of water (Medhi et al., 2011, Zayas et al., 2011). Around 50-55% of the initial raw material mass is released in the wastewater containing huge quantities of cellulose, hemicelluloses, tannins, resin acids, resins, Chlorophenols, lignin, and its derivatives produced by bleaching and washing of the pulp. Huge amounts of unsaturated fatty acids, furans, dioxins, diterpene alcohols, sulfur compounds, chlorinated organic compounds, *etc.* are also generated during paper making. The lignin and its derivative generated during the process of pulp formation using chemicals provide a dark brown color to the effluent which is persistent and recalcitrant in nature (Prasongsuk et al., 2009).

The effluent released from pulp and paper manufacturing industries have huge amounts of impurities that are suspended solids, turbidity, and color imparted by the lignin, which increases the BOD, COD, and also several other parameters that are beyond the permissible limits for CPCB guideline 2012. And, once this effluent is released, it leads to several long-term environmental issues like surface and groundwater pollution by contamination. (Dagar et al., 2022). In India, the paper and pulp industry are only relying on conventional primary and secondary treatments that are not enough to treat the toxic wastewater generated. Various cases have been studied based on the utilization of tertiary treatments for the purification of water like ozonation, adsorption, coagulation/precipitation, advanced oxidation, etc. However large-scale implementation of these techniques is still doubtful due to economic and infrastructure constraints.

According to the main rule of the IPPC directive constraining installation, holders need to utilize resources in a sustainable manner to reduce their environmental impacts. Because of the limited availability of water resources, industries particularly in water-deficit areas have the responsibility to contribute to water-saving activities. This problem is majorly applicable to the paper and pulp mills that should restrict the utilization of fresh water. However, the major environmental outcome of the paper processing industries is not merely the usage of huge quantities of fresh water but also the outflow of wastewater discharged from such industries, evolving from the various processes of paper production. In a case study, several distinguished treatment provisions have been experimented with to look for an exquisite solution to cut down the utilization of fresh water and also to reduce the quantity of effluents. There is a prerequisite requirement to find a suitable tertiary method of effluent treatment that can significantly decrease the pollutant concentration in water and enhance the water quality for adequate restoration of the water consumed in pulp and paper manufacturing industries.

2.2 Status of Paper and Pulp Processing Industries in India

India is amongst the 15th largest producers of paper/pulp worldwide with an average consumption of 9.3 Kg/Capita. It employs around 2 million people in its manufacturing units (http://www.careratings.com). According to the CPPRI Annual Report 2016, India has about 850 units contributing to the total paper and pulp production in India (http://papermart.in/2019/12/02/top-paper-companies-in-india-2019/). Out of the total paper production, 21% is produced from wood-based industries (Keswani, 2017). Trident, BILT, JK paper, TNPL IPC, etc. are the major paper and pulp-producing industries in India. Increased paper demand and limited forest-based raw materials pushed the GOI to encourage the settlement of small manufacturing units based on agro-residue fibrous raw materials. A large number of small industries having a capacity of 5-30t/day came into existence. These small-scale mills use non-conventional agro-residues and waste paper. Because of their small size, they operate without any chemical recovery systems and discharge black liquor as effluent that acts as a significant source of pollution of water discharged. The MoEF, under GoI, has classified the pulp and paper manufacturing industries as one of the top 20 highly polluting industries. The data for some studies conducted on the various parameters for wastewater generated from these industries are shown in Table 1. Apart from water pollution, air pollution and emission of greenhouse gases a big issues for paper and pulp industries (Gupta and Singh, 2015 Omid et al., 2015).

Table 2.1: Several characteristic parameters of wastewater effluent generated from pulp and paper processing industries

pH	Color (co-pt)	BOD (mg L^{-1})	COD (mg L^{-1})	Lignin (mg L^{-1})	Phenol (mg L^{-1})	Sulfate (mg L^{-1})	Total solids (mg L^{-1})	Refs.
6.4–7.3	660–1230	142–221	1170–1510	133–265	-	-	354–563	Eskelinen et al., 2010
7 ± 0.20	8942 ± 15.0	8296 ± 45	22,189 ± 39	1124 ± 12.09	364 ± 20.13	1089 ± 19.67	1799 ± 17.61	Chandra and Singh 2012
7.4 ± 0.1	1761 ± 2.3	185 ± 2.9	2420 ± 17	-	-	-	2359 ± 14.2	Garg et al., 2012
8.2 ± 1.0	2242 ± 56	385 ± 12	792 ± 70	436 ± 18	42 ± 2.5	993 ± 6	850 ± 30	Raj et al., 2014
8.5 ± 1.0	2538 ± 53.3	7250 ± 123.0	16,550 ± 507.2	800 ± 18.4	-	1003 ± 5.3	977 ± 7.2	Yadav and Chandra 2015
9.8 ± 1.3	7253 ± 64.2	2934 ± 38.3	6735 ± 45.3	1863 ± 52.6		852.4 ± 34.1	1753 ± 34.2	Rajwar et al., 2017
7.7 ± 0.02	1202 ± 6.53	180.54 ± 4.70	584 ± 3.62	-		-	1686 ± 10.58	Kumar et al., 2018

2.3 Production of Paper and Pulp

Cellulosic materials extracted from wood, rags, fiber crops, and waste paper, chemically or mechanically are the main paper industry raw materials. Heartwood, bamboo, sapwood, and eucalyptus are some of the most versatile species for wood extraction that are used in pulp production (Gupta and Gupta, 2019).

2.3.1 Mechanical Pulp

The pulp produced only using mechanical attrition without any chemical to produce lignocellulosic material is known as mechanical pulp. The softwood has a light color and is used as raw material. It gives a yield of more than 90%. The lignin available in the wood material is retained by the pulp. Due to this, the pulp produced has low strength as the lignin available in the pulp interferes with the hydrogen bonds between the fibers during paper production. The pulp produced by grinding of chips in between plates is referred to as RMP (Refiner Mechanical Pulp) whereas while refining if subsequent heating of the wood chips is carried out then along with grinding, it is known as thermo-mechanical pulp (TMP) (Bajpai, 2017).

2.3.1.1 Thermo-mechanical Pulp

Thermo-mechanical pulp is made up of processed wood chips that have been softened using heat treatment in a pre-steamed vessel and are the product of a two-step process that includes the removal of the bark and then chopping it into tiny chips (Rey et al., 2020).

2. 3.1.2 Chemi-thermo-mechanical Pulp (CTMP)

The CTMP operates by chemical pretreatment of the wood chips before refining to increase the brightness of the pulp. For this less aggressive chemical treatment is used in this process since the main purpose is just to make the fiber soft to be able to refine easily but not to remove the lignin. Chemi-thermo-mechanical pulps are pulps manufactured using these hybrid techniques (Gupta and Gupta, 2019).

2.3.2 Chemical Pulp

Chemical pulping of wood involves the process of boiling that extracts the cellulose by dissolving the bulk of the lignin binding the celluloses present in the wood. It leads to improved disintegration and further extraction of the cellulose fibers, resulting in high-quality papers (Rullifank et al., 2020).

2.3.2.1 The Kraft Process

The mechanism behind the kraft process is that wood chips are treated with white liquor, and a heated mixture of water, NaOH, and Na_2S is used for dissolving the connections of lignin, cellulose as well as hemicelluloses. This method is more cost-effective and suitable for nearly all known tree species, and it also improves the strength and brightness of the pulp. As the name "kraft" implies, the pulp produced by this process is much stronger than that produced by other pulping methods (Ainun et al., 2018).

2.3.2.2 Recycled Pulp

Since the recycled pulp is made from papers that have been printed with ink, it is also called deinked pulp. It has now also been used as a raw material in a variety of industries, including newsprint, toilet, and tissue paper (Boguniewicz-Zabłocka, 2020).

2.3.3 Washing and Screening of Pulp

This process of washing and screening uses a lot of chemicals and finally, the recovery of these chemicals is necessary because they interfere with downstream processes, and cause detrimental effects on the environment. Pulp washing equipment comes in a variety of forms. The majority of the mills use hot water to dispense dissolved materials in a pulp mat, although certain mills utilize pressing machines to drain out the unused chemicals with the stream of liquid. A spinning drum with a mesh cover of wire is revolved in the diluted suspension of the fibers, which is an old but still prevalent process. On the drum, the fibers form a mat, which is then sprayed with hot water in showers.

2.3.4 Bleaching

The blackness of the pulp is attributed to the chromophoric groups present; the bleaching successfully removes complete residual lignin content after pulping. In the current delignification process, both bleaching and pulping processes are used. However, the term "bleaching" has usually been reserved for delignification that occurs after the pulping process. The "bleaching" procedure is categorized into two main steps: oxygen delignification and the other one is ultimate bleaching (Boguniewicz-Zabłocka, 2020).

2.3.5 Oxygen Delignification: Delignification involves processing the chemical structure of lignin for better dissolution during paper production. This technique involves treating washed pulp with a strongly alkaline solution like NaOH. In lignin, when pH is on the higher side, it promotes phenolic group oxidation. The various steps carried out to bring about the ionization further followed by depolymerization of semi-degraded lignin to decrease its molecular weight. These LMW by-products show more water solubility due to which their extraction from the fibers becomes easy. The pulp needs to be washed before going for oxygen delignification since the black liquid solids consume excessive oxygen. Finally, oxygen is a relatively inexpensive bleaching agent offering significant operating cost and it also enhances the yield of pulp (Kaur et al., 2017). However, the oxygen delignification decreases the amount of AOX in the pulping process generating the effluent with lower biological/chemical oxygen demand and color.

2.3.6 Final Bleaching: It is a composite procedure which uses Cl_2, ClO_2, sodium hypochlorite, oxygen, H_2O_2, and O_3, among other commercial bleaching chemicals. By making efficient use of the chemicals, this method improves the pulp's strength. Since elemental chlorine (Cl_2) produces a lot of chlorinated organic molecules in the effluent; a lot of initiatives have been taken to reduce its use. As a result, modern bleach factories do not employ elemental chlorine. ECF plants stand for elemental chlorine-free bleach plants. Despite its size, when compared to Cl_2, ClO_2 is more effective in maintaining the strength of pulp but not efficient in delignification and bleaching (Antony et al. 2012).

2.3.7 Paper Making: The cellulosic fiber formed after consecutive compression and drying by both mechanical as well as chemical treatment of pulp fibers will result in suspension which after water removal results in the paper product. The fiber is usually mechanically treated by running it through refiners, which are revolving metal discs with moving steel bars attached to them. This therapy shortens the fiber and fibrillates it.

2.4. Impact of chemicals used in paper and pulp on the environment

The process of chemical pulping in the paper-making industry is the main source of air pollution since it requires very high temperatures. The main constituents of emissions are sulfur dioxide, particulate matter, various oxides of nitrogen, and other malodorous compounds (Zhao, 2020). Bleaching is an important process in paper-making industries and has a substantial impact on the industrial financial system. Bleaching provides brightness, cleanliness, and softness to the pulp as compared to the unbleached pulp. This is a very crucial step in manufacturing a variety of products like tissues, sanitary rolls adsorbent products, *etc*. Chemical bleaching of pulp is a combined subsequent process that is carried out to obtain high-quality brightness in the pulp. Compounds like resins, phenol, and lignin react with chlorine and its compounds and get transferred to extremely lethal pollutants. Hypochlorite and chlorine have been used for the bleaching process of chemical pulp for several years as these are cost-effective and generate excessively brighter pulp. Pollutants released during bleaching with chlorine and its derivatives are non-biodegradable and extremely toxic because of the chemical reactions taking place during the process. For many years various modifications have been carried out in the pulp bleaching industry to subsist with the environmental standards. Paper and pulp industries consume fresh water in high amounts and release huge amounts of polluted water as effluent. BOD, COD, AOX (Adsorbable Organic Halides), and lignin derivatives are in very high amounts in this wastewater and the release of adsorbable organic halides is the most critical issue as an effluent from the pulp plants. (Lehtimaa et al., 2010). The making process also generates a huge quantity of solid waste including bark, sodium salts, pulp screening rejects, and grit (Jiang et al., 2022)

Several research studies target to limit the generation of poisonous compounds and the volume of wastewater *by* making modifications in the process of bleaching in the pulp industry (McDonough, 1998, Tripathi et al., 2019). To reduce the number of contaminants in wastewater of bleaching plants, several encouraging perspectives like oxygen bleaching, use of peracids, extended pulping, ozone bleaching, elemental chlorine free (ECF), hydrogen reinforced extraction stage, and chlorine-free bleaching. The ECF ClO_2 for bleaching than the elemental and it also limits the use of dioxins and furans to lower than 1 PPT in the pulp (Tarkkanen et al., 2012, Patel et al., 2021). This process of bleaching brightens and strengthens the pulp (Bajpai, 2012). Discharge produced from ClO_2 bleaching consists of chloride components so it is not dispatched for the process of evaporation/recovery. It corrodes the recovery system and leads to unwanted air emissions during burning. Advancement in the bleaching process provides a better alternative to scale down the quantity of pollutants and effluents. To manage the limitations of the environment and fulfill the need for brighter and stronger pulp, there is a prerequisite to finding a suitable system that may reduce/replace the chlorine-based components during the bleaching process of pulp. The various environmental issues linked to the paper and pulp industry are shown in Figure 2.1.

2.5. Complexity of chemicals used in paper and pulp on the environment

The process of krafting is a governing process as it includes the system of recovery to recover the pulping chemicals. The quality of pulp produced by this process is more durable and darker in color so vigorous bleaching is required to obtain the desired product (Kumar et al., 2011).

In the 17th century, a chemical known as hypochlorite was used extensively for the bleaching of wood and pulp cloth. But later, in 1985 this was discovered to be the major source of emission of chloroform as reported by EPA. Early in 1900, chlorine came out as the leading step in the pulp bleaching sequence at the first stage as it was a cost-effective and effective bleaching agent. Chlorine and its components are known to be important bleaching chemicals while these are associated with organo-chlorine components and chloroform emergence because of the complication of pollutants from the bleach plant, with BOD, COD, and AOX quantities also estimated. According to an international perspective, AOX is not a new concept. AOX levels have been decreased by the developed countries while the elemental chlorine is still used by the developed countries for the process of pulp bleaching. AOX is one of the crucial factors in determining the toxicity of the bleaching pollutant (Chaudhary and Paliwal, 2018; Yin et al., 2022). The discharge limits of AOX are monitored by several government agencies in various countries because it is harmful and causes genetic disorders in both terrestrial and marine organisms (Sharma et al., 2014). Many organo-chlorine components were studied and their types depended on the amount of lignin remaining on the pulp and the chemical used for the bleaching. Several toxic components are produced during the traditional bleaching process consisting of certain carcinogenic agents, fatty acids, and resin acids as mentioned in Figure 2.2.

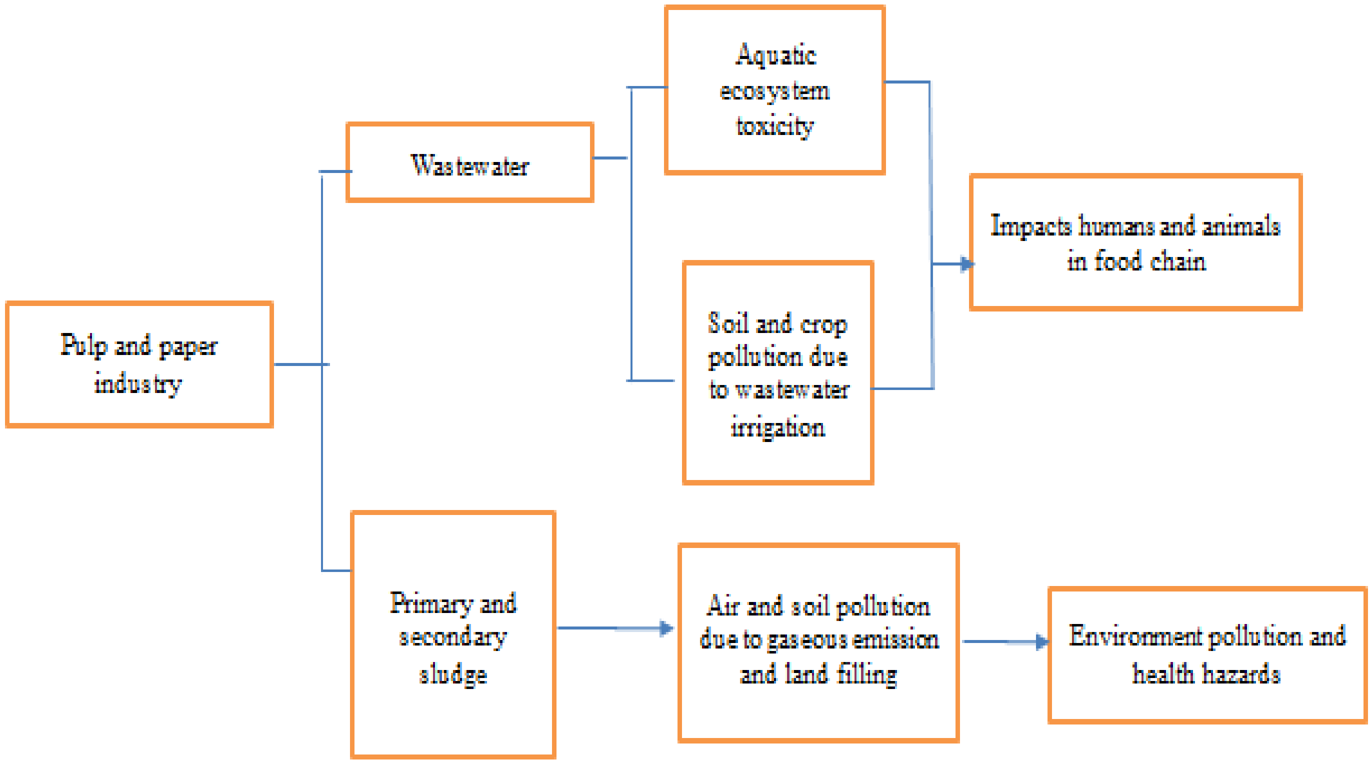


Figure 2.1: Pollution problems due to pulp and paper industry

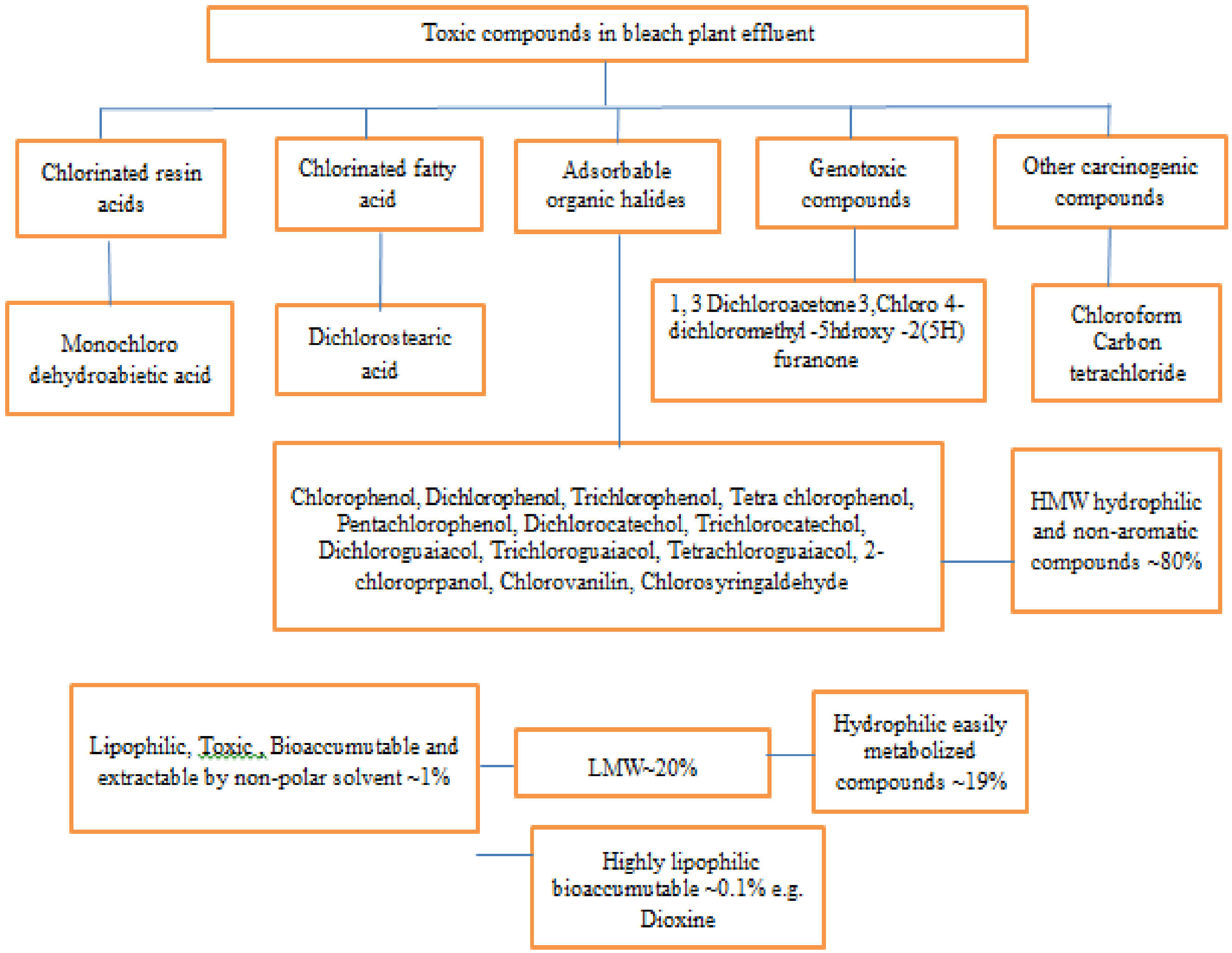


Figure 2.2: Noxious chlorophinotics in bleaching pollutants and their classification

Persistent organic pollutants are chloro-organics that tend to bioaccumulate and have the property to persist up to major toxic levels in pollutants (EPA, 1998, Dey et al., 2013). The toxicity of chlorophenolic compounds is defined by the Position and number of chlorines linked to the phenol related to the –OH group of benzene rings (Choudhary et al., 2013).

The proportion of HMW harmful compounds to LMW toxic chemicals used in bleaching is stated as 4:1 (Duarte et al., 2003). The toxicity of lower molecular weight compounds is comparatively greater as compared to high molecular weight compounds because HMWs are unable to infiltrate across the cell wall.

Previous studies reported the genotoxicity, mutagenicity as well as respiratory stress in marine animals when exposed to paper and pulp mill pollutants (Orrego et al., 2011). Chlorophenolics present in wastewater damage the metabolic system in fish (Hewitt et al., 2008, Nowake, 2006). Several studies have been developed in order to identify chlorophenolics present in the effluent produced from softwood and hardwood pulp (Berry and Luthe, 1992). The genotoxic effect has been confirmed by bioassay conducted on *Allium cepa* and *Tradescantia sp*. (Bhat et al., 2019). Various mutagens present in the effluent wastewater have been observed and recorded in the cells of zooplanktons found near the wastewater outlet site marking their entry into the food ecosystem (Stepanova et al., 2000). Moreover, the uncontrolled release of heavy metals like Cd and Cr from water mills has been observed in Assam, India (Borah et al., 2018). Irrigation with heavy metal-contaminated water leads to phytotoxicity and that leads to the incorporation of these heavy metals find to the food chain (Muchuweti et al., 2006). So, various waste-water effluent treatment methods such as the activated sludge method can treat the AOX coming as effluent but cannot reach the dismissal limits (Ashrafi et al., 2015).

2.6: Advancement in techniques of processing of pulp in Indian paper industry

The pulp and paper processing industry in India has improvised in a few previous years, yet several challenges are to be resolved. To match up with the increased paper demand, the Government of India motivated small-scale paper and pulp manufacturing units. However, because of the financial restraints, hypochlorite and elemental chlorine pulp bleaching is to date most versatile method to carry out bleaching in small-scale paper and pulp processing mills that produce poisonous components (Kaur et al., 2017b). Although there is constant growth in the Indian paper-making industries, a few of the mills have adopted the green bleaching technology. A few large-scale agro and wood pulp industries use the elemental chlorine bleaching process along with advanced ClO_2 to decrease the pollution load (Singh et al., 2019). Therefore, the utilization of ozone-type bleaching is confined to a single mill only. The use of bleaching techniques that can make pulp total and elemental chlorine-free partnered with advanced technologies is still under observation. Paper import and export markets might face the consequences if not able to cope with international standards in the future. Along with increasing environmentalism nowadays, there could be a ban on the use of products made using paper with chlorine-based bleaching as packaging material for food items. Hence, making it pivotal to embrace advanced techniques for the production of brighter, cleaner, and cost-effective paper. Various proficient techniques namely, oxygen bleaching, extended pulping, ECF (Elemental chlorine free) method, and peracids and ozone-based bleaching should be executed by the Indian pulp and paper manufacturing or processing industries. Application of such techniques will not only enhance the pulp quality but additionally reduce the poisonous components from the bleaching pollutants.

2.7: Techniques of wastewater treatment in paper and pulp industries

Considering the extensive waste generated by the paper processing industries, there is a requirement for immediate actions to manage the challenges of the future and secure the disposal of waste from these industries. The use of emerging techniques can make it convenient to use the waste generated in some other industries. Secondary utilization of the waste lowers the risks to the environment through the waste and also drives the industries to work efficiently.

Various treatments and technologies were also studied like reverse osmosis, ozonation, electrolysis, nanofiltration ultrafiltration, etc. (Figure 2.3). However, this problem persists as these technologies are not cost-effective and efficient commercially. Such monitoring led the paper and pulp industry to look for environment-favorable techniques for pulp production as these may engage with the non- chlorine-based bleaching techniques. Many alternatives are available for chlorine-based pulp leaching yet their implementation requires several process amendments with existing bleaching specifications.

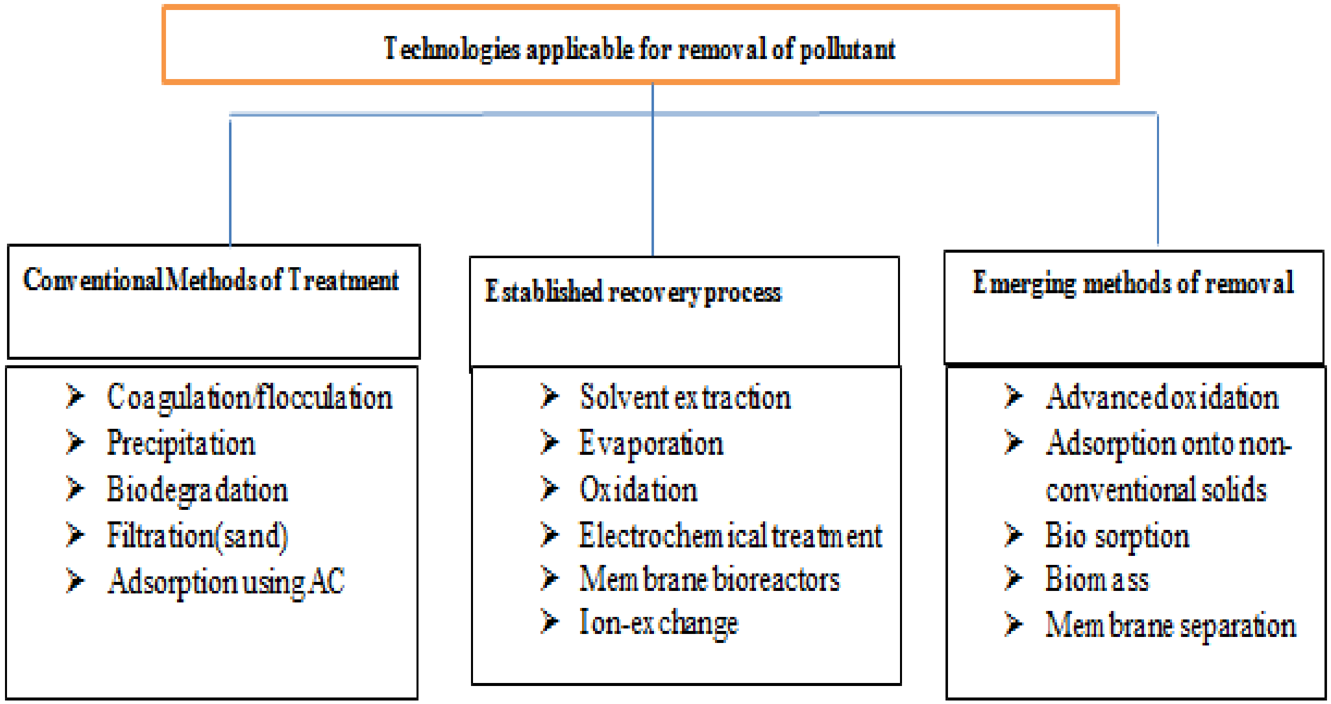


Figure 2.3: Classification of pollutant removal technologies available

2.7.1 Conventional methods of contaminant removal from effluent of Paper Processing Units

Paper and pulp industries consist of lignin components that are chlorinated, relatively synthetic, and aromatic in nature. One of the best ways to recover beneficial organic matter is by extracting lignin during pulp making. Since lignin has a complex structure; it becomes very difficult to break down as it gives technical and economic incentives to recover the biomass. The traditional processes to treat the wastewater are merely efficient to remove the lignin completely from the industrial effluents. Further, in order to remove lignin, certain biological processes engage the microorganisms like fungi and bacteria as a single-step procedure or a combination of several physio-chemical technologies. But, in comparison to the other methods, these biological processes are considered to be more sustainable, eco-friendly, and cost-effective for proper lignin depolymerization (Asif et al., 2019).

Lignocellulosic biomass is consumed worldwide to generate bioenergy and its upgraded commodities (Sagues et al., 2018; Nahak et al., 2022). It consists of entities of lignin, cellulose, and hemicellulose. After cellulose, lignin is one of the extensively found aromatic compounds on the Planet and is majorly present in the environment in vascular plants between their plasma membrane and the cell wall. Lignin is an unusual existing aromatic polymer that consists of several functional groups like phenylpropanoid, C-C bonds, and ether groups and has a 3-D molecular structure that is obstinate to biological/chemical deterioration (Ralph et al., 2019).

Earlier, lignin, the second most abundant natural polymer and the main residue of the pulp industry was recognized as a waste product but now recent developments have started to develop eco-friendly, biodegradable high-end products from it (Luo and Abu-Omar, 2017; Torres et al., 2020). The pulp industry alone generates nearly 50-70 million tons of lignin per year worldwide and the volume is predicted to increase to 4 times i.e. 225 million tons per year (Mandlekar et al., 2018; Torres et al., 2020).

Lignin is available in different conformities with different removal procedures. Generally, it is generated as a byproduct of paper manufacturing industries. The reactants present in the effluent of paper processing units are majorly organic and comprise lignin and its products, where lignin is the main constituent that is accountable for the color of paper in these industries. Hence, decolorization of this wastewater is an indicator of lignin removal. It has a large no. of implementations in food, chemicals, pharmaceuticals, biofuel, and several other industries. Elimination of lignin in paper manufacturing units by different methods is not aimed at such implementation purposes (Cotana et al., 2014). Lignin removal and its recovery techniques involve - (1) Physicochemical processes and, (2) Biological processes.

2.7.1.1 Physio-chemical techniques

These techniques are used for lignin removal from industrial wastewater and comprise filtration, ozonation, adsorption, coagulation/precipitation, advanced oxidation reverse osmosis, etc.

A) Coagulation/ Precipitation

Globally, Water industries consider coagulation as the major treatment units that combine smaller particles into larger aggregates (flocs). These techniques are a part of tertiary treatments in which metal salts/ chemical coagulants such as ferric sulfate, aluminum sulfate (alum), and ferric chloride are incorporated into the effluent streams to create greater flocks from the gathering of smaller particles (Jiang et al., 2022; Monika and Toczylowska-Maminska, 2020; Ang and Mohammad, 2020). Ghernaout, 2020 and Tetteh and Rathilal (2018) have recorded that coagulants like alum having high valence coagulants are often used as the concentration of metal ions reduces with high valence electrons. However, the optimum pH range is narrow (5.5-6.5). Wang et al. (2011) used the starch graft polyacrylamide as a flocculant and aluminum chloride as a coagulant for the treatment of effluent. An optimal condition was observed in his studies, where the rate of recovery of water and lignin was 72.7% and 83.4%, respectively, where the dosage of flocculants was 22.3 mg/L and dosage of coagulant was 871 mg/L at pH 8.35. Preliminary treatments of corn stover reduced the depletion of sugar and enhanced the removal of lignin with metal catalysts like Fe_2O_3, MgO, CuO, and NiO as reported by Li et al. (2018). Among these, MgO was observed as the best catalyst for the biomass preliminary treatments. This biomass can directly be transferred for the hydrolysis and fermentation processes. The combination of two stages i.e. a primary settling chamber and another one being coagulation with 1200 mg/L aluminum sulfate at a pH value of 6 resulted in approximately 98% turbidity removal, 93% COD reduction, 98% TSS removal, and 96% color rejection (Mehmood et al., 2019). Studies have also developed natural and green coagulants like Moringa olerifera, Nirmali seeds, chitosan, etc. and they have been reported to be effective in TSS, Turbidity, and COD reduction (Sandali et al., 2022).

However, the method is considered eco-friendly and efficient for the removal of TSS and turbidity only. However, this method produces large amounts of metal hydroxide toxic sludge, and disposal of this sludge is a great problem (Kurniawan et al., 2020). Moreover, this method also results in increased metal concentration in the treated effluent which causes complications for human health (Toczylowska-Maminska, 2020; Polizzi et al., 2023). The aluminum residues in the treated water can cause neurotoxicity and lead to Alzheimer's disease (Freitas et al., 2015 Freitas et al., 2018). Coagulation has low efficacy for fine particles and cold water along with sensitivity to pH change and this extreme pH range makes it difficult to reuse this water (Hubbe et al., 2016).

B) Electro-coagulation

Electro-coagulation is the application of electrical current to flocculate the contaminants without the addition of any coagulants (Butler et al., 2011). These days, there is high demand for electrochemical processes for the treatment of wastewater and degradation of pollutants from industrial wastes as these processes are more cost-effective and technologically possible (Bharath et al., 2018).

These processes include the soluble anodes positioned in the effluent followed by the production of flocs because of the dissemination of metal oxides from the anode. Aluminum electrodes have also proved to be highly efficient for lignin, phenol, and BOD recovery from the discharge of pulp processing mills (Ugurlu et al., 2008). Similarly, Katal et al., 2011 observed an 86% COD< 92% color and 96% phenol rejection from the paper mill at a current density set at 70mA/cm^2 at pH 5-7. This Al electrode was also used by Shankar et al. (2013) for the removal of lignin from synthetic wastewater in 75 min at pH- 7.6.

According to an experiment, the impact of several factors like voltage; pH, and electrolysis time was studied to reduce the lignin content, COD, and color in the effluent generated from these paper manufacturing units. Aluminum and iron were installed as Cathode and an Anode respectively. This study exhibited a successful implementation of this electro-coagulation technique in the wastewater treatment under optimal conditions *i.e*.; pH-5, 10V, operation time- 60 mins and current density 4.167 mA/cm^2, the lignin removal efficiency was 78.5%, for color removal, it was 100% and for total COD, it was 85%. This procedure visually cleared and decolorized the wastewater under optimal conditions. Removal of lignin, color, and COD are directly proportional to electrolysis time. Consumption of electrical energy and wastewater pH were 8.334 kWh/m3 and 8.2 respectively. Therefore, this process is successfully considered to be added to the pre-existing biological methods of wastewater treatment (Aghdam et al., 2016).

A case study conducted by Ebba et al., 2022, based on the effective removal of 92.30% color, 92.28% COD, and 83.33% turbidity from wastewater was achieved at the current density of 0.09A at pH 7.5 and electrolyte concentration of 3g/L with the reaction time of 1 hr. But the application of electro-coagulation requires regular replacement of the electrodes and the conductivity needs to be maintained making the technique high-cost (Jovanovic et al., 2021).

C) Adsorption

Adsorption techniques rely on mass transfer where the solute is transported onto the solid phase surface (Liu et al., 2020). An adsorbent provides binding sites for the pollutants. The commonly used adsorbents include silica, activated carbon, metal oxides, etc. (Prajapati and Mondal., 2019; Slatni et al., 2020; Prajapati et al., 2020). These days, the main aim of to use adsorption technique is for the efficient removal of color along with other organic compounds from waste. Nowadays magnetic adsorbents are very efficient for the removal of various life-threatening contaminants from effluent (Mehta et al., 2015). Pokhrel and Viraraghaum,2004 and Kamali and Khodaparast, 2015 have reported several adsorbents namely silica, fuller's earth, coal ash, and activated carbon. These adsorbents also exhibited sufficient lignin and color removal from the wastewater of pulp and paper manufacturing or processing industries. In the same type of case study carried out by Shawwa et al. in 2001, 90% of the parameters such as color and COD were removed from already bleached wastewater by the use of adsorption on activated coke. In a report, 80.4% color and 61% lignin removal were stated by Das and Patnaik (2000) via the process of adsorption from the blast furnace. Additionally, in tertiary degree treatments of adsorption and coagulation, a cheap absorbent *i.e.*, bentonite (4500 used with 400 mg/L polyaluminium silicate chloride was observed to remove color and lignin up to 41.38% and 60.87%, respectively **(**Duan et al., 2010**)**. Also, In another case study conducted by Mainardis et al. (2022), the PAC (Powdered Activated Charcoal) at 400 mg/L dosages is used as the adsorptive medium that leads to a reduction of the COD up to 50% from the paper and pulp mill effluent. The adsorption does not produce any toxic by-products, less energy-intensive (Kamaraj et al., 2020; Rashid et al., 2021). However it has certain disadvantages such as high investment cost and the cost of the adsorbents so cannot be used on a large scale (Al Sharabati et al., 2021).

2.7.1.2 Advanced Techniques for the Effluent Treatment of paper industries

To carry out the reduction and removal of the inorganic and organic wastes from the effluent released by paper and pulp processing industries as wastewater, several technologies have been developed. Emerging techniques of oxidation have been used to treat wastewater by various industries as well as solid wastes from the municipality (Cesaro et al., 2019 Alessandra et al., 2019). Several biological techniques and ozonation processes have a remarkable effect on the effluent treatment (Bajpai, 2017). Purkarova et al. in 2018 reported that 24% carbon efficiency was achieved using black liquor gasification signifying the clogging of apparatus by lignin along with the generation of hydrogen sulfide that is toxic for humans. Polymeric ferric aluminum sulfate chloride was composed of a steel mill waste that was further used along with polyacrylamide to form a composite coagulant and decreased COD (63%) and chroma (71.2%). This plan of action also worked at the industrial level (Yang et al., 2019). There is a process called aerobic granulation that has comparatively smaller biological footprints, finer shock resistance, and superior toxicity tolerance which proves to be efficient in treating wastewater with higher levels of COD and BOD (Morais et al., 2016). This technique eliminates lignin and tannins from the wastewater (Vashi et al., 2019). Microbial fuel cell is a novel technique made up of bio-electro-chemical devices that generate electricity and also treat wastewater by the use of the electrons generated during the anaerobic oxidation of the substrate present in wastewater (Pant et al., 2010). Recently, Membrane biofuel systems have been used in seawater desalination, wastewater treatment, hydrogen production, etc. (Rabaey and Rozendal, 2010; Ucar et al., 2017). Several benefits are enlisted from this microbial fuel cell like producing electrical energy, prohibition of the ventilation method, and certain centralizing or decentralizing uses (Neto et al., 2018). Treatment of effluent discharge from the paper and pulp processing industries by the use of biosorbents is an advanced technology to eliminate chromophoric components. Residues from agricultural-related industries prepare certain absorbents that provide a convenient and cost-effective process for the treatment (Kakkar et al., 2018). The emerging process of oxidation is also an assuring method for wastewater treatment efficiently. Along with the assistance of response surface technology, escalate the Fenton process to facilitate a decrease in COD to 1920 mg/L from 3200 mg/L (Kumar et al., 2019). 93% of COD can be removed by using this process of advanced oxidation. Electrocoagulation is another competent technique for the treatment of waste. It removes ammonia by 85.3%, color by 98.5%, TSS by 83.4%, and COD by 79.5% (Ali et al., 2018).

A) Primary and secondary sludge treatment techniques

Sludge from the paper and pulp industries adversely affects the environment because of its inorganic and organic properties. A report was generated by Mohammadi and his team members in 2019 in Sweden in which anaerobic digestion of sludge was used by the paper manufacturing industry (Mohammadi et al., 2019). For the purpose of comparison aerobic digestion was performed with incineration individually and then in combination with the pyrolysis *i.e.*, incineration + pyrolysis. Biochar was produced during the later system which is advantageous for the development of a sustainable ecosystem and for forest productivity in developed countries. Lignin can also be removed from sludge with the help of anaerobic digestion so it is available for deterioration and can be used for the generation of many useful products (Khan and Ahring, 2019). Production of Bio-gas was also reported by using anaerobic digestion where production at a very large scale is yet to be witnessed (Lopes et al., 2018). The use of enzymes in bio-bleaching is another option to reduce the pollutants in the paper and pulp industries. Enzymes like Laccase and Xylanase are extensively used for agricultural-based or wood pulp bio-bleaching. It enhances the quality of the products as well as reduces the consumption of chlorine for more strength and brightness (Kumar et al., 2019).

B) Oxidation procedures for wastewater treatment

The pollutants from paper and pulp industries are potentially toxic as these are produced in large quantities and are obstinate in nature. Several biological procedures are unable to eliminate such components. AOP known as the Advanced Oxidation process is recognized as the ability to harness the OH radicals reactivity. The advanced oxidation process generates comprehensive mineralization that modifies recalcitrant compounds to inorganic substances *i.e.*, H_2O_2 and CO_2, and fragmented mineralization (modifies them to additional biodegradable compounds) (Miral et al., 2021). The application of advanced technologies alone cannot efficiently achieve sustainable development, computational analysis and the use of artificial intelligence are also required in order to sort out the best integration technique for the reduction and removal of waste from our environment. These computational tools would facilitate the reduction of waste production and will make the process more energy efficient by the application of an appropriate model at each processing level. Several sophisticated and advanced models are required to be incorporated in this industry for further development. Lower sensitivity and higher reactivity of such radicals prove such processes as promising techniques. Because of the distinguished pulping procedures, pollutants from the several operations and processes also differ from one another, hence different oxidation procedures can be incorporated together to improve the removal efficacy. For efficacious oxidation of obstinate organic components hydroxyl radicals shall be produced constantly in situ and because of its chemically unstable nature, the production of hydroxyl radicals is generally sped up by the combination of oxidizing agents. From these processes, photo-Fenton with formula $UV/H_2O_2/Fe_{+2}$, Fenton's reagent written as H_2O_2/ Fe_{+2}, Ultraviolet radiations with H_2O_2 (UV/H_2O_2) and O_3 in different combinations (O_3/H_2O_2 and O_3/UV) are regarded as effective in paper manufacturing units for the oxidation of pollutants. The semi-chemical and Chemi-mechanical cellulosic pulping gives a higher yield of pulp and there is no reactive recovery system depending upon the combustion of organic material as occurs in pulp manufacturing procedures like chemical kraft procedure, or if the pulp is bleached along with the chlorine components, the effluents might have organo-chlorine compounds. Effluents present in industrial waste are extremely colored because of the chromophoric components from lignin derivatives, wood extracts, and organochlorine components that are all recalcitrant. The recalcitrant components are called persistent and build up in the environment. The aversion to disintegration might be because these cannot be accepted as a substrate by the living organisms in biologically active procedures. These are higher in stability and have chemical inertness because of their replacement with sulphonate, nitro, and halogen groups. These are insoluble and are highly toxic in nature. Compiled characteristics prove that these biological procedures are not enough to remove such compounds, and so technologically unconventional advanced systems have been developed for their treatment. It is evident that AOX procedures are technologically appropriate to remove the recalcitrant components from the waste of pulp and paper processing or manufacturing industries (Laura et al., 2014).

Advanced oxidation technology and electrochemical methods such as photo-Fenton and ozonation allow up to 100% COD removal (Hermosilla et al., 2015). But these methods consume a huge energy input reaching up to 96kW/Kg of COD removal along with generating secondary pollutants making it the biggest constraint for their use (Tunay et al., 2010; Lin et al., 2012).

C) Effect of ozonation for wastewater treatment

Advanced Oxidation Processes (AOPs) are those oxidation processes that involve OH radical and Sulfate Radicals ($\cdot SO_4^-$) generation that are applicable for the elimination of contaminants containing both organic and inorganic compounds from water effluent (Deng and Zhao, 2015). The use of AOPs (Advanced Oxidation Processes) to decimate the slow-degrading components is widespread. Hydroxyl radicals are significant oxidizing agents which have a potential of about 2.33V and have a higher reaction rate than accustomed oxidants like H_2O_2 and potassium permanganate (Wang et al., 2004). Hydroxyl radicals react with many organic and inorganic substrates with higher rates constant (Hoigne, 1997).

Since Ozone alone cannot completely oxidize the organic compounds and also the rate of reaction is low. So, Ozone combined AOP is a good option to treat effluents of several major recalcitrant pollutants. The ozone in combination with H_2O_2, ultrasound, and UV light increases hydroxyl radical generation efficiency (Rekhate and Srivastava, 2020). A significant drawback of ozonation is the generation of ozone at a high cost partnered with a short half-life. So, it is mandatory to produce the ozone at the site. Further, the efficacy of the process depends majorly on the efficacy of gas-liquid mass transfer which is relatively complex to obtain because of its low solubility in the liquefied solutions (Baban et al., 2003). A study identified numerous components in wastewater from paper and pulp industries. Evaluation of decolorization and efficacy of organic removal by traditional bubble reactors and biodegradability at different levels in ozonation is done (Kornmuller and Wiesmann, 2003 Soloman et al., 2009). The partial MS/GC analysis was carried out before and after the process of coronation and other biological procedures.

Aliphatic components and lignin-derived components were found in the processes of production of paper. Measurement of treatment efficacy was also determined by estimating TOC removal and decolorization. This study indicated that colorless effluents were yielded by ozonation and there was 90% decolorization after 45 min and had a comparable ozone competency rate of 20.0 mg O_3L^{-1}. The COD/BOD in the wastewater ratio scaled up from 0.10 to 0.32 with an ozone rate of 4.0 L'Min-1

(Kreetachat et al., 2007). Similarly, several kinetic-based models were developed for several ozone-based treatments that can considerably predict the rate of color removal and COD. These models can be used for process and reactor design for treating pulp processing wastewater by using ozone-based processes (Ko et al., 2009 Hamouda et al., 2009). Ozonation was found effective for removing the COD by 60% from process water, and 28% from bleaching water (Mainardis et al., 2020). The reported suggests that the process of ozonation enhanced the COD removal efficiency of traditional treatment plants from 46% to 81% when applied after tertiary treatment.

D) Membrane Technology

Membranes are a barrier separating two phases by movement restriction in a selective way (Ravanchi et al., 2009). The separation takes place because of variations in physio-chemical properties defined in the form of the geometry of the substance. The major classifications of membranes are isotropic membranes and anisotropic membranes depending on the symmetry (Sagle et al., 2004). The isotropic membrane has a uniform composition whereas non- uniform nature of anisotropic membranes possesses different structural compositions in various layers of the membrane (Ezugbe and Rathilal, 2020). The membranes can be made by either synthetic polymers namely Polytetrafluoroethylene (PTFE), Polyethylene Terephthalate, Polyethylene (PE), etc., or synthetic organic polymers like ceramics, zeolites, silica, etc. having numerous microscopic pores allowing only fine size particles and solvent to move (Aliyu et al., 2018). The movement of media/particles is based on the driving forces and hence the membranes are classified into four types namely equilibrium-based, non-equilibrium-based, pressure-driven, and non-pressure-driven (Jhaveri and Murthy, 2016).

Membrane separation constant process composed of mainly three effluent streams: a). Feed- the water stream entering the Membrane, b). Permeate- the water that crosses through the membrane (Product) and c). Retentate/concentrate- the water retained by the membrane (Singh, 2015).

The separation efficiency of this process depends on the selectivity of the membrane, flux, stability of the membrane materials, operating conditions, and the fouling of the membrane (Singh, 2015; Li et al., 2023).

The filtration technology of membranes driven by pressure such as Ultrafiltration (UF), Microfiltration (MF), Nanofiltration (NF), and Reverse Osmosis (RO) is widely used for the treatment of waste and brackish water (Erkanli et al., 2017). Micro and Ultrafiltration are usually applied to remove the suspended as well as colloidal particles through the mechanisms based on membrane pores size whereas the NF and RO are chosen for the elimination of dissolved contaminants like desalination or softening of hard water. The primary characteristics associated with various membrane technology-based filtration techniques are outlined in Table 2.4 below(Jose et al., 2011).

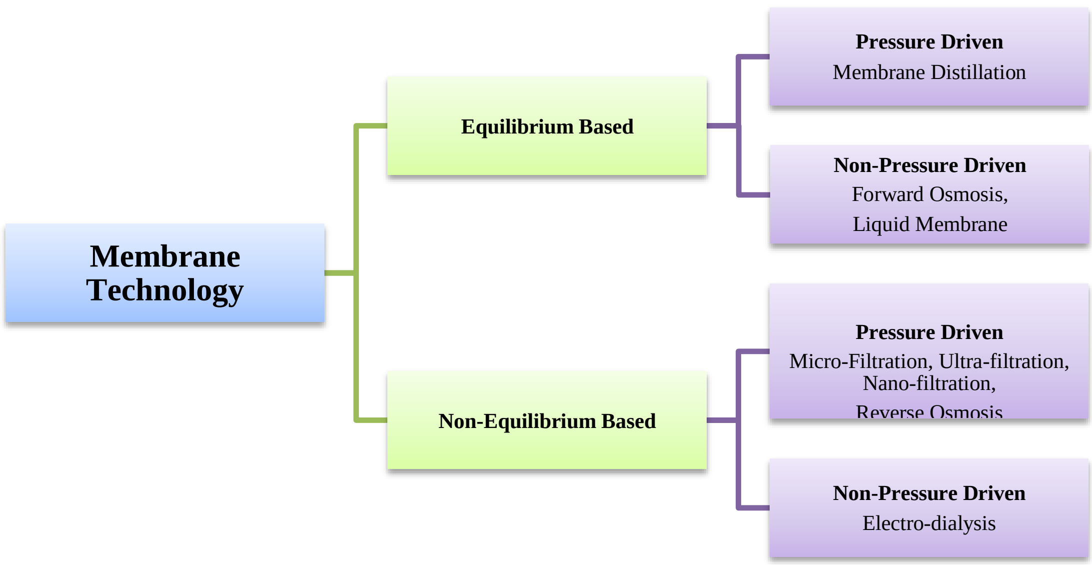


Figure 2.4: General classification of membranes

Table 2.2: Features of membranes utilized for wastewater treatment

Process	**Nominal pore size**	**Driving force**	**Average Permeability ($L/m^2.h$ bar)**	**Membrane Types**	**Retained solutes**
Micro	0.05-10µm	1-3 bar	500	Porous Both asymmetric and symmetric	Oil, fat, Grease, Micro-organisms, and micro-particles
Ultra	0.001-0.05µm	2-5 bar	150	Micro-porous and asymmetric only	Proteins, pigments, oils, sugar, organics, microplastics
Nano	≤2.0 nm	5-15 bar	<20	Tight porous and asymmetric thin film composite (thin film)	Pigments, sulfates, divalent, cations, divalent anions, lactose, sucrose. NaCl
Reverse Osmosis	~0.5 nm	15-75 bar	<5	Semi porous Asymmetric Composites (thin film)	All contaminants including monovalent ions

Source: Singh and Hankins, 2016

Micro-filtration (MF) is a technique that uses a pressure-accelerated membrane with the largest pore size (0.1-1.0μm), and ≥100K Daltons of average molecular weight cut-off (MWCO). The mechanism for the separation is physical sieving under a pressure gradient of 1-3 bars and is used mainly for large colloids, macromolecules, suspended particles, and microorganisms like bacteria (Ray et al., 2020). It hardly has any capacity to eliminate the biological and organic matter from the effluent, which can be used as a pre-treatment before RO/UF/NF (Torki et al., 2017).

With the 0.005-10μm removal range, ultra-Filtration lies between the range of micro-osmosis and reverse osmosis (Qu et al., 2014). It is prominently used in water filters as it removes all microbiological species, macromolecules along with humic materials such as lignin (Kruger et al., 2016). It has MWCO of ~10-100K Daltons. Ultrafiltration has a constraint in the removal of inorganic substances dissolved in the wastewater (Zhang et al., 2016).

Nano-filtration has the smallest pore size which leads to higher operating pressure and hence requires higher energy inputs. It removes all metal ions such as Fe, and Mg and is effective for heavily polluted water (Wagner 2001). It can easily eliminate sulfate ions from the wastewater thus helping in the removal of alkalinity and hardness of water. But the soluble elements cannot be separated using UF (Wang et al., 2016). In a case study analyzed by Xu et al. in 2016, it was found that UF is an effective method for the removal of heavy metals and dyes from textile wastewater.

Reverse osmosis is a pressure-driven process that removes nearly all the dissolved inorganic compounds, smaller particles, and ions from the water. RO is a technique that only allows water molecules to permeate and consistent hydrostatic pressure needs to be applied to overcome the osmotic pressure due to intrinsic factors. The pre-size of the RO is much smaller and tighter than UF. It can remove particles with a size smaller than 0.1nm and is thus used for the process of desalination of brackish water (Sagel and Freeman, 2004; Yan et al., 2016) and removal of pathogenic bacteria and biological compounds (Wood et al., 2017 and Komelsi et al., 2015).

This technique of membrane is one of the traditional processes used for the recovery of lignin in 1988. For the last many years, it has been observed that such membrane technologies are used at a large level to treat the paper and pulp-making industry's effluent. Further, the efficiency of the filtration process using membrane technology can be enhanced by incorporating several advanced pretreatment procedures.

In a study conducted by Chanworrawoot and Hunson, (2012), large-scale removal of quality parameters such as BOD, COD, TDS, and color from the industrial effluent post-treatment into a membrane electro-chemical bioreactor has been observed. The use of various ultrafiltration membranes was also reported by Gönder et al. (2012) to treat industrial wastewater which estimated sulfate as 97%, total hardness as 83%, spectral absorption coefficient as 95%, and COD as 89% at a pH of 10.

The membrane technology is known to be the best innovation as a lignin recovery solution. Lignin concentration is increased from 62 g/L^{-1} to 285 g/L^{-1} by the membrane technology. Moreover, the lignin recovered is in its purest form. This technique is adaptable and also scalable for numerous waste liquors of the paper and pulp industry. Cost-effective models state that this membrane-based recovery produces the purest lignin at the price of € 46-120 /ton, whereas the incorporation of diafiltration will make it costly (Tomani, 2010). Hence, this procedure of membrane-based filtration grasps a commitment to a highly economical and sustainable method for purest and concentrated lignin recovery including an approach to enhance the longevity and performance of the membrane. The ligno-boost technique may give a tough competition to this membrane-based filtration but later it was found that ligno-boost is only suitable for the black liquor and is more complicated to extend. Thus, this membrane filtration provides a distinctive combination of scalability, economy, and versatility to recover lignin from varied industries (Humpert et al., 2016).

Gholami et al. 2017 investigated the effluent treatments in combination with ultrafiltration membranes and AOP (Advanced Oxidation Processes). They reported the effects of 400mg/L caCl2, temperature -45ºC, and pH of 10 on membrane fouling by using RSM *i.e.*, response surface methodology. According to their study, the analysis showed an average thick layer on the ultrafiltration membrane that was decreased to 11.38uM from 75.37uM under SEM (Scanning electron microscope) after maintaining the optimum condition. This study introduced a hybrid approach *i.e.*, AOPs and filtration to treat wastewater from paper and pulp industries. Their study exhibited the usage of filtration procedures as a primary treatment of AOP that can be helpful to treat of AOP that can be helpful to treat wastewater that contains suspended solids and colloids at higher concentrations leading to invading few pollutions load to AOP so they may justify it both economically and technically. Therefore, according to their findings, the efficiency of removal was increased abruptly during the process of Filtration / S_2O_8 /Fe (II). Hence, because of its simplicity, easier installation, lesser space occupied by lower operational and economic costs, and lesser use of chemicals, it is a desirable procedure for treating wastewater from the Pulp industries (Gholami et al., 2017). An integrated approach of biodegradation followed by microfiltration integrating it with an economical ceramic-made membrane has achieved a COD removal of 87.60% and toxicity reduction of 94.50% (Goswami et al., 2019).

i) Ultra-filtration

This is the most recent technique for many municipal drinking water functionalities, though earlier it was generally used in several industrial applications like pharmaceutical or food industries. Ultrafiltration proved to be a tough competition to conventional wastewater treatments. In certain cases, a combination of conventional procedures with ultrafiltration is observed specifically for feed water with high fouling tendency and also eliminates certain particular contaminants. Nowadays, this technique is majorly used as a primary treatment for the reverse osmosis process known as IMS i.e., Integrated Membrane System. IMS is a very crucial and mandatory process to be implemented as a conventional primary treatment where the quality of water is raw or with very wide fluctuations assumed. This design of ultrafiltration is not considered for conventional filtration commercially because of its costly membrane. But, nowadays, its price has been reduced less than the conventional treatment and, therefore, ultrafiltration has become a competitive primary treatment system with reverse osmosis in a varied range of water quality (Wenten, 2008).

Bhattacharjee et al. 2007 have achieved a Total solid removal of 87% with ultrafiltration from Kraft black liquor which was 27% more than the conventional sedimentation method coupled with adsorption. Neves and his team members experimented in 2016 to analyze the applications of ultra-filtration membranes and micro-filtration as a substitute for wastewater post-treatment from paper and pulp industries. Several filtration tests were carried out in the laboratories. Optimum conditions like pressure, rate of flow, and interval of backwash were considered and maintained for each membrane during the experimentation. Various parameters were assessed like COD, ABS 254, turbidity, total solids, color removal efficacy, performance flux tannin/lignin, *etc.* concerning the performance of ultrafiltration and microfiltration membranes. These membranes were then chemically cleaned using a hypochlorite solution. These results state that the membranes improved the quality of wastewater and cleaning of membranes with chemicals was very effective (Neves et al., 2017). The use of Ultra-filtration increased the lignin removal from 62 g/l to 285 g/L in an experiment conducted by Humpert et al., 2016. A fixed bed membrane bioreactor developed by Izadi et al., 2019 reduced the COD up to 99%, Ammonia up to 97%, and Nitrite up to 97%. However, the efficiency of removal varied to 99% for COD removal, 83% for ammonia removal, and 90% for Nitrite removal with a hybrid airlift membrane bioreactor (Izadi et al., 2020). Sonaware and Murthy (2022), Observed that the addition of synthesized Zinc imidazolate framework-8 (ZIF-8) onto a Polyvinylidene fluoride membrane increased the removal of around 93.93% COD, 93.57% BOD, and 99.30% TSS in permeate. The nanoparticles also decreased the membrane fouling.

ii) Nano-filtration

Anthropogenic activities in all fields like food, medical industry, aquaculture, and clean water are scarce. As there is only 3 percent of freshwater available on the earth that needs advanced techniques to treat wastewater to generate harmless output for the environment and further can be used for several plans (Mulyanti and Susanto, 2017).

Membrane technology provides lower requirements of energy, environmental stability, higher selectivity, and less cost which when combined with other separation techniques makes it easier to scale up without the addition of any chemicals (Mulder, 1996). Existing problems can be solved by using membrane-based techniques to treat wastewater from paper and pulp industries. These techniques are generally pressure-driven and are dependent upon the size of their pores, and various membranes such as UF/MF/NF/RO.

As stated by Bunai et al. in 2013, rapid development has been observed in the application of Nanofiltration membranes in the last few years. Its use was majorly seen in the 1980s (Mohammad et al., 2015)**.** Its pores size is generally 1-5 nm and its operating pressure is 7-30 (Shon et al., 2013) in order to isolate solutes of lower molecular weights like glucose, salts, and lactose and it is influenced to remove hardness, heavy metals, and dyes. Hence, Nano filtration is a pressure-driven technology that assures wastewater treatment in the future, which may end the consumption of freshwater (Bunani et al., 2013; Gómez et al., 2007) and reuse of groundwater that cannot be done by using conventional methods of waste treatments. Lower operating pressure and high permeate flux permit the Nano-filtration to substitute RO (Reverse Osmosis) in several applications. Consumption of low energy in comparison to reverse osmosis and high rejection as compared to UF (Ultra-filtration) compel NF (Nano-filtration) to obtain more recognition for the treatment of wastewater. The various studies showing the removal potential of different membrane techniques is shown in Table 3:

Table 2.3: The effectiveness of using different membrane processes on wastewater

Membrane process used	Effluent treated	Results	Reference
UF	Vegetable Oil	COD >91% TOC >87% TSS =100%	Mohammadi and Esmaeelifar, 2004
UF	Wastewater from Poultry Slaughterhouse	COD >94%, BOD >94%, Fats ~99% TSS ~98%	Yordanov, 2010
UF	Greywater	Turbidity >94.7 TSS ~100%	Oh et al., 2016

UF	Municipal wastewater after primary clarifier	COD >28%	Ravazzini et al., 2005 abd Dittrich et al., 1996)
UF	Synthetic Dye wastewater	98% rejection of dyes	Lin et al., 2016
MF	Wastewater from Synthetically emulsified oil	Organic contaminants removal >95%	Wang et al., 2009 and Putatunda et al., 2019
MF	Greywater	COD >45% Color >73%	Kim et al., 2007b
MF	Oily Wastewater	Organic Additives ~90%	Chang et al., 2014
MF	Domestic Sewage	Nitrogen~97% Phosphorous~97%	Zuo et al., 2018
MF	Municipal wastewater	COD >76.4% TP >99.5%	Zhoa et al., 2019
NF	Textile Industry	COD>57% Color >100% Salinity >30%	Ellouze et al., 2012
NF	Grey water	Below detection limit	Guilbaud et al., 2010
NF	Industrial and synthetic dye wastewater	Color removal >99% Cl^- rejection >15%	Koyuncu, 2003)
NF	Synthetic and real fermentation broth	Glycerol ~5.2-6% Citric acid >45% Succinic acid >80%	Wozniak and Prochaska, 2014
NF-RO	Leachate from Dumpsite	95% water recovery	Rautenbach et al., 2000
MF-RO	Urban wastewater	Pharmaceuticals and pesticides and removal to the level of discharge	Rodriguez et al., 2015 and Mendret et al., 2019
UF-RO	Metal finishing industry	~90-99% contaminants rejection	Petrinic et al., 2015
UF-RO	Oily wastewater	Oil~100% Grease~100% Turbidity ~100% TOC=98% COD= 98% TDS =95%	Salahi et al., 2011
UF-NF/RO	Effluent from Pulp mill	Phenol =95% COD =95%	Sun et al., 2015
NF	Dye Wastewater	Dye~ 99.9%	Mi et al., 2020
UF	Dye Wastewater	Dye ~ 95%	Yang et al., 2021

iii) Problem associated with the use of membrane Filtration

The deposition of contaminants like suspended solids, organic material, etc from the input water surface or within the membrane stream which thereby leads to flux decline is known as fouling (Speth et al., 1998). The deposition of foulants reduces the membrane's active surface area and hence decreases the efficiency and membrane performance. The more the fouling, the higher the pressure, and hence energy is required for proper functioning (Kucera, 2015) Membrane-based filtration has obtained much recognition in the forest industry to full-scale operation from the lab scale levels. The fouling of this membrane consists of a remarkable barrier for their wide applications. Hence, to ensure an economical system of membrane filtration process, an improvised explanation of membrane fouling and initiation of a control plan of action is required. This decreases the efficiency, and so enhances the maintenance and operational costs. Though a lot of studies have been reported regarding this but yet this study is a topic of research in paper and pulp industries as fouling of these membranes is yet not understood completely. Such studies have targeted several factors like fouling mechanisms (Puro et al., 2011), fouling-characterized primary treatments (Koivula et al., 2011); and fouling prevention and cleaning regimes (Maartens et al., 2002; Li et al., 2002 and Weis et al., 2003). Therefore, it is important to discuss the recent advancements and innovations in the management of this membrane fouling. Membrane fouling is linked with several parameters which are further divided into 3 categories: - (1) process conditions *i.e.*, temp., pH, VRF (volume reduction factor), and hydrodynamic conditions; (2) treated material *i.e*. (molecule shape, concentration, and hydrophobicity); (3) membrane characteristics *i.e*. (pore, size, hydrophobicity, surface chemistry, morphology, and zeta potential). Hence, it is considered that membrane surface chemistry and membrane solute interaction are responsible for understanding the criteria of fouling (Kale and Singh, 2016).

Therefore, this can be concluded that primary treatments not only showcase a decrease in membrane fouling but also improve membrane filterability chemical cleaning also plays a crucial role in maintaining membrane permeability while implementing various cleaning regimes. Moreover, hydrodynamic, and operational conditions optimize control of the attainment of higher filtration capacity and low membrane fouling (Bokhary et al., 2017 Bokhary et al., 2018). The UF membrane permeability was restored using a two-step cleaning process that utilizes a consortium of six polysaccharides and extractive degrading enzymes as a cleaning agent and then subsequently followed by an alkaline cleaning agent (Rudolph et al., 2018). Poojamnong et al., (2020) analyzed the foulants using the fluorescence regional integration (FRI) and found that protein-like structures including tyrosine and tryptophan-like humic substances are the main fouling agents in the eucalyptus plant pulp-paper mill outflow water. Backwashing with double Ionized water followed by NaOH solution reduces the fouling to 54.60% and 76.60%. Finally soaking the membrane with NaOCl solution reduces the fouling to 97.80%. Application of Electro-coagulation for foulant removal can be applied in pretreatment. This not only reduced the fouling of membranes but also improved the membrane flux by 31% by removing the organic pollutants containing benzoic rings and carboxylic groups from the pulp-paper mill discharge for low-pressure RO membrane treatment (Gong et al., 2022).

2.7.1.3: Biological removal of contaminants from the effluent of the pulp and Paper manufacturing industry

Effluent wastewater treatment biologically is yet another option to remove color and lignin from the pulp and paper manufacturing industry's effluent and lignin degradation is dependent upon several micro-organisms (Abhishek et al., 2017; Ghosh and Thakur, 2017; Yadav et al., 2022; Zhao et al., 2022). It seems to be a more economical and environmentally sound technique than physical and chemical methods. Lignin generated from such units' color incorporation in the effluent and its presence in the waste can pose a great environmental threat (Haq et al., 2017; Zainith et al., 2019; Li et al., 2022). The degradation or decolorization occurs by two processes *i.e*. (1) biosorption (Brown and Chang, 2011; Abhishek, et al., 2017); (2) activity of microorganisms and enzymes. In these processes, the remains of a microbial cell play an important role in lignin discoloration, and Basidiomycota and Ascomycotin, white-rot fungi are majorly related to lignin breakdown (Haq et al., 2016).

Wood-decaying fungi have the potential for lignin degradation faster as compared to other microorganisms present in the environment. Cellulose and Hemicellulose can also be used by these microorganisms except lignin as the source of carbon for their potential growth (Blanchette, 1995). Asina et al. in 2016 concluded that white-rot fungi are lignolytic and so it degrades lignin for their growth and metabolism. It degrades lignin by selective or non-selective process of delignification. In the selective process, lignin degraded but cellulose remained intact while in the non-selective process, every component of the plant cell degraded (Blanchette, 1995). Table 2.4 shows the various potential microorganisms reported to treat paper and pulp manufacturing industry discharge.

Several microbes like Rhodococci, Acinetobacter, Pseudomonads, Sphingomonas, *etc*. can valorize lignin (Beckham et al., 2016). Hence this process of lignin degradation is not well known to date. An anaerobic degradation of native lignin with lower molecular weight is reported and interpreted. Further, genetic engineering can be adopted for the modification of microbial strains and metabolic pathways, to obtain more lignin yield and the value-added products of lignin degradation by increasing fungal growth (Brown and Michelle, 2014).

Table 2.4: Biological treatment of Pulp and paper mill Wastewater

Microbial species	Pollution parameters	References
Bacterial Species		
Aeromonasformicans	**COD>70%** **Lignin>80%** **Color>90%**	**Gupta et al., 2011**
Pseudomonas fluorescens	**COD>79%** **Lignin>50%** **Color>75%** **Phenol>70%**	**Gupta et al., 2011**
Paenibacillus sp., Aneurinibacillusaneurinilyticus, and Bacillus sp.	**COD>52%** **BOD_5>65%** **Lignin>28%** **Color>39%** **Phenol>64%**	**Chauhan and Thakur 2002**

Serratiamarcescens, Citrobacter sp., and Klebsiella Pneumonia	**COD>80%** **BOD_5>70%** **Color~80%**	**Raj et al., 2007**
BrevibacillusparabrevisMTCC 12,105	**COD>60%** **BOD>51%** **Lignin>42%**	**Chandra et al., 2011**
Planococcus sp. TRC1	**COD>85%** **Lignin>74%** **Color>96%** **Phenol>81%**	**Hooda et al., 2018**
Fungal Species		
Meruliusaureusand Fusariumsambucinum	**COD>89%** **Lignin>80%** **Color>80%**	**Malaviya and Rathore, 2007**
Phanerochaetechrysosporium	**COD>89%** **Lignin~80%** **Color~80%**	**Saritha et al ., 2010**
Phanerochaetechrysosporium	**COD>55%** **Lignin>70%** **Color~80%**	**Chopra and Singh, 2012**
Trametesversicolor	**COD~82%** **2,4,5-trichlorophenol>90%**	**Pedroza-Rodríguez and Rodríguez-V´azquez 2013**
Bierkanderaadusta Phenarochetecrysosporium	**Total Organic Carbon ~35%** **Lignin~75%**	**Costa et al., 2017**
Pleurotusostreatus	**Lignin>37%**	**Li et al., 2019**
PleurotusostreatusEB016	**COD~ 100%** **Phenol~90%**	**Heinz et al., 2019**
Scenedesmus sp.	**COD~75%** **BOD_5~85%**	**Usha et al., 2016**

and bacterial enzymes. The biological removal of lignin removal is considered more sustainable, eco-friendly, and cost-effective (Haqa et al., 2020).

CHAPTER 3
MATERIAL AND METHODS

The proposed study comprises three main aspects namely, 1). To study the current and advanced wastewater treatment technologies for Star Paper Mills Pvt. Ltd and Bindal Paper Mills Ltd., 2). To explore the possibilities for reduction of pollution load with application pretreatment and membrane filtration, 3). To facilitate increased reuse and restoration of purified water back into the process and reduce the wastewater footprint of pulp and paper manufacturing industries. This chapter describes the details of materials, instruments, methodology, and statistical analysis used during the investigation.

3.1 Selection of Paper Mill

In the present work, we have selected two paper mills viz. Star Paper Mills Pvt. Ltd. in Saharanpur, Uttar Pradesh, India (29.97°N, 77.55° E) and Bindal Paper Mills Ltd. in Muzaffarpur, Uttar Pradesh, India (29°28'18.17"N, 77°47'9.50"E) were selected for effluent collection. Star paper mill is a wood-based industry utilizing the wood of Eucalyptus and popular writing and printing paper along with paper boards with a production capacity of more than 72,000 MT/annum. It utilizes around 12000 m^3/day of water in its processing and discharges around 400 m^3/day of sludge and 1700 m^3/day of effluent into the environment which is ultimately discharged into the Hindon River, a tributary to river Yamuna. Bindal Paper Pvt Ltd is an agro-based byproduct such as “bagasse- a fibrous material of rice and wheat straws. This mill produces different variety of along with waste paper for the production of printing and writing grade paper having a production capacity of 11,0000 MT/annum utilizing 8500-9000 m^3/day of water and releasing approximately 7000-7500 m^3/day of effluent in Kali tributary of Hindon River.

Figure 3.1: Location Map of Paper mills selected for the current study

3.2 Collection of samples

Grab/ Spot samples of effluent coming out of the plant, primary clarifier (partially treated), and Secondary clarifier (final treated effluent) of the ETP were collected in plastic bottles from the two-paper mill (i.e., wood-based and agro-based). The bottles were pretreated by soaking in 10 percent HNO_3 acid and repeatedly rinsed with distilled water. Plastic bottles were properly labeled at the neck and bottom. The samples were kept in an ice box at $4°C$ and then brought to room temperature before analysis.

3.3 Determination of physicochemical parameters of the samples

Water quality parameters like pH and EC were analyzed on the spot. Whereas the parameters like BOD, COD, Color, Lignin, Sodium, and Potassium were analyzed in the laboratory. Table 3.2 presents the physico-chemical parameters for the analysis of effluent from the primary outlet and secondary outlet. The instruments and procedures used for the analysis of measuring parameters are depicted in the table.

Table 3.1 Physico-chemical parameters for analysis

SN	Physico-chemical parameters	Method used
1	pH	Potentiometer
2	Chemical oxygen demand (COD)	Open efflux (Potassium Dichromate Method)
3	Biochemical Oxygen Demand (BOD)	5 Days Incubation at 25°C
4	TDS	IS: 3025 (Part 16)
5	TSS	Gravimetric Method
6	Color	Spectrophotometric Method
7	Lignin	APHA method 5550B
8	Electrical Conductivity	Conductivity Meter
9	Sodium and Potassium	Flame Photometer

3.3.1 pH

PH is an important parameter to judge the microbial activity during wastewater treatment that explains the hydrogen ion concentration of the sample. IS 3025: Part 11 was embraced to determine the effluent pH value (IS:3025 Part 11, 1983). HM Digital pH meter PH:200 was utilized for measuring the water outflow pH. The pH meter has a resolution of 0.01 with an accuracy of ±0.02. Glass sensor and glass reference tube are used as a probe in this meter. pH meter was calibrated before taking the readings.

3.3.2. Electrical Conductivity

EC is used to measure the potential to pass the electric current through the sample. EC of the samples was measured on the spot using the HM digital AP-2 Aquapro water tester EC meter. This meter measures both EC and temperature simultaneously with a temperature range of 0-80º C. It was calibrated with 700µS solution. The working range of EC is 0-999µS/cm with a resolution of 1µS/cm and an accuracy of ±2%. The EC meter was calibrated from time to time. The samples having EC values above the working range were first diluted before taking the reading.

3.3.3 Total dissolved solids (TDS)

TDS is a term that talks about the total solids dissolved in effluent wastewater. Indian Standards (IS): 3025 (Part 16) was embraced for the analysis of TDS (IS: 3025 Part 16, 1984). A glass filter of size 40-60 µm was used to filter the collected effluent sample. The filtrate obtained was dried in a disk by the phenomenon of evaporation. The filtrate is further dried at approximately 180º C until consistent weight is obtained. The dried filtrate is then cooled by using a desiccator to accurately balance temperature and mass. The increase in disk weight is the TDS.

$$TDS, \text{mg/L} = \frac{(X2 - X1) \times 1000}{V} \qquad (1)$$

Where X_1 depicts the weight of the dish (mg)

X_2 depicts the cumulative dried residue weight + the dish (mg)

V depicts the sample volume

3.3.4 Total Suspended Solids (TSS)

The availability of suspended solids affects the water quality by inducing unfavorable physiological reactions making it important to analyze the TSS to control the different types of wastewater treatment process available and comply with the effluent regulations. The standard protocol IS: 3025 (part 17) for TSS analysis was followed.

For analysis, weighted glass fiber was used to filter the effluent sample. The filtrate retained on the glass fiber filter is dried at approximately105°C, till it attains a constant weight. A desiccator was used to cool the filtrate and for the balance of weight and temperature. The process continued till we obtained a constant weight. The weight altered of the filter represents the amount of TSS in the effluent.

$$TSS, \text{mg/L} = \frac{(A - B) \times 1000}{Sample\ volume, ml} \qquad (2)$$

Where A depicts the Filter weight and the residual weight (mg)

B depicts the filter weight (mg)

3.3.5 Chemical oxygen demand (COD)

COD is a parameter that defines the oxygen needed by the chemicals for oxidation of matter present in discharge effluent and is oxidizable. The COD was determined using the strong oxidizing reagent (dichromate) through the closed reflux method. The steps to determine COD are identified as per IS 3025 (Part 58) was used for analysis. The effluent samples were oxidized using a mixture of $K_2Cr_2O_7$ and Concentrated H_2SO_4. The sample, $K_2Cr_2O_7$, and Concentrated H_2SO_4 were taken in a ratio of 1:2:3. A pinch of $HgSO_4$ was added to remove chloride ion interference. The samples cooled and digested at 140ºC for two hrs. and were titrated with 0.01 N FAS.

The COD of the samples was calculated using

$$\text{COD (mg/L)} = \frac{(X-Y)\times N\times 8000}{V}$$

Where X is the volume FAS used in black,

Y is the volume of FAS of the sample,

N is the FAS normality

V is the sample volume

3.3.6 Biochemical oxygen demand (BOD)

BOD gives information regarding the present biological and organic pollutants in the wastewater discharge. It is measured by the determination of the dissolved oxygen content change due to the degradation of organic matter by microbes. The standard method mentioned in IS 3025: Part 44 was used for the assessment of the BOD of effluent. The amount of dissolved oxygen change is assessed for 5 days at 27±2º C temperature using the standardized iodometric method. The divalent manganese sulfate solution is added followed by a strong alkali solution. The oxidized manganese reverts to its divalent state when iodide ions are present and the iodine liberates equivalent to dissolved oxygen content. The Dissolved oxygen amount is measured post titration done by sodium thiosulphate solution by using an indicator known as the starch indicator.

3.3.7 Color

The color is the most important contaminant added by the pulp and paper manufacturing industry. It affects transparency and gas solubility. The major color source in the paper and pulp manufacturing industry is known as lignin. The standard method for color detection is Pt-Co. another name for this Pt-Co method is Hazen color (Standard 2120B). The measurement is based on the Hazen color scale ranging from 0-500 Platinum Color Units (PCU). A unit PCU is an amount of equivalent color produced by 1 mg/L concentration of platinum in the chloroplatinum ion.

The effluent was centrifuged at 10000 rpm for 30min and the pH of the supernatant solution was adjusted to approximately 7.6±0.05 and the absorbance to be adjusted at 465 nm was recorded using a Hitachi spectrophotometer (Model: U-2000).

The color units were calculated using:

$$CU\ (Pt\ Co) = \frac{A \times 50}{D}$$

Where A is the estimated color of the diluted sample

D is the dilution factor.

3.3.8 Lignin

Lignin is the most abundant natural polymer synthesis of commonly occurring monolignol precursors widely existing in the cell walls of plants. Due to the 3-D network of the linkages of monolignols the structure of lignin is extremely complex. Since most of the raw material is plant-based lignin as a coproduct of the pulp industry imparts color to the effluent. The lignin content was determined by the standard 5520B method of APHA [APHA 5520B, 1992]. The total extractives present in the effluent were spectrophotometrically determined using liquid-liquid extraction. A mixture of toluene and ethanol in a 2:1 ratio was used as extraction solvent. 50ml of the effluent was extracted in 100ml of the extraction solvent twice. The total amount of lignin was determined by diluting 2ml of the extract with 3% H_2SO_4 in a volumetric flask and the absorbance was recorded at 280 nm.

3.3.9 Sodium and Potassium

The determination of Sodium (Na^+) and Potassium (K^+) was carried out using flame photometry as per the standard (IS 3025: Part 45, 1993) method. The color intensity transmitted to the flame of a Meker-type Burner is measured by the flame photometer. The emission intensity of the filtered effluent samples, blank, and standard samples were determined at 589 nm for sodium and 766 nm for Potassium using a Systronics (Model-128) flame photometer. The concentration of Sodium and Potassium were calculated.

3.4 Optimization of parameters for Membrane Filtration

3.4.1 Experimental Setup for Optimization

The experimental design and the parameter optimization were done by the Grey-based Taguchi method and thereafter an ANOVA test was carried out. The Taguchi method is an instrument based on the combination of mathematical model and statistical calculation which incorporates fractional factorial experimental designs known as orthogonal arrays to minimize/ control the number of trials and parameters (Montogomery, 2004). The experiment was performed in a pilot plant through a benchtop membrane filtration unit (Figure 1). The volume of the feed solution was 5L for Ultrafiltration Membrane. The permeate coming out of the membrane setup was collected in a separate beaker and kept on electronic balance to calculate and analyze the flux.

3.4.2 Analysis of Flux decline and COD rejections

The flux decline (FD) was calculated to examine the outcome of the Working conditions on resistance applied on the membrane setup using the following equation:

$$Flux\ Decline = \frac{Jp(0) - Jp(i)}{Jp\ (0)} \quad (1)$$

The value of Flux declines indicates the membrane fouling during the experiment, Higher the value for flux decline, the more the membrane fouling.

The COD rejection rate was opted as a primary response parameter to evaluate the organic matter removal efficiency of membrane setup and was calculated as:

$$COD\ Rej\ \% = (1 - \frac{Co}{Ci}) \tag{2}$$

Where C*o* and C*i* represent the COD concentration of permeates and feed, receptively.

3.4.3 Experimental setup of the Taguchi Technique

Taguchi was used for designing the experiment to get a systematic decision tool to analyze the interaction among the control variables and optimization of parameters. The steps used for the methodology are described below and shown in Figure 3.2.

Step 1:

The Taguchi tool is based on the utilization of fractional experiment design namely OA for the reduction of the number of experiments for optimum working condition analysis. All the experiments were performed in completely randomized order with 4 different factors in 3 levels. So, an L9 (3^4) orthogonal array with 8 DF was selected for the same. The shortlisted factors with their ranges are pH: 6-8; Temperature: 20-32; Trans-membrane pressure: 2-4 Bar and Flow rate: 30-60 l/hr. To evaluate the optimum conditions, Flux decline caused by fouling of membrane and COD Rejection rate was shortlisted as the response attribute.

Step 2:

Sound to Noise Ratio (S/N ratio), affected by the criteria of the response variable finalized for optimization, is used to statistically measure the performance of the experimental results. The noise is always present in making the response variable deviate from the mean value. The S/N ratio hence makes it possible to find the deviation of characteristics performance from the desired value of the result. It is also used to determine the optimum operating conditions. S/N ratios have three divisions, 1). the-larger-the-better; 2). the-smaller-the-better; 3). the-nominal-the-better. For better performance of membrane and to achieve minimum flux decline by fouling of membrane, "the smaller the better option was chosen for the S/N ratio". The COD rejection rate was optimized "for the larger the better". The following equation shows the calculation of the ratio:

$$The\ larger\ the\ better\ \left(\frac{S}{N}\right) = -10\ log\ log\ \{\frac{1}{n}\ \sum_{i=1}^{n}\ \ \frac{1}{Yi^2}\} \qquad (3)$$

$$The\ smaller\ the\ better\ \left(\frac{S}{N}\right) = -10\ log\ \{\frac{1}{n}\sum_{i=1}^{n}\ \ Yi^2\} \qquad (4)$$

Where n denotes the number of repetitions performed to identify an experimental combination and *Yi* denotes the performance value of the *ith* experiment.

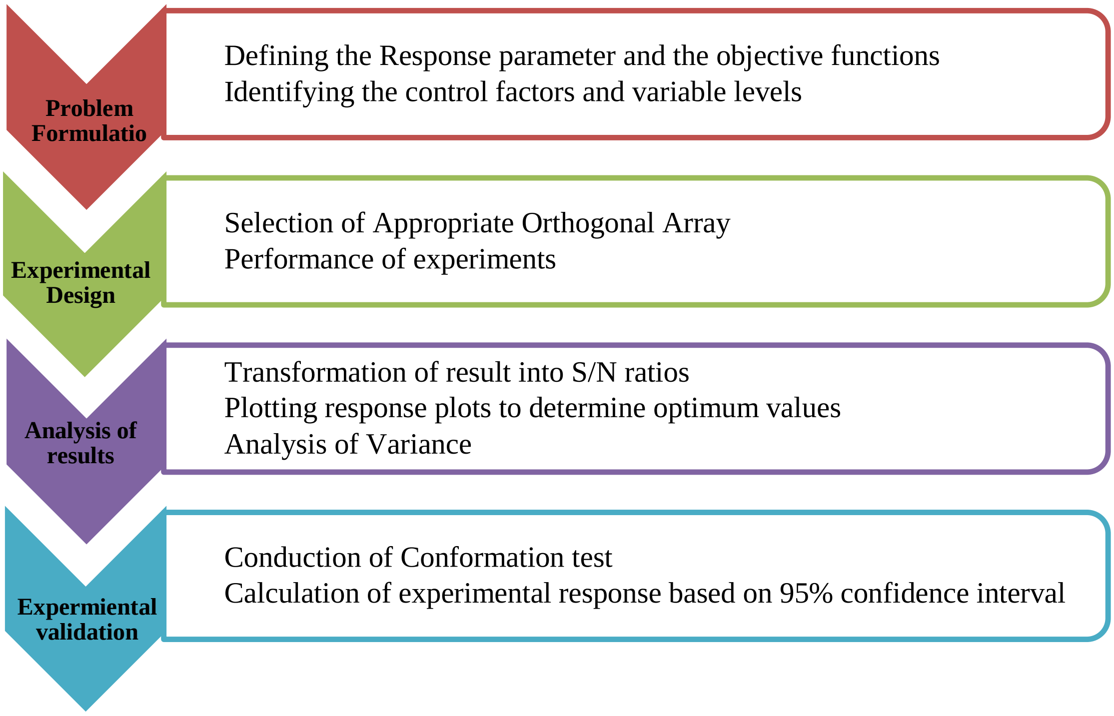


Figure 3.2: Flow Diagram of steps followed to optimize the ultrafiltration membrane for removal of pollutants using Taguchi method

3.5 Optimization of Multi Response using Grey relational analysis:

The relationship and influence of different parameters in a multi-response problem are very complex and do not provide a clear solution. This insignificant, poor, and uncertain information is known as grey. Grey Relational Analysis resolves this tangled uncertainty of multi-response parameters in the system and optimizes the problem by reaching a single response optimization problem known as a single relational grade (Panda et al., 2016).

Step 1:

Firstly, the value is normalized to avoid the difference in units and reduce the variability. To achieve this, a suitable value between 0 to 1 derived from the original value is decided (Haq et al., 2008). This step makes it easy to comparison between the original data. To minimize the response Smaller-the-better is used for normalizing the scale into an acceptable range using the below-mentioned formula (Tosun and Pihtili, 2010):

$$x*_a\ (k) = \frac{x_a\,(b) - x_a\,(b)}{max\, x_a\,(b) - x_a\,(b)} \tag{5}$$

Where, a =1,…,e; (e is the no. of experiments conducted)

b=1,…,r. (r denotes the no. of response attributes)

$x_a(b)$ = the original sequence

$x^*_a(b)$ = the sequence after processing of data

max $x_a(b)$ = the biggest value of $x_a(b)$

min $x_i(k)$ = the lowest value of the $x_a(b)$

x =desired value.

Step 2:

In this step of problem solving the grey relational coefficient (ξ_a(b)) is calculated from the normalized values using the equation:

$$\xi_a\ (b) = \frac{\Delta + \xi\Delta max}{\Delta oa(b) + \xi\Delta max} \tag{6}$$

Where Δ_{0a} is the deviation of the reference sequence and the comparability sequence calculated using

$$\Delta_{0a} = \|xo(b) - xa\ (b)\| \tag{7}$$

Where $xo\ (b)$ = the reference sequence

$xa\ (b)$ = the comparability sequence

Δ_{min} = least values of the absolute differences of $(\Delta_0 a)$

Δ_{max} = largest values of the absolute differences of $(\Delta_0 a)$

ξ = identification coefficient usually taken as 0.5.

Step 3:

The next step involves the calculation of grey relational grade (GRG) using the following equation :

$$y_a = \frac{1}{r}\sum_{b=0}^{n} \xi_a(b) \tag{8}$$

Where y_a = requisite grey relational grade for the *i*th experiment

r = no. of response attributes

The grey relation grade is the representation of the quality of characteristics and shows a correlation between the reference and comparability sequence of the experiment (Haq et al 2008)

Step 4:

The optimum level of process indicating the best functioning of the membrane is determined by choosing the values with high grey relation grade, which can be obtained using the average grade values for each selected level of the process. The highest average grade value was selected as optimal operating conditions for multi-response problems.

Step 5:

After finalizing the optimal conditions, ANOVA at a 5% confidence level was performed to evaluate the significance of different working parameters. This helps to find the contribution of each parameter to the response variables.

Step 6:

The conformational experiment was conducted after finding the optimal working combinations of different parameters. For the optimal working conditions, the predicted value of GRG was calculated using the equation (Sahoo and Sahoo 2013):

$$y_p = y_b + \sum_{i=1}^{o} (y_{mean} - y_b) \quad (9)$$

Where, yb = the cumulative mean GRG

y_{mean} = mean GRG at the optimal level for each chosen parameter

o = no. of significant parameters

3.6 Experimental set up for membrane filtration with pretreatment

The study is performed to test the efficacy of various assembled effluent treatment technologies. Three technologies viz., Pre-filtration unit (combination of pre-filter, sediment filter, and pre-carbon filter), Ultrafiltration (UF) membrane, and Reverse Osmosis (RO) membrane are set up in two different assembly combinations. The first combination depicts the series assembly of Pre-filtration and UF Membrane in series (Figure 3.3), while the second combination depicts the series assembly of Pre-filtration, UF Membrane, and RO Membrane in series (Figure 3.4). These combinations are employed with a diaphragm-type pump (Specifications in Table 3.3) that operates up to 140 PSI. The pump circulates the effluent from the pre-filter set up to the UF membrane. A flow meter is connected to collect the permeate (i.e., the treated water) from the experimentation unit.

Table 3.2 Specification of Pump used in the study

SN	Parameter	Value
1	Nominal flow rate	1.5 LPM
2	Maximum pressure output	140 PSI
3	Maximum pressure input	60 PSI
4	Current	1.10 A
5	Voltage	24 V

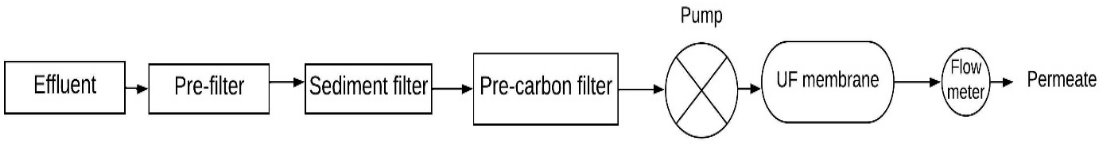


Figure 3.3. Flowchart of experiment set up (Combination 1: Pre-filtration and UF Membrane installed in series)

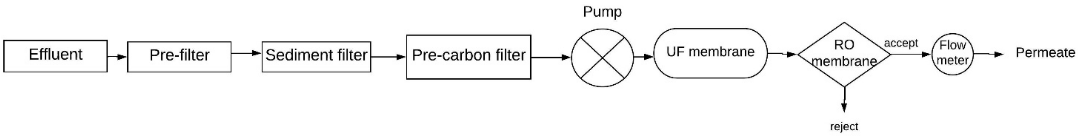


Figure 3.4. Flowchart of experiment set up (Combination 2: Pre-filtration, UF Membrane, and RO Membrane in series)

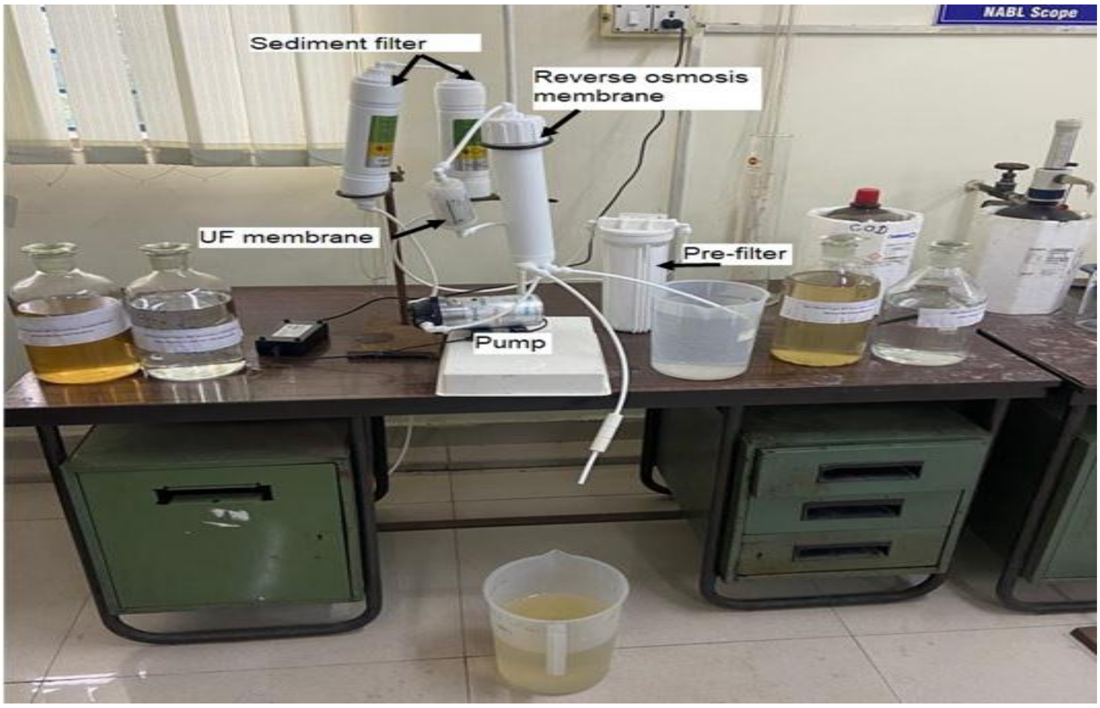


Figure 3.5. Experiment set up effluent treatment

3.7 Statistical Analysis:

Each treatment has 3 replicates. All data are denoted as the mean value ± standard deviation (Mean ± SE). Many differences among various means were determined by ANOVA at a 5% significance level using SPSS Version 19.00.

The present study was subjected to two multivariate statistical techniques to set up the relationship among the various complex data sets. For multivariate statistical tools, cluster analysis (CA), correlational analysis, and principal component analysis (PCA) were used.

For Correlation and comparison analysis, the Spearman test was used.

CHAPTER 4
RESULTS AND DISCUSSION

Membrane technology has come up as an alternative and advanced method for waste water treatment for water reuse and can help in overcoming the problem of water scarcity. The best advantage of membrane technology is that the water can be directly reused in the industrial process without any further treatment. To assess the potential of using membrane technology in pulp and paper manufacturing mills, one of the most water-intensive industries was selected. Wastewater samples were collected and the Physico-chemical parameters were characterized periodically to determine the level of the pollution loading. Various parameters affecting the water quality were analyzed in the laboratory as per (APHA 2005) standard methods.

4.1 Water Consumption by Bindal Paper Mills

Bindal Paper Mills Ltd. Muzaffarnagar, UP, is an integrated pulp and paper plant with a capacity of 110 MTons/Annum of writing and printing grade paper using agro waste like bagasse, wheat straw, and hardwood and poplar grown by the farmers.

Table 4.1: Fresh water consumption in different processes in Bindal Paper Mills

S.N.	Process	Freshwater consumption, m^3/ day* @ 230 TPD paper production
i.	Raw material Preparation (Including Chemical Preparation & Hydrant)	316
ii.	Pulp Mill (Pulping, Pulp Washing & Bleaching)	2850
iii.	Paper Machine & Stock Preparation	3500
iv.	Boiler (Recovery & Utility)	1600
v.	Domestic	1100
vi.	Miscellaneous	1824
vii.	Total	11190 (52.3 m^3 / T finished paper) #

4.2 Characterization of effluent generated from Bindal Paper mill Ltd

Throughout the study, the focus was on assessing and controlling the existing TSS, TDS, COD, BOD, color, and pH owing to the peculiar wastewater from the pulp and paper industry. Effluents treated after secondary treatment of agro-based industry are shown in Table 4.2. Depending on the raw materials (agricultural and wood-based in our study), operating process, and climate conditions, pollutants vary in the effluent. The effluent from the industry is characterized by alkaline pH, high biological oxygen demand (BOD), chemical oxygen demand (COD), suspended particles (primarily fibers), fatty acids chlorides, sulfates, nitrates, lignin, and its derivatives, and a large number of mutagenic pollutants (Kumar et al., 2022).

As seen from Table 4.2, the TSS in agro-based paper mill effluent (56 mg/L) after secondary treatment is lower than most of the pollutants. The pH value of effluent from the secondary clarifier is slightly alkaline (7.46±0.44). The BOD and COD values of the effluent coming out of the secondary clarifier were observed in the range of 27±0.56 mg/L and 216±9.11 mg/L respectively. It has been reported that BOD and COD of untreated effluent in pulp and paper mills fall in the range of 650-1500 mg/L and 2000-10000 mg/L, respectively (Gupta, A., & Gupta, R., 2018). The TSS value of the effluent is 1911±38 mg/L, however the secondary clarifier has been able to reduce it to 56±0.96mg/L. The average value of TSS from the paper industry is 1980.65-2785.79mg/L (Singh and Tripathi, 2020). The color of the untreated effluent was recorded to be 1206±4.1 PCU, and it has been reported in the literature that wastewater from pulp and paper industries has the color of untreated effluent in the range between 1000-12000 PCU (Kumar et. al., 2022). Similarly, the Lignin content is 1004±6.2 which is extremely lesser than the reported effluents from the pulp and paper industry between 11000 and 250000 mg/L (Butani and Mane, 2017; Den5). The Bindal Paper Pvt ltd is agro waste-based industry and the lignin content in the agro waste is lesser in comparison to the wood-based industries (Tao et al., 2018). The effluent lignin content in the study after the secondary treatment was found to be 126 mg/L.

Table 4.2: Physico-chemical properties of effluent from Bindal paper mill

Parameter	ETP Inlet	Effluent after primary clarifier	Effluent after secondary clarifier	CPCB limits
pH	7.39	7.82	7.46±0.44	5.5-9.0
TSS mg/L	1911	130	56±0.96	100
TDS, mg/L	2967	2889	2215±56.45	2100
COD, mg/L	1465	936	216±9.11	250
BOD, mg/L	653	494	27±0.56	30
Color, PCU	1206	945	412±21.1	500
Lignin, mg/L	1004	856	126±7.71	-
Conductivity, µS/cm	3125	2942	2630±81.21	-
Sodium (Na), mg/L	183	141	71±2.3	-
Potassium (K), mg/L	14.6	12.1	8.8±0.27	-

4.3 Effluent treatment technique available in Bindal paper mills

The current ETP of Bindal Paper Mill is based on the conventional activated sludge process along with advance tertiary treatment techniques comprised of an equalization tank, the primary clarifier, an aeration tank, a secondary clarifier, a Sand filter, Dissolved Air Floatation, a Direct Membrane filter (DMF), Sludge Bed Drier (SDB) and chemical recovery plant. The mill has also placed an Online Continuous Effluent Monitoring System (OCEMS) at the final output to monitor the quality of the treated effluent 24×7. The treated wastewater after the secondary clarifier is released into the Hindon River via a concrete underground drain.

The Layout of the existing ETP at Bindal Paper Pvt ltd, Muzaffarnagar, UP, India is shown in figure 4.1.

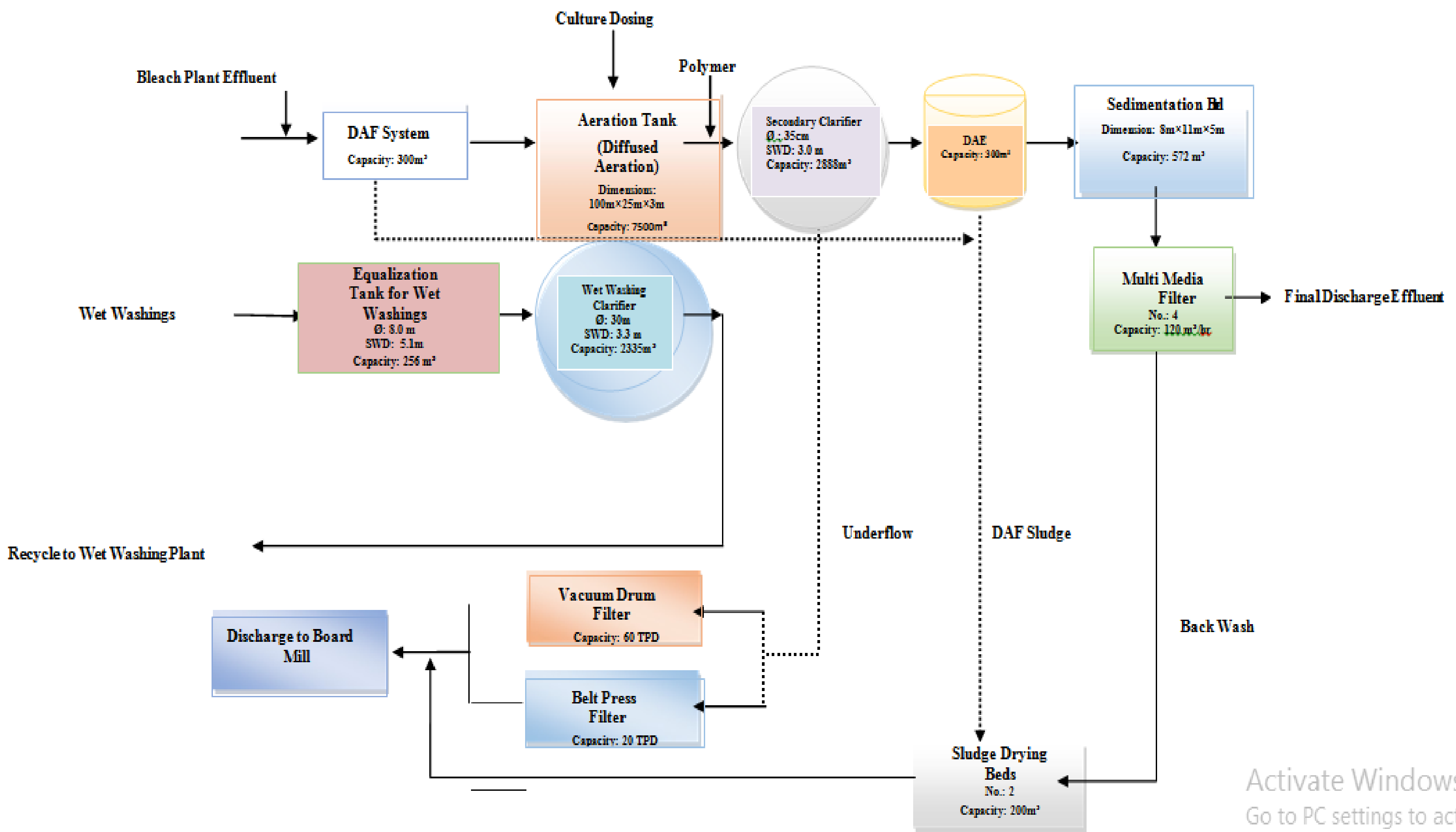


Figure 4.1 ETP Flow Diagram, Bindal Papers Mills Ltd

4.4 Efficiency of Effluent treatment plant at Bindal Paper Mills

With the introduction of a new Charter by the Central Pollution Control Board in 2015 that focuses on the reduction in consumption of fresh water and efficient and stringent effluent discharge norms, Bindal Paper Mills Ltd has worked on different concepts and adopted a systematic plan to achieve Zero Liquid Discharge. These Strategies involve 1). Pollution load reduction at source, 2). Zero or Minimum discharge of effluent from the individual sections of the Mills and lastly, 3). Up gradation of the existing effluent treatment plant.

To achieve the reduction of pollution load at the source the mill has started treatment and reusing the water from wet washing water after clarification to reduce the pollution load on the Effluent Treatment Plant. Mill has adopted the Oxidative Chemical Bleaching, a new technology of bleaching instead of conventional Hydrogen peroxide. Moreover, they have introduced enzymes instead of bleaching chemicals which further reduces the pollution load.

Moreover, the mill has established 4 components micro-particle and micro-polymer-based retention control to achieve approximately 85% first-pass retention and 65% of first-pass ash retention. Also, the mill has introduced an especially suited biocide program to avoid the boil out of the paper mill. The heat and chemical recovery has been installed from the dissolver vent in the chemical recovery boiler.

To ensure Zero or Minimal effluent discharge from the individual segment of the mills the following measures are taken by the mill.

1) Pulp Mill

In the pulp processing mill, the freshwater is used solely for the preparation of chemical stock solutions, and the water is used as a coolant for cooling the glans in the pumps. The back water generated after washing of raw material was earlier filtered in a sand and pith filter and conical tanks. The underflow water from the conical tanks was passed through the sand riffler having numerous compartments that settle the sand the pitch present in back water. But now the underflow water of the sand riffler is carried to the clarifier which helps in further settling of suspended solid.

After screening of the pulp, it is taken for bleaching with bleaching sequence D-O/Oxidative. The introduction of a new OX sequence for bleaching has further substantially reduced the pollution load.

Below is the table showing the reduction in the effluent generation in the pulp mill

Table 4.4: Reduction in effluent generation after recycling in Bindal Paper Mill Ltd

Purpose	Fresh Water Consumption before Recycling (m^3/hr)	Fresh Water Consumption After Recycling (m^3/hr)	Fresh Water Savings (m^3/hr)
Chemical Preparation	35	35	0
Wet Washing	20	0	20
Water used on DO washer	93.5	0	93.5
Pump sealing water	35	15	20
Air Compressor water recycling	2	2	0
Sum Total	~185.5	~52	~133.5

The seal water generated after gland cooling is now collected in a pit and filtered through Vibro- screen to be reused as coolant instead of the freshwater. Hence, the fresh water earlier utilized just for gland cooling can be conserved.

2. Paper Mill

Fresh water in a paper machine is used in the wire and press part of the mill and for the preparation of chemical stock. For recycling of white water in this section two specially designed Dissolved Air Floaters (DAF) have been installed. One of the DAFs treats the excessive cloudy water of the disc save and water that comes out is used in the bleaching section of the pulp mill. The second DAF recovers the water coming along with centri-cleaner rejection. The clean water coming out is used at the wire and press section. The 2nd DAF, the fresh water in the paper mill, is only used in lubrication showers of rolls, chemical stock preparation, and high-pressure showers of wire and press parts. Thus, the operation of the paper machine has become a completely closed loop.

Below is the table showing details of Fresh water consumption before and after the Dissolved Air Floater.

Table 4.5: Fresh water consumption in Bindal paper Mill Ltd before and after Dissolved Air Floater (DAF)

<table>
<tr><th>Section of Mill</th><th>Consumption of Water before treatment (m^3/hr)</th><th>Replacement with Treated Wastewater (m^3/hr)</th><th>Fresh Water saving (m^3/hr)</th><th></th></tr>
<tr><td>Preparation of chemicals (1)</td><td>8</td><td>8</td><td>0</td><td>8</td></tr>
<tr><td>Wire Part of the mill (2)</td><td colspan="4"></td></tr>
<tr><td>Wire Rolls + Apron Lips + Trim Knock Off + Ceramic Lube + Window Cleaning + Couch Fog + Couch Cleaning + Deckle Board</td><td>177.48</td><td>177.48</td><td rowspan="2">177.48</td><td rowspan="2">86.12</td></tr>
<tr><td>Wire HP + Sheet Wetting + Deckle Lube Top</td><td>86.12</td><td>0</td></tr>
</table>

Press Section				
Suction Pick up (Lube + Flush) + Suction Press (lube + Flush)	93.3	93.3		
Felt (HP + Lube)	46	0	93.3	46
TOTAL (3)	139.3	93.3		
Cooling Tower (4)	26	26	26	0
Sum Total (1+2+3+4)	437	297	297	140.0

Table 4.6: Reduction in effluent by water recycling in Bindal Paper Mill Ltd

Section of Mill	**Fresh water consumption before Recycle (m^3/hr)**	**Fresh water consumption after Recycle (m^3/hr)**	**Percentage reduction in Fresh Water Consumption**
Pulp Mill	185.5	52	72
Paper manufacturing unit	437	140	69
Power House	66.7	62.5	6.3
Others	0.8	0.8	0
Total (m^3/hr)	690.0	253.3	63.3
Total (m^3/Day)	16,560	6,080	
Production (TPD)	200	300	
Fresh Water Consumption (m^3/Ton of paper)	~82.8	~20.3	

4.6 Up gradation of ETP plant in Bindal Paper Mills Ltd

The combined results of the above-mentioned changes on the effluent load have been shown in the table below:

Table 4.7: Change in the total Load on the Effluent treatment plant of Bindal Paper Mills Ltd after up gradation of ETP

Stream of wastewater		Change in total ETP load (%)		
		Before Modification	After Modification	
Paper Machine and Stock Preparation		~ 10	Nil	Approximately 60 % reduction in ETP load has been observed after the application of the Zero/Minimal Discharge Strategy
Chemical Recovery Plant		~ 10	Nil	
Pulp Mill	Raw Material Washing Plant	~ 40	~ 10	
	Bleach Plant	~ 40	~ 30	

4.6.1 Technical Changes in the Effluent Treatment Plant

The pollution load of effluent treatment plants was reduced to approximately 60% just by implementing the zero-discharge strategy through modification. The Influent of the treatment plants is bifurcated into two streams: 1). Wet Washing Effluent, 2). The bleach Plant Effluent.

1) Wet Washing Effluent:

The effluent from wet washing is primarily treated with hydrated lime also called lime softening and then transferred to an equalizing tank where a consortium of aerobic as well as anaerobic cultures is added to control the BOD and COD of the waste water.

The pre-activation of microbial cultures was achieved using the influent and air diffuser. Then these activated cultures are inoculated in the effluent stream where they work as catalysts to decompose the biomass. Finally, the effluent is allowed to settle the clarifier for 5-6 hrs of retention time and finally used for the washing of raw material.

2) Bleach plant effluent:

The effluent generated from the bleaching plant has very low TSS in a range between 80-120 PPM but the temperature is very high in a range of 55-60ºC. But conventional treatment like activated sludge treatment needs a range of 30-35ºC. Hence, it is necessary to reduce the temperature for further treatment.

4.6.2 Implementation of the latest technology in the Effluent Treatment Plant of Bindal paper Mills Ltd

1) Mist Cooling system:

Usually, the mills install cooling towers to reduce the temperature of the effluent but Bindal paper mills have adopted a mist cooling system which forms micro mist particles of size of around 5 microns. The effluent collected in the equalization tank is used as a feed to the mist cooling system. Here the temperature of the effluent reduces to around 32-35º C, along with this these mist particles also absorb oxygen from the atmosphere.

The effluent from the bleaching plant and the excess clarified wet-washing effluent are transferred to the aeration at a rate of 0-15%. Table 4.7 below shows the characteristics of combined effluent (Bleach Plant + Wet washing) at the aeration tank inlet:

Table 4.8: Characteristic of effluent at aeration tank input in Bindal paper Mills Ltd

BOD	750-800 mg/L
COD	2200-2500 mg/L
TSS	100-110 mg/L

2). Installation of Mist Aeration System

Earlier, the mill effluent feeding the aeration tank is passed through the activated sludge process treatment stage where the surface aerators are installed. Now, the mist aeration system has been installed in the aeration tank. This system exposes the mist to the atmosphere and this mist falls back after absorbing oxygen from the atmosphere. The reduction of BOD and COD after the Activated sludge process is as in table 4.8

Table 4.9: Pollution Reduction after Activated sludge Process in Bindal paper Mills Ltd

Parameter	Percentage Reduction
BOD_5	~95-97
COD	~80-85

4.7: Installation of tertiary Treatment System at Bindal Paper Mill Ltd

Conventional Secondary treatments like biological processes do not reduce the pollution load to the level that water can be reused in the paper making and also do not provide satisfactory results for protecting the river water from pollution by waste discharged from the effluent treatment plant of the mill. For tertiary treatment, physico-chemical methods based on solid-liquid separation have been employed and a particularly designed Dissolved Air Floatation system has also been installed.

The physicochemical method consists of Coagulation, Flocculation, and Separation process. Coagulation neutralizes the negative charges contained by pollutants in wastewater. On the other hand, flocculation focuses on the development of flocs, which separates the solid part of pollutants present in the effluent. Finally, the flocs produced are removed from the liquid by air diffusion. Below is the table providing the variation of various pollutants using the physicochemical treatment:

Table 4.10: Pollution Reduction after Implementation of Tertiary Treatment in Bindal paper Mills Ltd

Pollution parameter	Tertiary Treatment inlet	Tertiary treatment Outlet	Percentage Reduction
BOD_5 (PPM)	27±0.56	~8-10	>65
COD (PPM)	216±9.11	~100-120	>65
TSS (PPM)	56±0.96	~15-20	>60
Color (PCU)	412±21.1	~50-60	>80
Turbidity (NTU)	50±1.12	~12-15	>70

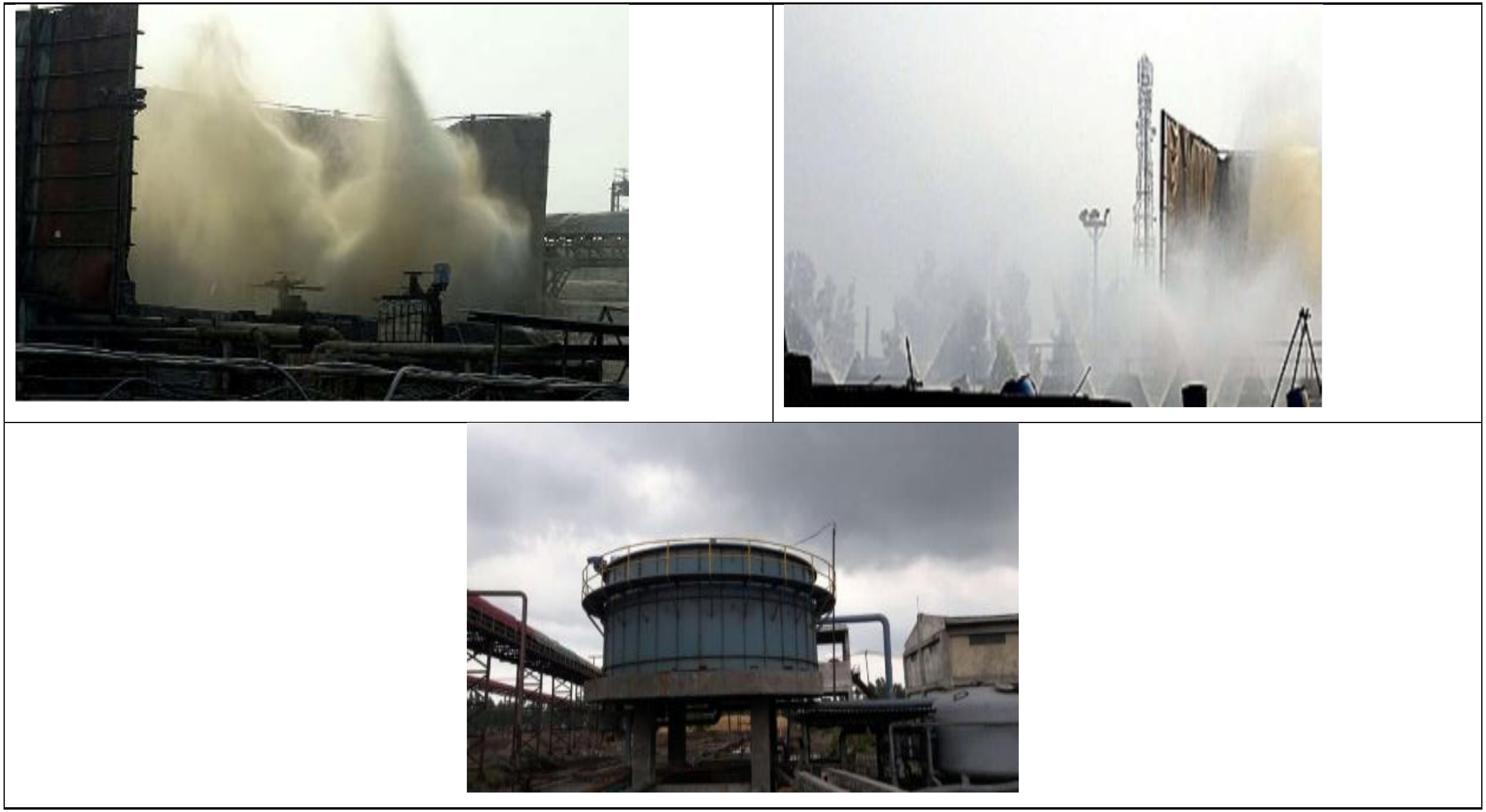


Figure 4.2: ETP at Bindal Paper Mills Ltd

4.7.1 Water Consumption by Star Paper Mills Pvt Ltd

Star paper utilizes the wood of eucalyptus and poplar for paper making. The mill has 4 paper machines with a production capacity of 300 Tonnes/day. The mill utilizes traditional chlorine bleaching technology and also involves an oxidative extraction stage which utilizes oxygen and peroxide. Moreover, the mill also has an oxygen delignification process installed before the bleaching to reduce the consumption of bleach chemicals and reduce the pollution load.

Star paper mills utilize around 12000 m^3 of freshwater (groundwater) daily, which is abstracted from the bore wells. Ten bore wells are available for water extraction but only 5 are functional at any given point of time. The flow meter is installed in the bore wells. The consumption of fresh water during different processes in paper making by the mill is mentioned in table 4.10. The stock preparation and paper-making machine utilizes a maximum amount of water (3500 m^3/day) followed by pulping which utilizes 2850 m^3/day.

Table 4.11: Fresh water consumption in different processes in Star Paper Mills Pvt Ltd.

S.N.	Process	Freshwater consumption, m^3/ day* @ 230 TPD paper production
1.	Raw material Preparation (Including Chemical Preparation & Hydrant)	316
2.	Pulp Mill (Pulping, Pulp Washing & Bleaching)	2850
3.	Paper Machine & Stock Preparation	3500
4.	Boiler (Recovery & Utility)	1600
5.	Domestic	1100
6.	Miscellaneous	1824
7.	Total	11190 (52.3 m^3 / T finished paper) #

***Figures provided by the Mill** *# finished production: 214 tpd*

4.7.2 Physico chemical Properties of waste water generated from Star Paper mills

Throughout the study, the focus was on assessing and controlling the existing TSS, TDS, COD, BOD, color, and pH owing to the peculiar wastewater from the pulp and paper industry. Effluents treated after secondary treatment of wood-based are shown in Table 4.11. In India, most integrated paper mills use wood-based raw materials such as eucalyptus, bamboo, etc. A region's climatic conditions and geography determine the physicochemical properties of wood.

As seen from Table 4, the pH of the effluent is in the alkaline range (8.12±0.34) which is well within CPCB limits. The TSS from wood-based paper mills (i.e., 36±1.12 mg/L) after secondary treatment is lower than most of the pollutants. The BOD and COD of the effluent were 715±9.21 mg/L and 1395±32.1 mg/L respectively. It is reported that BOD and COD of untreated wastewater in pulp and paper mills fall in the range of 170.32-670.42mg/L and 705.52-2000.55 mg/L, respectively (Singh et al., 2020). The effluent treatment plant in the mill could reduce the BOD and COD in the range of 20-27 mg/L and 183-216 mg/L (Table 1). The TDS is still on the higher side though (i.e. between 1933±97.1mg/L and 3114±102.1 mg/L). It is important to mention the efficacy of the effluent treatment plant in the reduction of the color of the effluent from 1421±71.4PCU - 615±23.21 PCU as most wastewater from pulp and paper industries has the color of untreated effluent in the range between 1000-12000 PCU (Kumar et.al., 2021). Mechanical and chemical effluents from the pulp and paper industry have lignin content between 11000 and 250000 mg/L (Butani and Mane, 2017; Deng and Zhao, 2015). The effluent lignin content in our study after the secondary treatment was found to be 168±8.78 mg/L. Likewise, the electrical conductivity, sodium, and potassium content are at the desired levels (Izharul et al., 2016 Izharul et al., 2017).

Table 4.12: Physico-chemical properties of effluent from Star paper mill

Parameter	ETP Inlet	Effluent after primary clarifier	Effluent after secondary clarifier	CPCB limits
pH	8.41	8.25	8.12±0.34	5.5-9.0
TSS mg/L	2016	170	36±1.12	100
TDS, mg/L	3114	2791	1933±87.46	2100
COD, mg/L	1395	891	183±11.09	250
BOD, mg/L	715	502	20±0.79	30
Color, PCU	1421	1002	615±23.21	500
Lignin, mg/L	1192	931	168±8.78	-
Conductivity, µS/cm	3281	3028	2820±91.23	-
Sodium (Na), mg/L	216	178	28±2.3	-
Potassium (K), mg/L	16.23	12.98	4.2±0.27	-

4.7.3 Effluent treatment technique available in Star Paper Mill

The current ETP of Star Paper Pvt ltd is based on the traditional activated sludge process and comprises mostly of the primary clarifier with a capacity of 10087 m^3 with a Side Water Depth of 4m; an aeration tank with 11 aerators of 50HP capacity; A secondary clarifier with a capacity of 6005 m^3. The plant is also installed with a belt press for dewatering the sludge generated in primary and secondary clarifiers. The water in the primary clarifier is fed after screening through the bar screen to remove the suspended solids. The overflows of the primary clarifier are pumped into the aeration tank equipped with 11 surface aerators, each with a capacity of 50 HP and a 50 HP blower to provide proper aeration to maintain the content of dissolved oxygen levels essentially required to maintain the dissolved oxygen levels for appropriate microbial metabolic activity. The under flow of the primary clarifier dewatered to a consistency of 30% with the help of a sludge filter press. The effluent after aeration is transferred to a secondary clarifier with a capacity of 6005 m^3 and a side water depth of 3.65 m. The underflow of the secondary clarifier is recycled back to the aeration tank to maintain the level of mixed liquor-suspended solids to around 3000 mg/L. and the rest underflow is dewatered using the volute press. The treated wastewater after the secondary clarifier is released into the Hindon River via a concrete underground drain. The mill has also placed an Online Continuous Effluent Monitoring System (OCEMS) at the final output to monitor the quality of the treated effluent 24×7.

The Layout of the existing ETP at Star Paper Mill Ltd, Saharanpur, UP, India is shown in figure 4.3

4.7.4 Efficiency of Effluent Treatment Plant at Star Paper Mills

The adequacy of the various units of the existing ETP at Bindal Paper Mills Ltd was assessed based on influent retention time in each unit, surface overflow rate in primary and secondary clarifiers, volumetric loading rate, and estimation of oxygenation capacity of existing aerators in the aeration tank for oxidation/degradation of BOD load available. Table 4.3 summarizes the assessment of ETP adequacy.

The information on pH, TSS, TDS, COD, BOD, and color were analyzed in September 2021. Based on the studies conducted to assess the adequacy of the Effluent Treatment plant (ETP) at Star Paper Mills Ltd, Saharanpur, CPPRI has the following observations to make:

I. The retention time available in the primary clarifier, aeration tank, and secondary clarifier is sufficient to facilitate the satisfactory removal of suspended matter in clarifiers and the oxidation of biodegradable organic matter in the aeration tank. More than 50% of TDS is removed from the primary clarifier outlet to the secondary clarifier outlet As per the information received (September 2021), the secondary clarifier was able to significantly reduce the TSS from 123 mg/L to 53 mg/L.

II. The number of surface aerators provided in ETP i.e. 11 × 50 HP = 550 HP, 1 × 30 HP = 30 HP and 1 x 50 HP air blower are adequate to provide sufficient oxygen for oxidation of biodegradable organic matter. The aeration capacity is adequate to treat an even higher BOD load than the existing BOD load provided the ETP is operated under optimum conditions. BOD is lowered from 356 mg/L at the outlet of the primary clarifier to a sufficiently lower amount of 20 mg/L. Likewise, COD is reduced from 894 mg/L to 252 mg/L.

III. The mill has significantly reduced its water consumption with the adoption of modern fiber line technologies like oxygen delignification, new pulp washers as well as optimization of process operations and water requirement process-wise, good housekeeping practices.

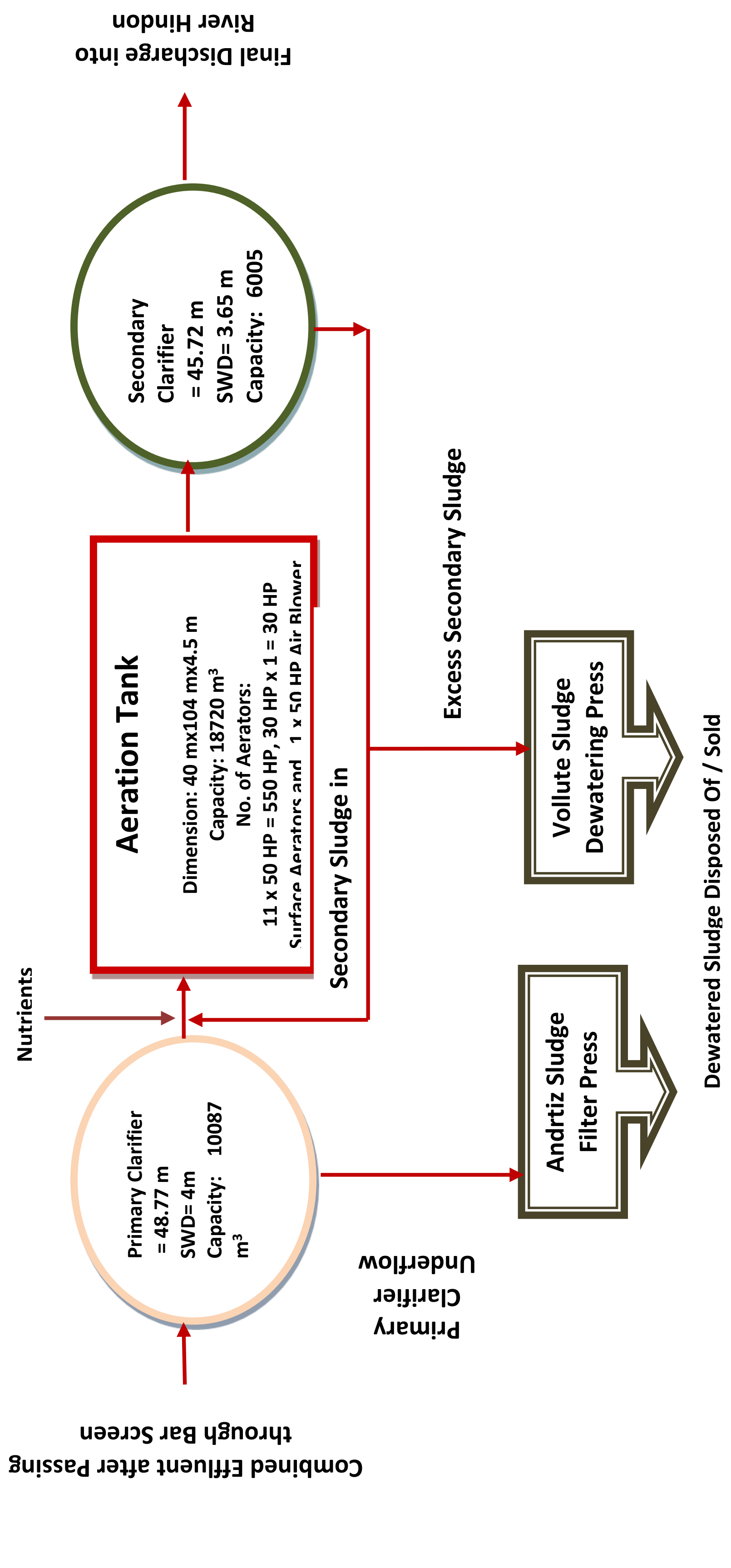


Figure 4.3: Layout of Existing ETP at Star Paper Mills Ltd., Saharanpur

Figure 4.4: ETP at Star Paper Mills

Table 4.13: Efficiency evaluation of the Effluent Treatment Plant of Star Paper Mills

S. No.	Unit	Design Specifications	Remarks
1.	Primary clarifier Effluent flow, m^3/hr.: 347	Capacity: 10087m^3 Surface Area, m^2: 2522 Retention time, hr.: 29 SOR, m^3/m^2/day: 3.3	Adequate to handle even excess effluent volume up to 30,000 m^3 /day
2.	Aeration tank Effluent flow after recirculation**, m^3 / hr.: 434 BOD load after primary clarifier#, kg/ day: 4110 kg/hr: 171	Capacity: 18720 m^3 Retention Time:43 hr VLR, kg BOD/ m^3 /day: 0.22 F/m ratio: 0.11 No of Aerators:12 Total HP: 630 O_2 required: 342 kg/hr Available O_2: 528 kg/hr	Adequate in terms of handling capacity Aeration is adequate to treat even excess BOD load up to 6500 kg/day i.e., more than the present BOD load
3.	Secondary Clarifier Effluent flow. m^3/hr: 434	Capacity, m^3: 6005 Surface Area, m^2: 1645 Retention time, hr: 13.8 SOR, m^3/m^2/day: 6.33	Adequate
4.	Sludge Filter Press Volute Press	Capacity: 10 tons dry solids /day Capacity: 4.8 tons dry solids /day	Adequate to handle ETP sludge

4.8 Optimization of the membrane using the Taguchi Method

The experimental design outcome of the Taguchi method for using Ultrafiltration membrane wastewater treatment of Paper and pulp mill was performed (Orrego et al., 2011). The experiments were performed in completely randomized order with 4 different factors in 3 levels. So, an L9 (3^4) orthogonal array with 8 DF was selected for the same (Kadhim et al., 2020). The experimental matrix is shown in table 4.13. The shortlisted factors with their ranges are pH: 6-8; Temperature: 20-32; Trans-membrane pressure: 2-4 Bar and Flow rate: 30-60 l/hr (Table 4.14). The flux decline and COD rejection in each experiment are shown in Table 4.13. The maximum flux decline of 18.48% caused due to fouling has been observed in experiment no. 5

whereas a minimum flux decline of 5.43% was observed in experiment no. 8. For COD rejection Maximum COD rejection has been achieved in experiment no. 6 with a percentage of 19.64% and a minimum of 7.14 % has been observed in experiment no. 1.

Table 4.14: Operating parameters and their labels and levels

Parameter	Labels	L1	L2	L3
pH	A	6	7	8
Temp	B	20	26	32
Pressure	C	2	4	6
Flow Rate (l/hr)	D	30	60	90

Table 4.15: Experimental layout using L9 (3^4) orthogonal array following the Taguchi method

Experiment No.	pH	Temperature(°C)	Pressure(bar)	Flow Rate (l/h)
1	**6**	**20**	**2**	**30**
2	**6**	**26**	**4**	**60**
3	**6**	**32**	**6**	**90**
4	**7**	**20**	**4**	**90**
5	**7**	**26**	**6**	**30**
6	**7**	**32**	**2**	**60**
7	**8**	**20**	**6**	**60**
8	**8**	**26**	**2**	**90**
9	**8**	**32**	**4**	**30**

The overall mean S/N value for FD caused by fouling was calculated as -21(dB) and for COD rejection it was 22.10 (dB). As seen in Figure 4.5 for flux decline, the flow rate shows the lowest variation. However, from the slope of the graph, it can be easily predicted that pH shows remarkable variation around the mean S/N. The maximum variation in the pH was calculated as -4.1. On the other hand, for COD rejection both pressure and flow rate have minimum variation whereas pH and Temperature show the maximum variation.

4.8.1 Effect of pH:

Figure 4.5a depicts the effect of pH on the fouling of membranes. The highest S/N ratio of -17 was observed at pH 8 and the S/N ratio drastically decreased at pH 6 and

7. It may be because of the Iso-electric point of the membrane as the membrane gets positively charged at lower pH. Also, the negatively charged dissolved compounds in wastewater affect the same. Least membrane fouling was observed at pH 7. For COD decline a positive and higher value of S/N is required under optimum conditions. In Figure 4.5b the largest S/N ratio has been observed at pH 7 and a decrease in pH value negatively impacts the COD rejection.

4.8.2 Effect of Temperature

Figure 4.5a depicts the effect of temperature on the fouling of ultrafiltration membranes. It can be observed that the S/N ratio shows a negative correlation with temperature. Hence the least fouling was observed at 32° C. Due to the reduction in solvent viscosity with increased temperature leading to increasing the flux. At higher temperatures, the membrane also expands thermally impacting the fouling. Maximum level of membrane fouling is observed at 20° C. For COD rejection, the S/N ratio is found maximum at 32°C making it best suited for the treatment process.

4.8.3 Effect of trans-membrane pressure

Figure 4.5a depicts the effect of trans-membrane pressure on the flux decline and COD rejection. The S/N ratio was observed highest at 2 bar indicating the highest flux decline as per Darcy's law, the increase in trans-membrane pressure increases the initial and final flux value. However, applying higher trans-membrane pressure could lead to membrane blocking resulting from concentration polarization. Application of more trans-membrane pressure results in the accumulation of huge amounts of pollutants on the surface of the membrane forming a gel layer. This gel layer increases osmotic pressure leading to higher flux decline by cutting down the driving force (Gonder et al., 2011). Trans-membrane pressure does not seem to have any impact on the COD rejection since the S/N ratio is near the average values.

4.8.4 Effect of Flow Rate

Figure 4.5 depicts the effect of flow rate on the fouling of membranes. The largest S/N ratio of -21.2 Db was observed at 90 l/hr. S/N ratio increased with increasing flow rate.

At least FD was observed at 30 l/hr. This can be attributed to the adsorption of more pollutants forming a thick layer onto the surface of the membrane at higher flow rate values. For COD rejection the S/N ratio has slightly maximum values at higher flow rate.

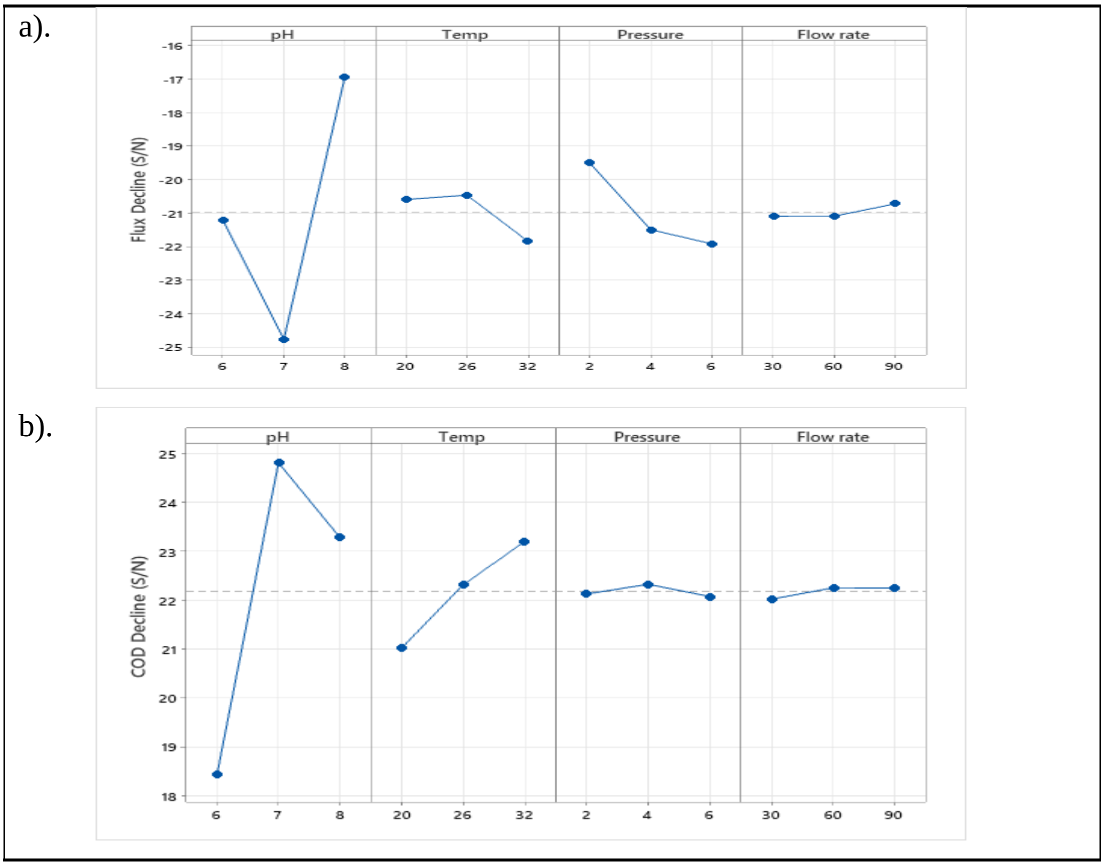


Figure 4.5: Mean effect curves for S/N ratios for a) Flux Decline; b) COD rejection Rate

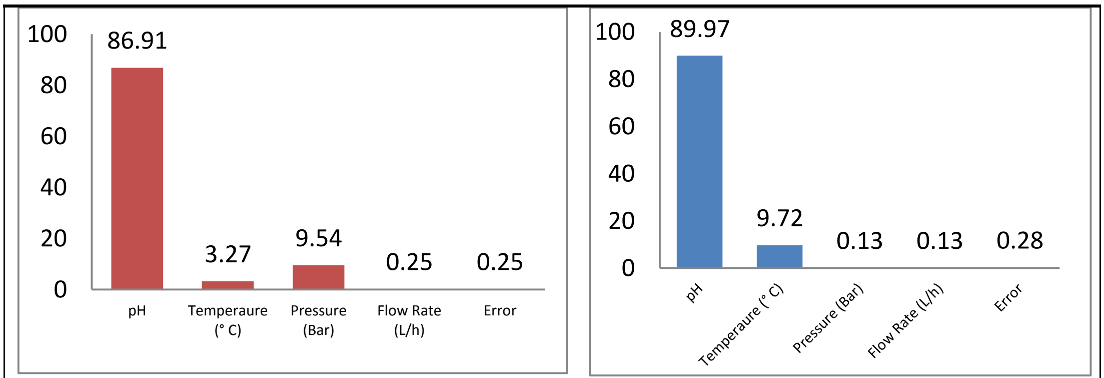


Figure 4.6: Percentage contribution of each factor to the response process

4.8.5 Statistical analysis of variance (ANOVA)

The significance effect of each factor on the filtration process was determined by ANOVA and F-test. In this current experiment with 4 factors at 3 levels, the degrees of freedom for each factor and error are 2 and 8, respectively. The critical F value at 95% confidence is 4.459 which is greater than the calculated value for pH, temperature, and Pressure and had a significant effect on Flux decline. While for COD rejection, all factors have significant effects on the response. The p-value also indicates the significance of various factors on the response, and from the table, it can be easily interpreted that pH, temperature, and Pressure have a significant impact on both Flux decline as well as COD rejection.

The percentage contribution (P%) quantitatively evaluates the effect of each selected parameter on the Response attributes. The percentage contribution of all factors on FD and COD rejection rate is depicted in Figure 4.15. From the plot, it can be easily seen that pH (P%= 86.91 and 89.97) was the most crucial factor for flux decline as well as COD rejection rate. The errors resulting from uncontrolled noise were below 50% that are not significant for reliable results.

Table 4.16: Experimental responses of the Taguchi Orthogonal Array L_9

Experiment No.	pH	Temp	Pressure (bar)	Flow rate (L/h)	Flux Decline (%)	COD rejection (%)
1	6	20	2	30	9.41	7.14
2	6	26	4	60	11.68	8.71
3	6	32	6	90	13.78	9.36
4	7	20	4	90	17.12	15.63
5	7	26	6	30	18.48	17.17
6	7	32	2	60	16.36	19.64
7	8	20	6	60	7.61	12.74
8	8	26	2	90	5.43	14.86
9	8	32	4	30	8.37	16.41

Table 4.17: ANOVA results for Flux Decline and COD rejection for each factor

	Source	DF	Seq SS	Adj SS	Adj MS	F	P
Flux Decline	pH	2	92.312	92.3116	46.1558	340.06	0.003
	Temp	2	3.477	3.4768	1.7384	12.81	0.072
	Pressure	2	10.147	10.1466	5.0733	37.38	0.026
	Flow rate	2	0.271	Pooled			
	Residual Error	2	0.271	0.2715	0.1357		
	Total	**8**	**106.206**	**66.5463**	**33.2732**	**630.54**	
COD rejection	pH	2	66.5463	7.1989	3.5994	68.21	0.000
	Temp	2	7.1989	Pooled		630.54	0.001
	Pressure	2	0.1069	Pooled		68.21	0.000
	Flow rate	2	0.1041	0.2111	0.0528	630.54	
	Residual Error	2	0.2111	66.5463	33.2732	68.21	
	Total	**8**	**73.9563**	**7.1989**	**3.5994**	**630.54**	

4.9 Optimal results for multi-response parametric optimization

4.9.1 Calculation of Grey Relational Grade

The data of the experiment was normalized for both Flux decline (%) and COD Rejection (%) called grey relational generations using Eq. (5). The grey relational coefficients were computed from the normalized data with the help of Equation 6 using the distinguishing coefficient as 0.5. The Grey Relational Grade (GRG) has been calculated with the help of Equation 7 using the grey relational coefficients. The obtained values of all the factors are presented in Table 4.17. These results after conversion into single grade have been utilized for optimizing the multi-response factors.

The effect of each selected parameter at different chosen levels is predicted from the value of GRG and shown in Figure 4.7. The mean GRG of all the parameters is shown in Table 4.18. The optimal working experimental combination of parameters was chosen based on higher mean GRG values. A higher GRG value implies a stronger positive relation between the reference sequence and hence enhanced performance of the membrane. Thus, the optimal parameter settings for multi-responses come out to be A3-B31-C1-D2. The variation of largest and smallest values of mean Grey Relation Grade for respective parameters were 0.401 for pH, 0.105 for Temp, 0.215 for pressure, and 0.033 for Flow rate respectively (Table 4.20). Thus, indicating the maximum influence of the pH on multi-responses followed by pressure and temp and then flow rate.

4.9.2 Result of ANOVA for Grey Relational Model

The ANOVA results considering grey relational grade for depicting the significance of the result are presented in Table 4.19. The value in the table indicates the level of significance for different selected process parameters on multi-responses. Based on results obtained from ANOVA, it can be concluded that pH and pressure are the most significant selected parameters affecting the membrane performance as it has a p-value lower than 0.05 at a 95% confidence level. Temperature and flow rate do not show any significant result on both response parameters.

4.9.3 Confirmation test of optimal Parameters

A confirmation Test was conducted to evaluate the improvement of the GRG parametric setting for the optimal parameters obtained in Ultra-filtration. GRG optimal parametric setting becomes 0.69234 which is close to the predicted value *i.e*. 0.88414. Table 4.20 reveals the grey relational grade of both responses *viz.* FD and COD rejection is improved significantly (0.1918) by setting the optimal parametric working combination. From this analysis, the selected parameters are optimized and the minimum value of flux decline and higher COD rejection is achieved through the Grey Relational-based Taguchi approach.

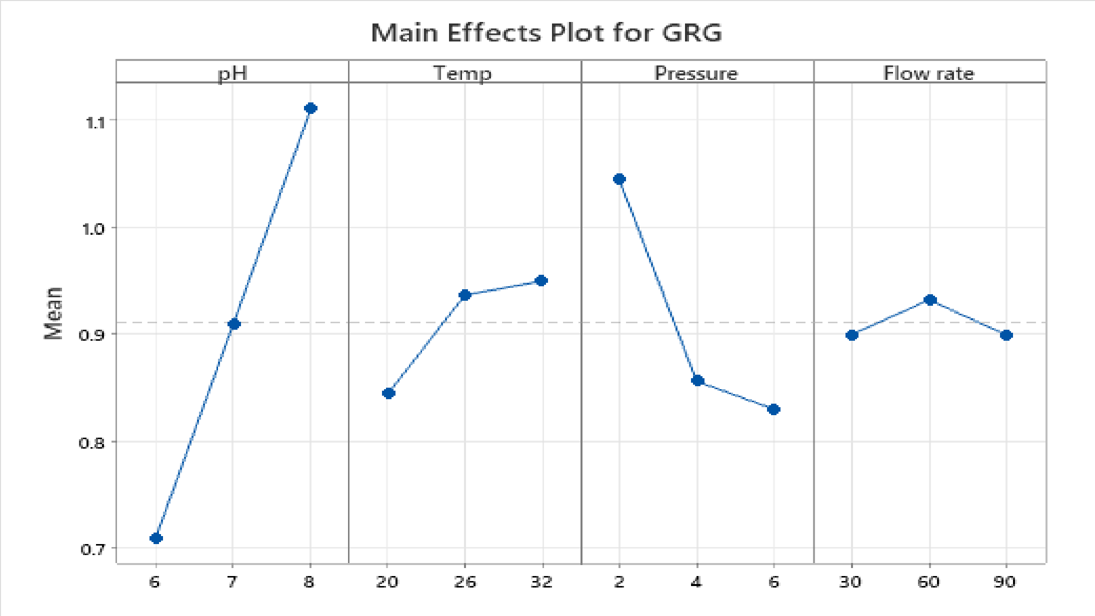


Figure 4.7: Mean effect curves for S/N ratios for Grey Relational Grade

Table 4.18: Grey relational coefficient and grey relational grade values for flux decline and COD Rejection

Exp. No.	Normalization		Deviation sequence		Grey Relation Coefficient		GRG	
	FD (%)	CODr (%)	FD (%)	CODr (%)	FD (%)	CODr (%)		Rank
1	0.695	0.000	0.305	1.000	1.000	0.537	0.768	7
2	0.521	0.126	0.479	0.874	0.822	0.586	0.704	8
3	0.360	0.178	0.640	0.822	0.706	0.609	0.657	9
4	0.104	0.679	0.896	0.321	0.577	0.981	0.779	6
5	0.000	0.802	1.000	0.198	0.537	1.154	0.845	5
6	0.162	1.000	0.838	0.000	0.602	1.610	1.106	2
7	0.833	0.450	0.167	0.550	1.207	0.767	0.987	4
8	1.000	0.618	0.000	0.382	1.610	0.912	1.261	1
9	0.775	0.742	0.225	0.258	1.110	1.061	1.086	3

*FD= Flux Decline; COD_r= COD Rejection

Table 4.19: Main effects on mean grey relational grade

Level	pH	Temp	Pressure	Flow rate
1	0.710	0.844636971	**1.045112253**	0.899755856
2	0.909971243	0.936802149	0.856125567	**0.93225311**
3	**1.111206891**	**0.949673174**	0.829874474	0.899103328
Delta	0.401	0.105036202	0.21523778	0.033149783
Rank	1	3	2	4

Total mean grey relational grade = 0.910

Table 4.20: Results of ANOVA on grey relational grade

Source	DF	Seq SS	Adj SS	Adj MS	F	P
pH	2	0.241530	0.241530	0.120765	112.06	0.009
Temp	2	0.019693	0.019693	0.009846	9.14	0.099
Pressure	2	0.082732	0.082732	0.041366	38.38	0.025
Flow rate	2	0.002155	Pooled			
Residual Error	2	0.002155	0.002155	0.001078		
Total	8	0.346111				

Table 4.21 Confirmation experiment

Parameter	Value	Level	Factor Setting	Predicted	Experimental
pH	**8**	**3**	**A3-B3-C1-D2**	**0.88414**	**0.69234**
Temp	**32**	**3**			
Pressure	**2**	**1**			
Flow rate	**60**	**2**			
Improvement in GRG = 0.1918					

Table 4.22: Permeate Quality (Process Performance) under optimum conditions

Parameter	Permeate Quality	Percent removal
COD (mg/L)	**179**	**17.10%**
BOD (mg/L)	**21**	**22.20%**
TDS (mg/L)	**1951**	**11.90%**
TSS (mg/L)	**0.0001**	**99.99%**
Color (PCU)	**108**	**73.70%**

4.9.4 Regression Analysis

Regression analysis for both the response Flux decline and COD decline was done with the help of multiple regression models as shown through Equation (a) and (b)

Flux Decline (%) = $21.7 - 2.24 X_1 + 0.121 X_2 + 0.72 X_3 + 0.0004 X_4$ ………… (a)

COD Rejection (%) = $-14.5 + 3.13 X_1 + 0.275 X_2 - 0.197 X_3 - 0.0048 X_4$ ……. (b)

Where X_1 represents pH, X_2 represents Temp, X_3 is Pressure and X_4 is Flow rate.

4.10 Performance of Membrane Filtration Unit for Bindal Paper Mills (Agro-Based Industry)

Effluent samples collected after secondary clarifiers were passed through two different combinations *viz*. (1). Pre-filtration and UF membrane installed in series, (2) Pre-filtration, UF Membrane and RO Membrane arranged in series.

4.10.1 Effect of Membrane Filtration on pH value in Agro-based industry

pH is an important parameter to ensure the proper quality of the wastewater. Extreme levels and presence of particulate matter, and the accumulation of toxic chemicals are the major causes of pH change in wastewater. As shown in Figure 4.8, the pH value of the effluent of the secondary clarifier has an average value of 7.46±0.44. The pH of the wastewater generated is closely related to the microbial activity that impacts the secondary treatment process of the activated sludge process. The pH of the effluent was reduced to 7.19 ±0.12 with the combination of pre-filter and Ultra-filtration. On the other hand, the pH was further reduced to 5.74±0.51 with the addition of the RO membrane. The alkalinity of the water relies upon the presence of the bicarbonates of calcium and magnesium. After removal of these minerals in reverse osmosis the hydroxyl ions react with the CO_2 present in the air and form carbonic acids hence lowering the pH of the water.

4.10.2 Effect of Membrane Filtration on TSS value in Agro-based industry

Total Suspended solids are the key factor in monitoring the quality of the water and are used in the assessment of food microbe ration and also provide information reading the removal efficiency and membrane fouling rates (Claudia et al., 2018). The effluent collected from the secondary clarifier was already low with a TSS value of 56±0.96, indicating that the secondary treatment itself is sufficient to reduce the TSS load. The change in the TSS level with both combinations of membrane filters is shown in Figure 4.9. Both the combinations of membrane filtration configurations were able to eliminate the TSS. However, the application of the RO process immediately after Ultrafiltration can cause scaling to the RO membrane due to the presence of dissolved minerals like calcium carbonates, phosphates, and sulfates (Lu et al., 2020).

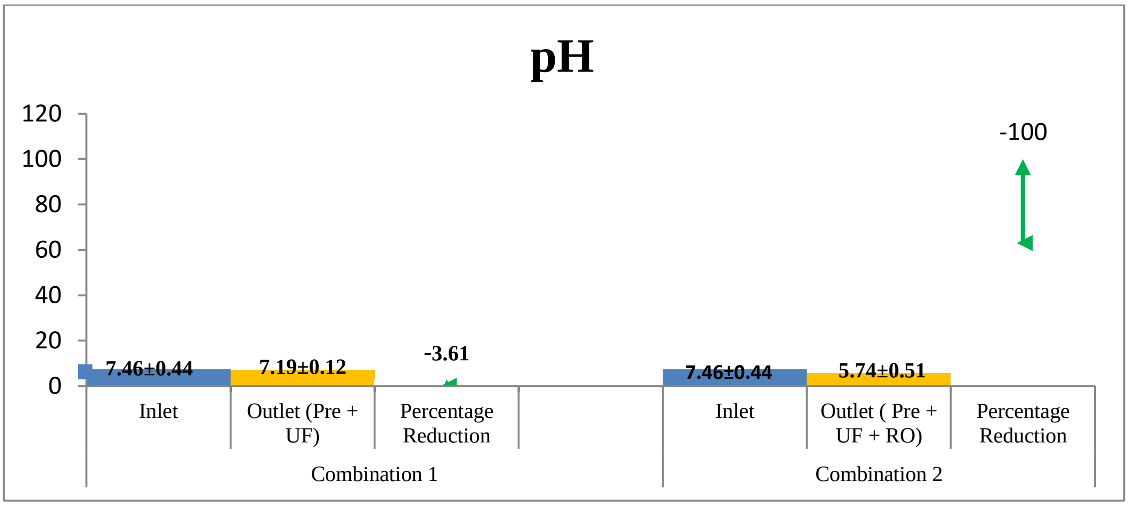


Figure 4.8: Effect of Different Treatment Setups on the pH Rejection in Agro-based Industry

So, adding the pre-treatment filter is an advantageous step. Many researchers have observed the total suspended percentage removal of 93.3% - 95.1% by using the membrane bioreactor (Komesli et al., 2015; Maqbool et al., 2014). On the other hand, the conventional activated sludge process only has a removal efficiency of 60-70%.

4.10.3 Effect of Membrane Filtration on TDS value in Agro-based industry

The Total Dissolved Solids (TDS) measures the concentration of all inorganic and organic elements dissolved in water. The effluent coming from the secondary clarifier has a TDS value of 2215±56.45 mg/L. However, the removal of TDS is not satisfactory with pre-treatment and UF, as only 7.7% (2215±56.45 mg/L to 1951±41.23 mg/L) of TDS could be reduced. The combination of the RO-UF filtration after pre-filtration reduced 88.26% of TDS (Figure 4.10). Similarly, Yordanov, 2010 and Oh et al., 2018 on Wastewater from the poultry slaughterhouse and Grey water respectively, the TSS rejection of approximately 98% and 100% has been achieved by Ultrafiltration.

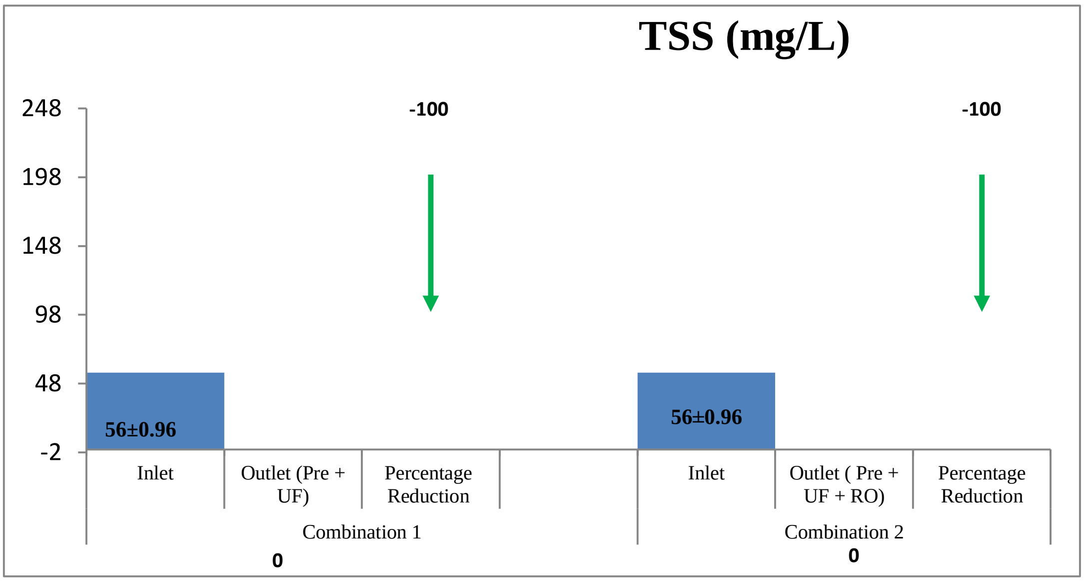


Figure 4.9: Effect of Different treatment setups on the TSS rejection in Agro-based Industry

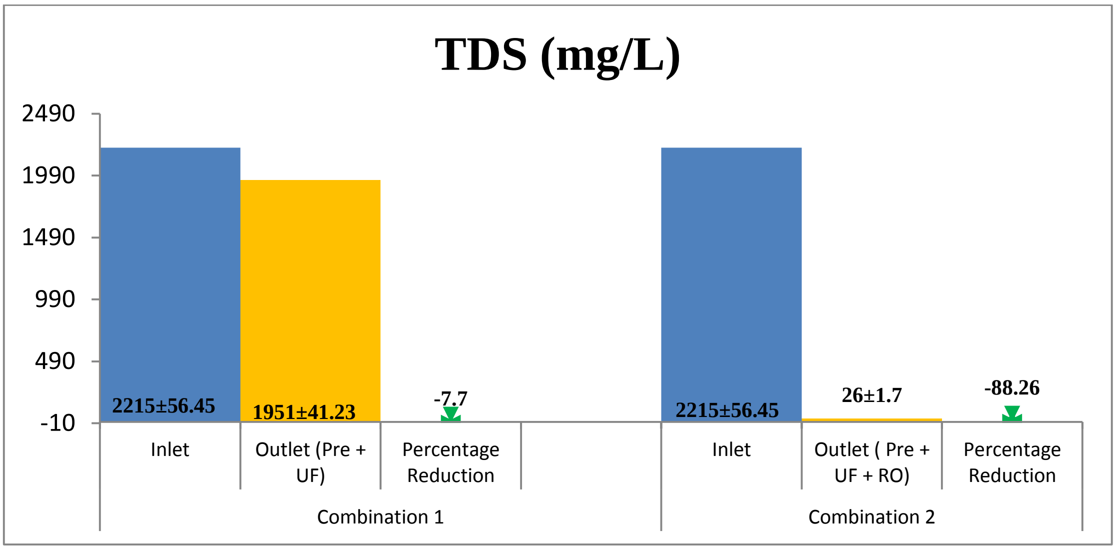


Figure 4.10: Effect of Different treatment setups on the TDS rejection in Agro-based Industry

4.10.4 Effect of Membrane Filtration on COD value in Agro-based industry

The Chemical Oxygen Demand (COD) value of the secondary clarifier has an average value of 216±9.11 mg/L indicating the inefficiency of the conventional treatment method to remove the load from wastewater. To determine the COD value, the reduction potential obtained by pre-filtration followed by UF was 17.12% (216±9.11 mg/L to 179±10.76 mg/L). However, the addition of RO membrane along with pre-filtration and UF appeared to reduce 100% of COD from the effluent (Figure 4.11 and 4.12). These results are in agreement with the study conducted by Sun et al., 2015 on Phenolic wastewater generated from paper mills, and with the application of a combination of UF-NF/RO a reduction of 95.5% in COD has been observed. Al-Shammari et al., 2018 also observed a percentage COD reduction of 97% from the wastewater using a membrane bioreactor. Similarly, Jadhao and Dawande 2012 observed a reduction of approximately 97.00% COD achieved by UF from the hospital wastewater. The use of electrochemical oxidation along with reverse osmosis has reduced COD by 98.5% from the wastewater generated by the yarn fabric industry (Bouchareb et al., 2021 Khan et al., 2022).

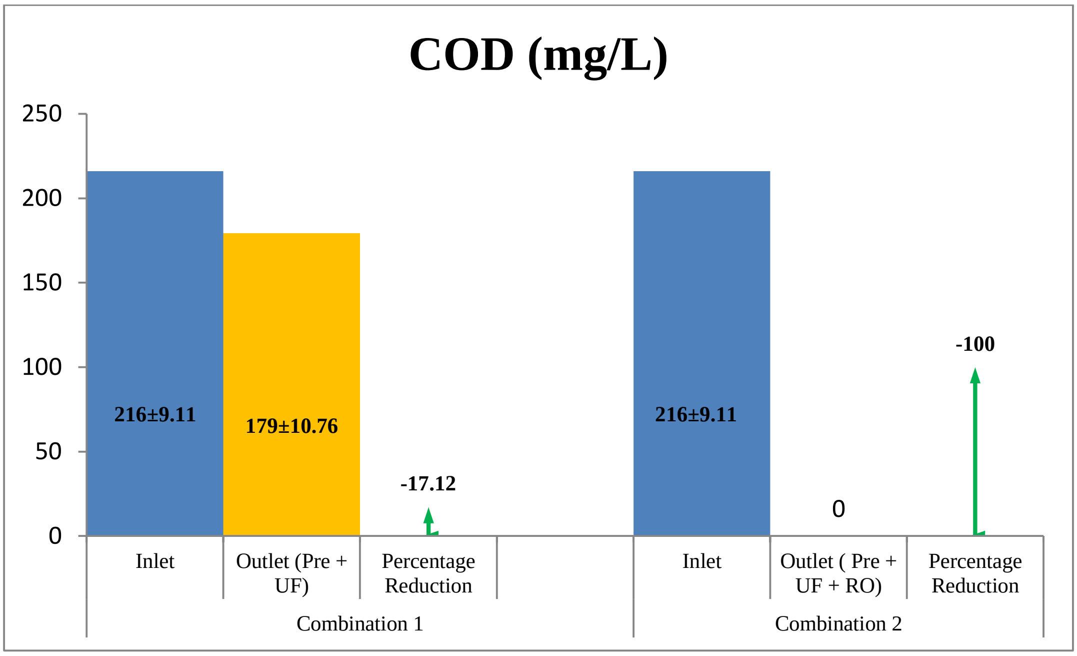


Figure 4.11: Effect of Different treatment setups on the COD rejection in Agro-based Industry

4.10.5 Effect of Membrane Filtration on BOD value in Agro-based industry

Biological oxygen Demand (BOD) refers to the amount of oxygen required to decompose the organic contaminant from wastewater. The paper and pulp industry wastewater generally has very high BOD values due to the addition of organic matter in the process. The effluent from the secondary clarifier has an average BOD value of 27±0.56mg/L. The BOD reduction potential using pre-filtration and UF was 22% (27±0.56 mg/L to 21±0.71 mg/L). However, the addition of RO membrane along with pre-filtration and UF appeared to reduce 100% of BOD from the effluent (Figure 4.12). However, the high organic matter in wastewater has a greater possibility of clogging the membrane which needs to be backwashed after regular intervals to maintain the flux. Yordanov, 2010 also observed a reduction of more than 94% in BOD of the wastewater from the slaughterhouse with the application of Ultrafiltration. Other studies have also documented a BOD removal rate of more than 96% (Rodriguez-H. et al., 2015).

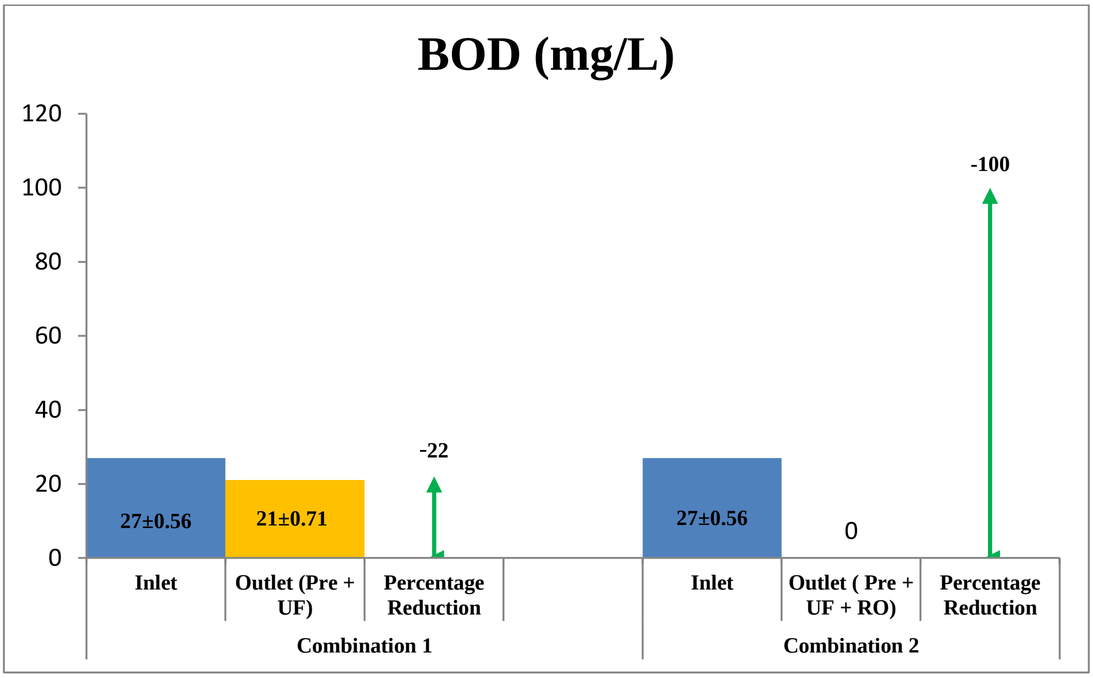


Figure 4.12: Effect of Different treatment setups on the BOD Rejection in Agro-based Industry

4.10.6 Effect of Membrane Filtration on Color in Agro-based Industry

Pre-filtration and UF in series could reduce color from 412±21.10 PCU to 108±10.2 PCU *i.e.* by 73.78% (Figure 4.13). Similar results have been reported by Cebeci., in 2017 where the use of Nanofiltration alone has removed the color by 98% from the wastewater generated from the textile industry that was rich in methylene blue. In the study conducted by Bouchareb et al., in 2021, the use of reverse osmosis in addition to electrochemical oxidation in a yarn fabric wastewater generation has completely removed the color indicating that coupling of membrane along with some advanced techniques can help to overcome the problem of water treatment.

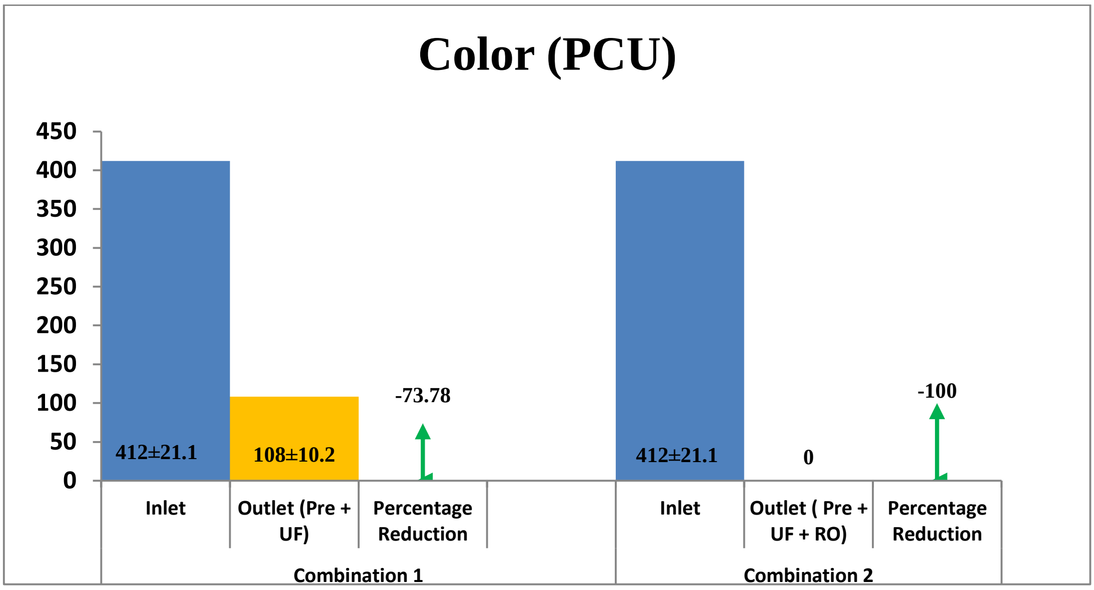


Figure 4.13: Effect of Different Treatment Setups on the Color Rejection in Agro-based Industry

4.10.7 Effect of Membrane Filtration on Lignin in Agro based industry

Lignin is the primary component that contributes to color addition in the paper industry. When lignin removal from effluent discharge is the aim, the pre-filtration followed by UF membrane is a significant step that reduces 83% (126±7.71 mg/L to 22±1.01 mg/L) of lignin. The additional RO membrane attached eliminates 100% lignin content from the effluent denoted by Figure 4.14. Ellouze et al., 2012 show the use of the NF technique to treat textile wastewater. Also, the reported use of UF alone rejected 98% of the dye content from dye industry wastewater (Lin et al., 2016).

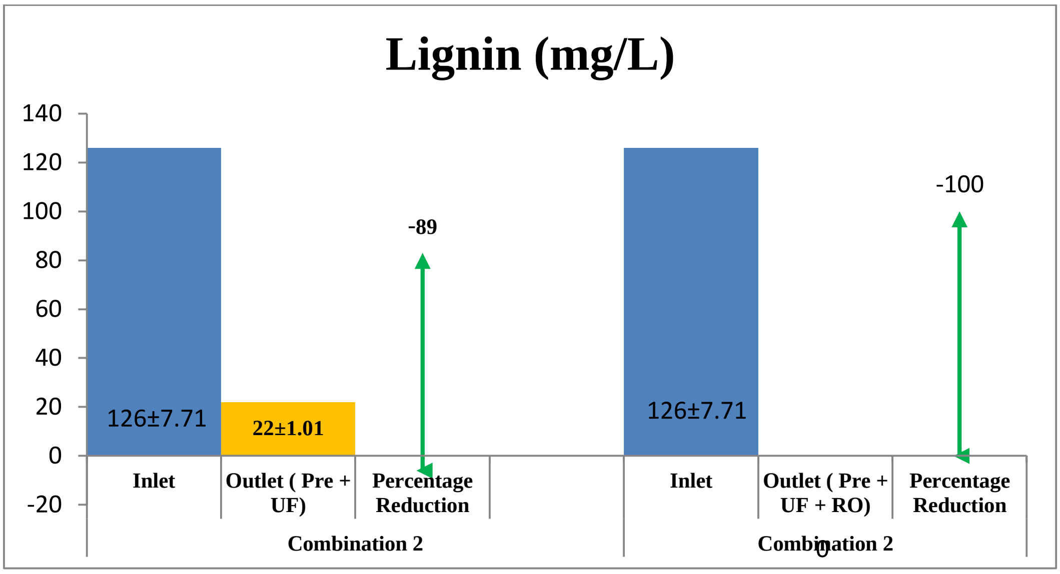


Figure 4.14: Effect of Different treatment setups on the Lignin Removal in Agro-based Industry

In a study conducted by Singh et al., 2023, the use of forward osmosis only rejected around 90% of the lignin content from the pulp and paper wastewater. In a similar study on the extraction of lignin from the wastewater, a plant of 60 m^3/h extracted 12 tons of lignin and 825 Kg of lignans per month using an Ultrafiltration membrane of 150k Da (Gambier et al., 2020 and Amar et al., 2017).

4.10.8 Effect of Membrane Filtration on Conductivity in Agro-based Industry

Conductivity is an important parameter in the determination of water quality as it states the content of inorganic minerals available in the water. The effluent coming out of the secondary clarifier has conductivity with an average of 1365±81.21 mg/L. The reduction potential via pre-filtration followed by UF was 31% (1365±81.21 mg/L to 942.9±79.37 mg/L). However, the addition of RO membrane along with pre-filtration and UF further reduced the conductivity to 98.16% from the effluent (Figure 4.15). The results obtained from a study conducted by Bouchareb et al., in 2021, the use of reverse osmosis membrane has decreased the conductivity by 97.1% in comparison to the 31.2% removal by electrochemical oxidation. Similarly, Cebeci in 2017 reported the removal of 95% conductivity from 1225µS to 223 µS in textile industry wastewater using Nanofiltration.

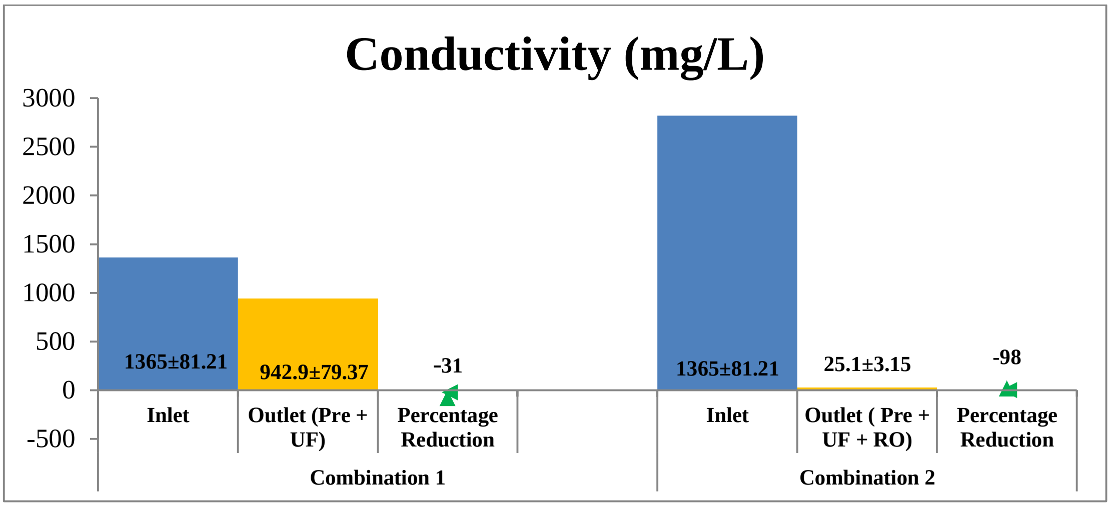


Figure 4.15: Effect of Different Treatment Setups on the Conductivity in Agro-based Industry

4.5.9 Effect of Membrane Filtration on Potassium and sodium content in Agro based industry

The use of various chemicals in the pulping process increases the content of sodium and potassium in the mill wastewater. The effluent coming out of the secondary clarifier of the effluent treatment plant has an average respective value of 4.2±0.17 mg/L and 28±2.1 mg/L for potassium and sodium. For consideration of Potassium and sodium, the reported reduction potential by pre-filtration and UF was 14.28% (4.2±0.17 mg/L to 3.6±1.1 mg/L) and 19% (28±2.1 mg/L to 22.70±1.72 mg/L), respectively. However, the addition of RO membrane along with pre-filtration and UF further reduced the Potassium and Sodium ions to 98% and 87% from the effluent (Figure 4.16 and 4.17). Reverse osmosis is more effective than Ultra-filtration in the removal of ions like Potassium and Sodium from the wastewater.

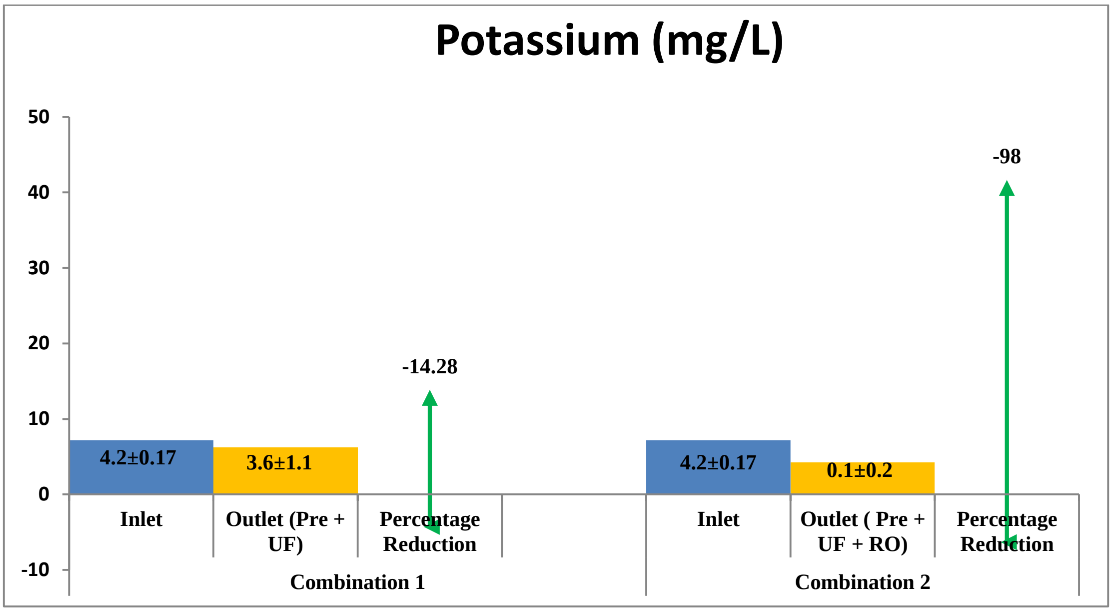


Figure 4.16: Effect of Different Treatment Setups on Removal of Potassium in Agro-based Industry

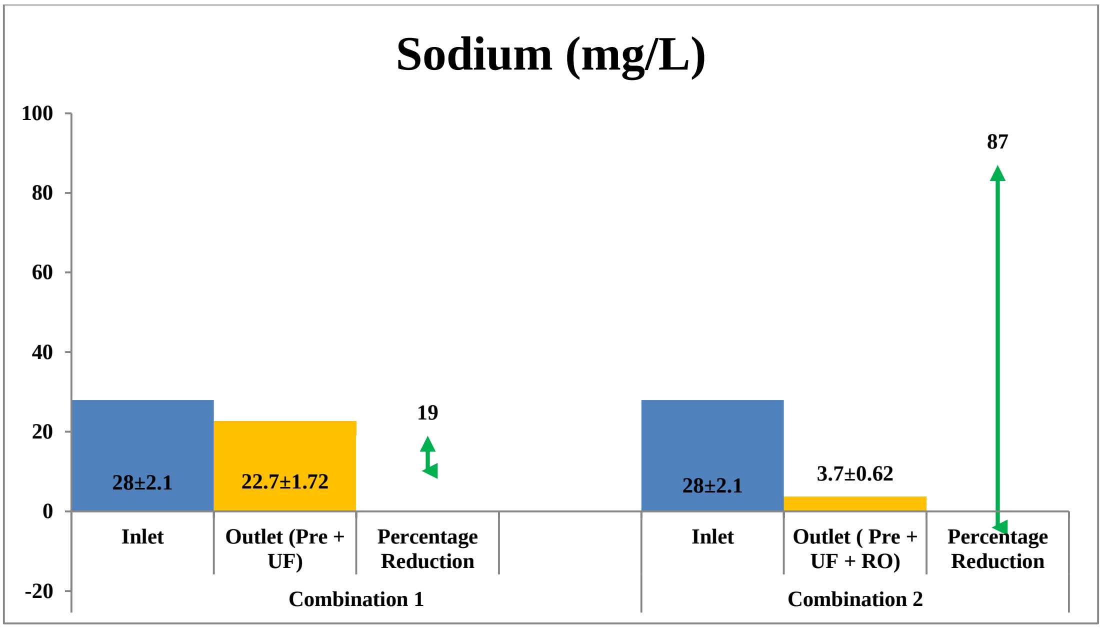


Figure 4.17: Effect of Different Treatment Setups on Removal of Sodium in Agro-based Industry

The results obtained from our study are in good agreement with various previous studies on removing pollutants from wastewater using both RO and UF membranes. There has been a significant decrease ($P<0.05$) in the concentration of all pollution in both treatment methods (Table 4.22). Membrane filtration at low pressure is reported to be best for bleach effluent treatment in pulp and paper mills (Shukla et. al., 2013

Lindströ and Nordin, 1976). Likewise, it has also been reported that the performance of using RO in the reduction of pulp and paper industry effluent is higher than UF and nanofiltration (Shukla et. al., 2010). In addition, Li et.al.2004a have recommended the combined RO-UF membrane process for the treatment of wastewater.

4.5.10 Assessment of the use of water for recycling in the plant after membrane treatment in Agro based industry

The reclamation of wastewater for water recovery can easily help to achieve the zero-discharge level in the paper and pulp industry. Several technologies such as adsorption technology (Alothman and Wabaidur, 2019), advanced oxidation (Oturan and Aaron, 2014), and activated charcoal (Luukkonen et al., 2019). However, the quality of water reclaimed cannot be reused in the industry. Membrane technology especially Ultrafiltration and Reverse Osmosis has given promising results to reclaim m water from municipal wastewater treatment plants. The permeate of both combinations was compared with the feed water characteristics required by the pulp and paper industry listed in Table 4.23. The effluent from the secondary clarifier cannot be reused for the production of paper since none of the parameters fall under the prescribed tolerance limits. The pre-filter used in the process protects the Ultrafiltration used downstream from the large suspended solids. Microfiltration units have been applied in many studies (Shang et al., 2011 and Holman and Ohlinger, 2007) but Ultrafiltration has the distinct advantage since it allows choosing the pore size from a range of 0.1- 0.001µm as per the requirement, moreover, it removes soluble organic particles, along with the pathogenic bacteria, thus produce a permeating quality of TDS below 2000 mg/L that lies close to the legal standards for irrigation water (CGWC, 1991; Oron et al., 2006). This combination of unit operation has been applied in full-scale wastewater treatment plant effluent reclamation (Hamoda et al., 2009). The application of pre-treatment and ultra-filtration has, however, reduced the pollution load but except TSS none of the parameters lies in the tolerance regime. However, the permeate from the series combination of pretreatment, Ultrafiltration, and RO unit has been able to achieve the tolerance limits for Color, TDS, and TSS to be reused in the production of all the grades of paper.

Table 4.23: Change in the various water parameters with different treatment membrane setups in the Agro-Based Industry

	TSS (mg/L)	TDS (mg/L)	COD (mg/L)	BOD (mg/L)	Color (PCU)	Lignin (mg/L)	pH	Conductivity (µs)	Potassium (mg/L)	Na (mg/L)
Inlet (Secondary Clarifier)	56±0.96	2215±88.26	216±9.11	27±0.56	412±21.1	126±7.71	7.46±0.44	1365±81.21	4.2±0.17	28±2.1
Outlet (Pretreatment + UF)	0*	1951±56.45*	179±10.76*	21±0.71*	108±10.2*	22±1.01*	7.19±0.12*	942.9±79.37*	3.6±1.1*	22.7±1.72*
Outlet (Pretreatment + UF + RO)	0*	26±1.70*	0*	0*	0*	0*	5.74±0*	25.1±3.15*	0.1±0*	3.7±0.62*

*Significant at P<0.05, Two-tailed independent t-test

Table 4.24: Water Quality requirement by Paper and pulp industries in India

	Effluent from different processes			**Tolerance for water for pulp and paper industry (BIS)**			
Water quality parameter	**Secondary Clarifier Effluent**	**Permeate from pretreatment and Ultrafiltration unit**	**Permeate from pretreatment, Ultrafiltration, and RO unit**	**Ground Wood paper**	**Kraft Paper Bleached**	**Soda and Sulphite Paper**	**High-Grade Paper**
Color	615	176	0	20	15	10	5
TDS	1933	1784	0	500	300	300	300
TSS	36	0	0	25	25	25	10
COD	183	146	0	NS	NS	NS	NS

NS- Not specified

4.10.11 Statistical analysis of the various treatment setups in Bindal Paper Mill Ltd

The correlation of all the pollution parameters is shown in Table 4.24. A maximum correlation of 0.96 has been observed between color and sodium content in the effluent stream. The least correlation has been observed between Total Solid solids and pH, an indication that there is no correlation between the TSS and the pH of the Wastewater.

The result of hierarchical cluster analysis of all pollution parameters from the effluent of all four sources viz. primary clarifier, secondary clarifier, Pretreatment + Ultrafiltration, and Pretreatment + Ultrafiltration + Reverse Osmosis is shown in figure 4.18. The dendrogram shows two major clusters. Cluster 1 involves the effluent generated from the primary clarifier and secondary clarifier indicating the same quality of effluent. Cluster 2 involves the effluent coming out from the Pretreatment + Ultrafiltration and Pretreatment + Ultrafiltration + Reverse Osmosis indicating a pronounced reduction in the pollution load using membrane technology.

Table 4.25: Correlation Matrix of the Water Quality Parameters of Bindal Pvt Ltd

	pH	TSS	TDS	COD	BOD	Color	Lignin	Conductivity	Na+	K+
pH	1									
TSS	0.23287	1								
TDS	0.95995	0.491409	1							
COD	0.56988	0.849759	0.765281	1						
BOD	0.53305	0.782516	0.719133	0.987012	1					
Color	0.67196	0.758373	0.825665	0.972603	0.964967	1				
Lignin	0.58314	0.733311	0.748814	0.976303	0.994453	0.978302	1			
Conductivity	0.86647	0.533339	0.919486	0.782165	0.747009	0.891708	0.793986	1		
Na+	0.65657	0.751009	0.806422	0.954274	0.945415	0.99582	0.963655	0.906025	1	
K+	0.52294	0.169634	0.527726	0.231962	0.163045	0.138355	0.128117	0.160025	0.064565	1

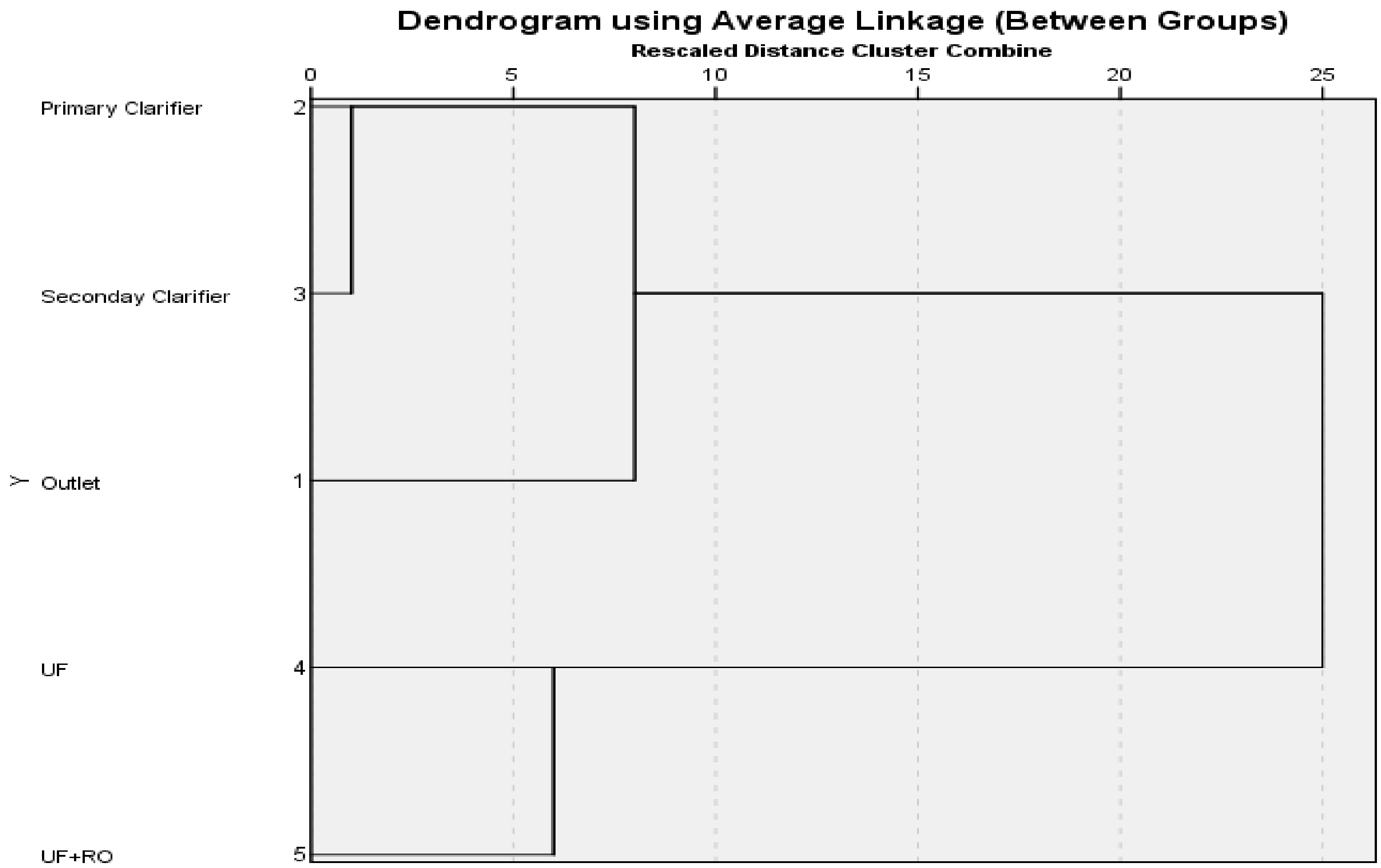


Figure 4.18: Dendrogram showing hierarchical cluster analysis of effluent of different treatments for Bindal Paper Mills

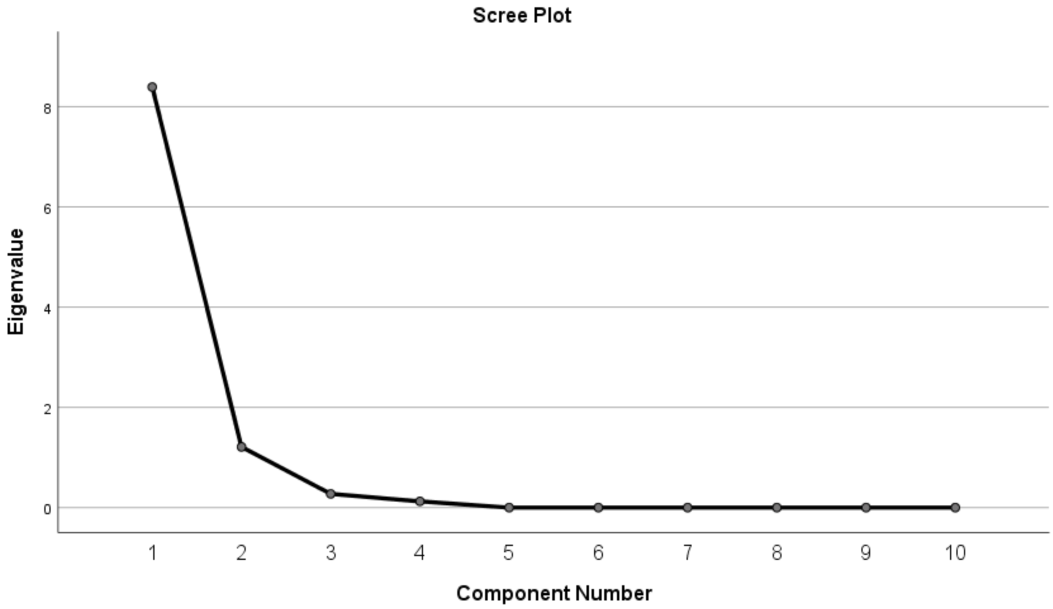


Figure 4.19: Screen Plot of the component analysis for Bindal Paper Mills Ltd

The screen plot of Bindal Paper Pvt Ltd is shown in Figure 4.19. The screen plot depicts the eigenvalue for each individual Principal Component. Components 1 and 2 having Eigenvalues of more than one are selected. The elbow of the curve shows only two components falling above value one.

The adequate number of clusters is determined by the agglomeration coefficient as shown in table 4.25. The agglomeration schedule was obtained at the various clustering stages hierarchically by using various parameters along with different treatment methods. The optimum number of clusters determined is 2 and the variation in agglomeration coefficient shows heterogeneous cases.

Table 4.26: Agglomeration coefficient of cluster analysis for Bindal Paper Pvt Ltd

Stage	Cluster Combined		Coefficients
	Cluster 1	Cluster 2	
1	2	3	2115457.060
2	4	5	4596155.752
3	1	2	5612867.660
4	1	4	13679932.985

4.11 Performance of Membrane Filtration unit for Star Paper Ltd- a wood-based Industry

Effluent samples after secondary clarifiers were passed through two different combinations *viz*. (1). Pre-filtration and UF membrane installed in series, (2) Pre-filtration, UF Membrane and RO Membrane in series.

4.11.1 Effect of Membrane Filtration on pH in wood-based industry

The pH value has a great impact on the activity of the microorganisms which in turn affects the composition of the enzymes and hence dissociation of the organic matter from the waste water. As denoted in Figure 4.20, the outflow water pH from the secondary clarifier has a pH value of 8.12±0.34. Alkaline conditions have a strong impact on the membrane as higher pH negatively charges the membrane, hence impacting the membrane performance (Kimani et al., 2022; Chen et al., 2023). The feed water pH was reduced to 7.89 ±0.12 by integrating pre-filter and UF. On the other hand, the pH was further reduced to 5.12±0.71 with the addition of RO membrane due to the removal of all minerals and absorption of CO_2 from the atmosphere (Vigneshwaran et al., 2021).

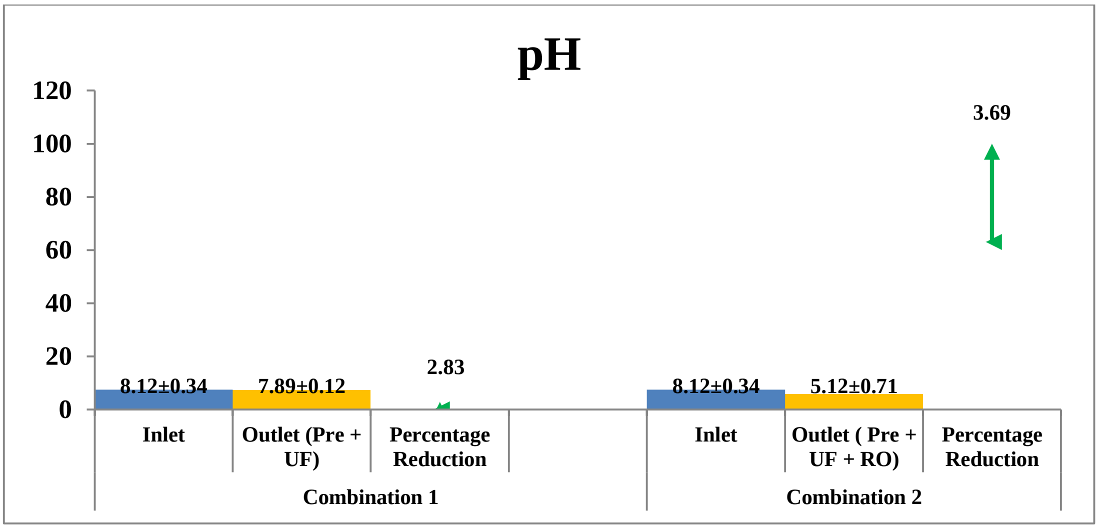


Figure 4.20: Effect of Different Treatment Setups on the pH rejection in the wood-based industry

4.11.2 Effect of Membrane Filtration on TSS in wood-based industry

Total Suspended solids (TSS) have been recognized as the most valuable data for membrane technology and provide information about nutrient recovery from wastewater. The secondary clarifier effluent has an average TSS value of 36±1.12 mg/L. The change in the TSS level with both combinations is shown in Figure 4.21, both the combinations of membrane filtration configuration were able to entirely reduce the TSS. Similar results have been obtained by Czuba et al., 2021 in a pilot scale experiment that has been used for the recovery of water and nutrients from the secondary effluent of the wastewater treatment plant, and a reduction of 80% in TSS value has been recorded.

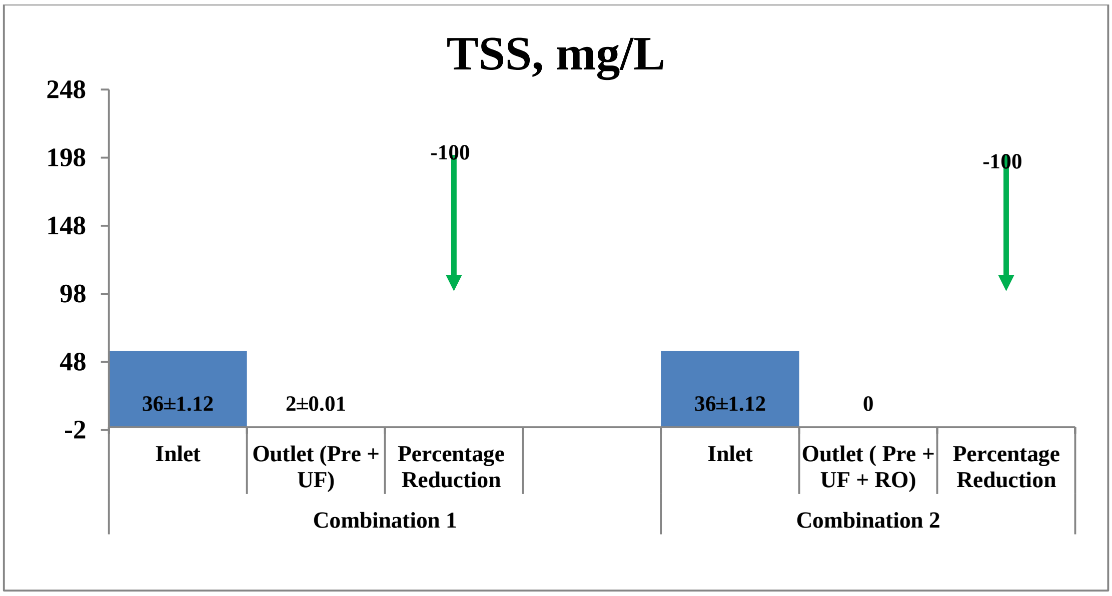


Figure 4.21: Effect of Different treatment setups on the TSS rejection in wood-based industry

4.11.3 Effect of Membrane Filtration on TDS in wood-based industry

The effluent of the secondary clarifier has an average TDS value of 1933±87.46 mg/L. However, the removal of TDS is not satisfactory with pre-treatment and UF, as only 7.7% (1933±87.46 mg/L to 1784±46.23 mg/L) of TDS could be reduced. The combination of two membranes namely RO and UF along with pre-filtration reduces 87.46% of TDS denoted in Figure 4.22. Yordanov, 2010 and Oh et al., 2016 also observe similar type of results when they treat poultry slaughterhouses and Grey water. The use of UF reduced the TSS by 98% to 100%.

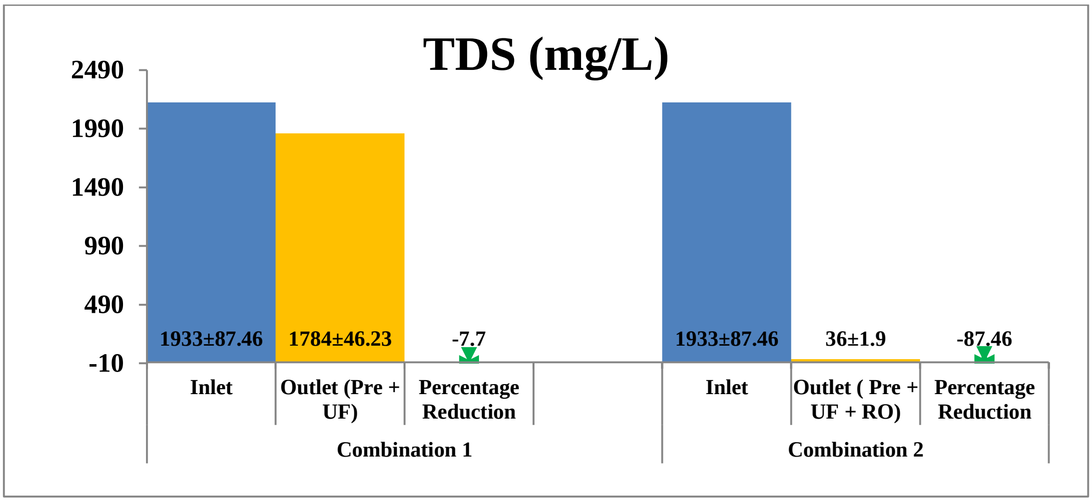


Figure 4.22: Effect of Different treatment setups on the TDS rejection in wood-based industry

4.11.4 Effect of Membrane Filtration on COD in wood-based industry

For COD and BOD, the reduction potential by using pre-filtration followed by UF was 20.2% (183±11.09 mg/L to 146±9.76 mg/L) respectively. However, the addition of the RO membrane along with pre-filtration and UF appeared to reduce 100% of COD and BOD from the effluent (Figures 4.23 and 4.24). These results are in agreement with the study conducted by Sun et al., 2015 on Phenolic wastewater generated from paper mills, and with the application of a combination of UF-NF/RO a reduction of 95.5% in COD has been observed. Yordanov, 2010 also observed a reduction of more than 94% in BOD of the wastewater from the slaughterhouse with the application of Ultrafiltration. In a study conducted by Xu et al., 2019, the use of coagulation as pretreatment before Ultrafiltration for the treatment of secondary effluent from a recycled paper mill resulted in 91.81% COD rejection.

4.11.5 Effect of Membrane Filtration on BOD in wood-based industry

The biological Oxygen Demand of the effluent generated from the secondary clarifier has an average value of 20±0.79 mg/L. The pre-filtration and UF in combination have reduced the BOD by 30% (20±0.79 mg/L to 14±0.81 mg/L).

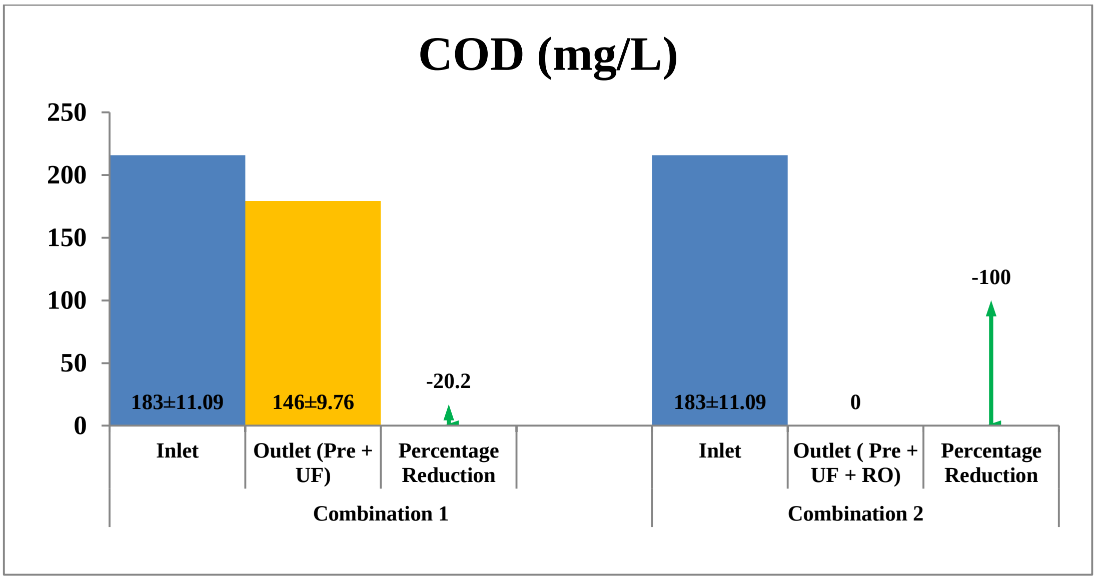


Figure 4.23: Effect of Different treatment setups on the COD rejection in wood-based industry

However, the addition of RO membrane along with pre-filtration and UF appeared to reduce 100% of BOD from the effluent. Similar results were observed by Jadhao and Dawande in 2012, where a reduction of approximately 97.00% was achieved by UF from the hospital wastewater. Moreover, the addition of RO further eliminated 99.00% of the BOD. Butze et al., 2020 have also achieved a BOD reduction of 94% from the sewage wastewater using the membrane technology.

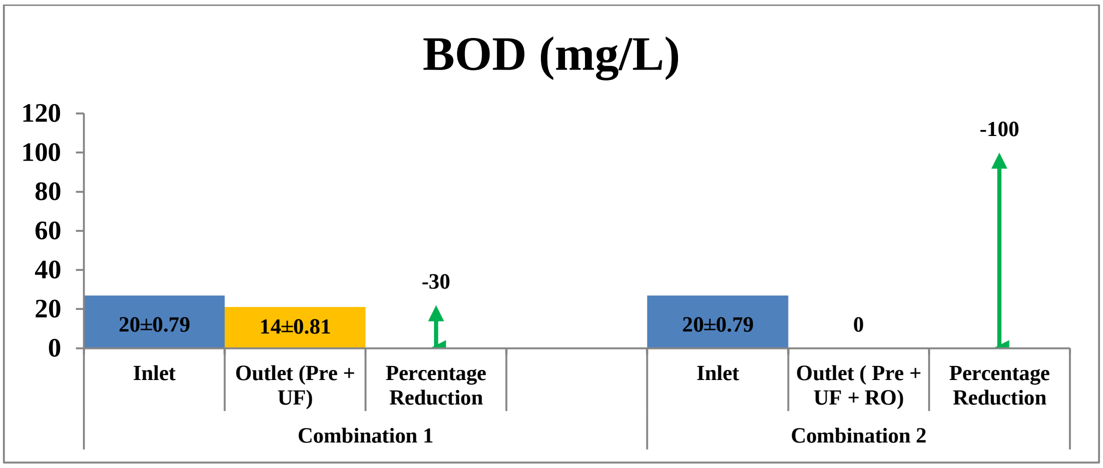


Figure 4.24: Effect of Different treatment setups on the BOD rejection in wood based industry

4.11.6 Effect of Membrane Filtration on Color in wood-based industry

Lignin is the main component of color addition in the paper industry. The effluent generated from the secondary clarifier has a color with an average value of 615±23.21 PCU. Pre-filtration and UF in series could reduce color from 615±23.21 PCU to 176±11.2 PCU *i.e.* by 71.5% (Figure 4.25). However, the addition of Reverse osmosis in series has completely removed the color component from the feed water. Ellouze et al., 2012 reported that the nano-filtration technique used to treat textile industry effluent led to 100% color reduction of effluent. Also, the use of UF alone led to 98% synthetic dye rejection from wastewater (Lin et al., 2016).

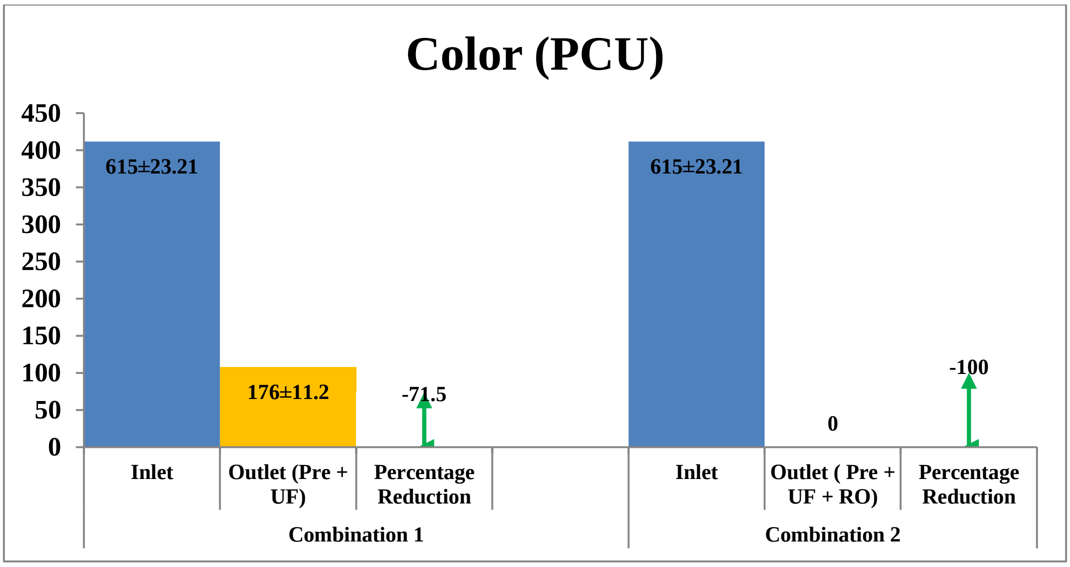


Figure 4.25: Effect of Different treatment setups on the Color rejection in wood-based industry

4.11.7 Effect of Membrane Filtration on Lignin in wood-based industry

The deep and dark coloration of wastewater discharge from the mill is caused by lignin and its derivative. The industrial lignin has a diverse complex structure due to various chemical treatments making them recalcitrant. Hence, its removal from wastewater is crucial which otherwise causes detrimental effects on aquatic life. When the elimination of lignin is required from the wastewater effluent of the pulp and paper industry, the pre-filtration followed by UF performed well as it reduced 89% (168±8.78 mg/L to 18±1.11 mg/L) of lignin. When we attach the additional RO membrane, the entire lignin content (100%) is eliminated from the effluent denoted by Figure 4.26. In a study conducted by Ebrahimi et al., in 2015, the use of two-stage MF/UF of MWCO values ranging from 1kDa to 20 kDa has achieved 73% rejection of residual lignin. Similarly, in a study performed by Neves in 2017, the ultra-filtration removed approximately 82% of lignin from the paper and pulp manufacturing and processing industry wastewater. The utilization of 1 KDa Nanofiltration has removed 70% of lignin from the process water of thermochemical pulp plants (Courbalay et al., 2021).

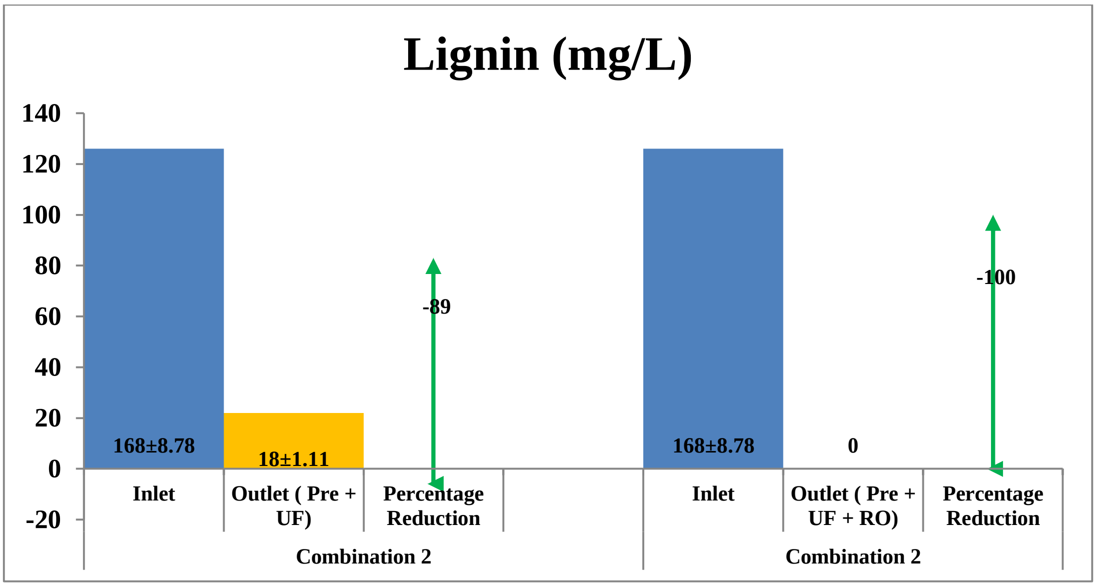


Figure 4.26: Effect of Different treatment setups on the Lignin removal in wood-based industry

4.11.8 Effect of Membrane Filtration on Conductivity in wood-based industry

Conductivity measures the ability of the water to pass an electrical current. It is affected by the presence of inorganic dissolved solids such as anions (chloride, nitrate, sulfates) or cations (sodium, magnesium, calcium, etc.). In terms of conductivity, the reduction potential by use of pre-filtration followed by UF was found to be 24% (2820±91.23 mg/L to 2142.9±84.35 mg/L). However, the addition of RO membrane along with pre-filtration and UF further reduced it to 98% from the effluent (Figure 4.27).

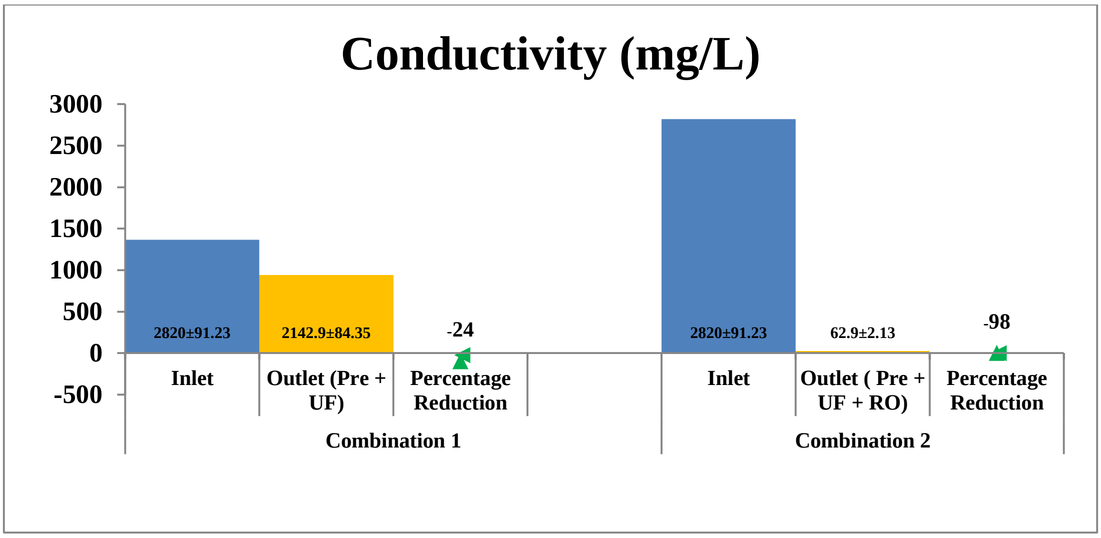


Figure 4.27: Effect of Different treatment setups on the Conductivity in wood-based industry

4.11.9 Effect of Membrane Filtration on Potassium and Sodium content in wood-based industry

The effluent generated from the secondary clarifier effluent has high amounts of sodium and potassium with an average of 71±2.3mg/L and 8.8±0.27mg/L respectively. The presence of excessive sodium and potassium in wastewater may lead to an increase in the salinity and reusing the same water in the mill process may lead to damage in the machinery. When Potassium and sodium are considered as parameters, the reduction potential when pre-filtration was used along with UF was 18.18% (8.8±0.27 mg/L to 7.2±0.08 mg/L) and 8.8% (71±2.3 mg/L to 65.3±1.82 mg/L), respectively. However, the addition of RO membrane along with pre-filtration and UF further reduced the Potassium and Sodium ions to 98% and 91% from the effluent (Figure 4.28 and 4.29). Reverse osmosis is more effective than Ultra-filtration in the removal of ions like Potassium and Sodium from the wastewater. In a study conducted by Fatehizadeh et al., 2018, the use of nano-filtration effectively reduced the concentration of Na^{+} and K^{+} in permeate from 67.1mg/L to 51.8 mg/L and from 2.51 mg/L to 1.32 mg/L from the brackish water.

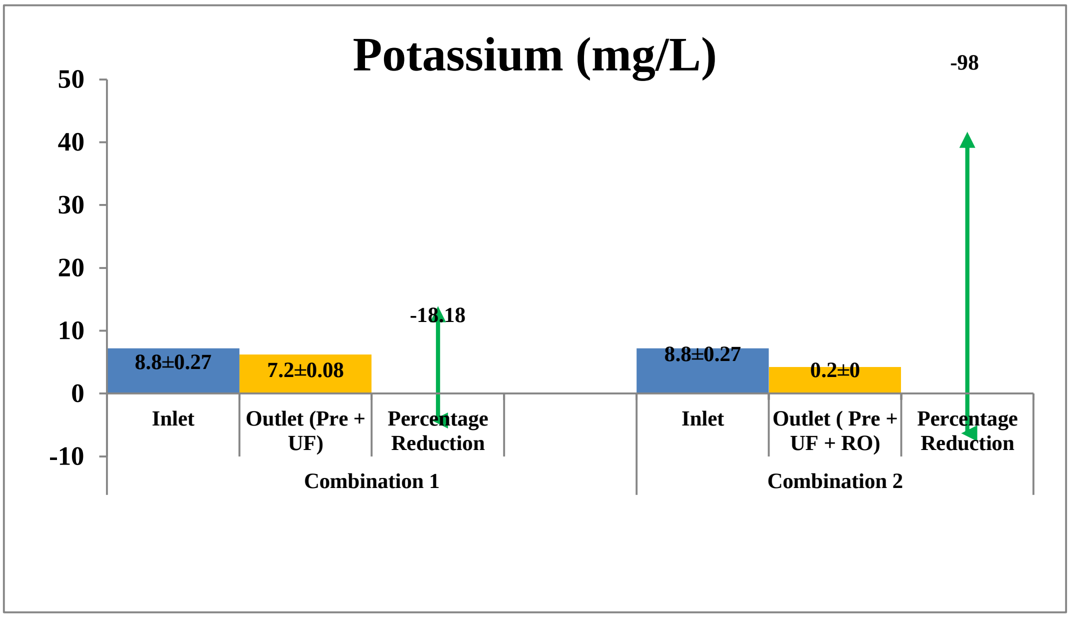


Figure 4.28: Effect of Different treatment setups on removal of Potassium in wood-based industry

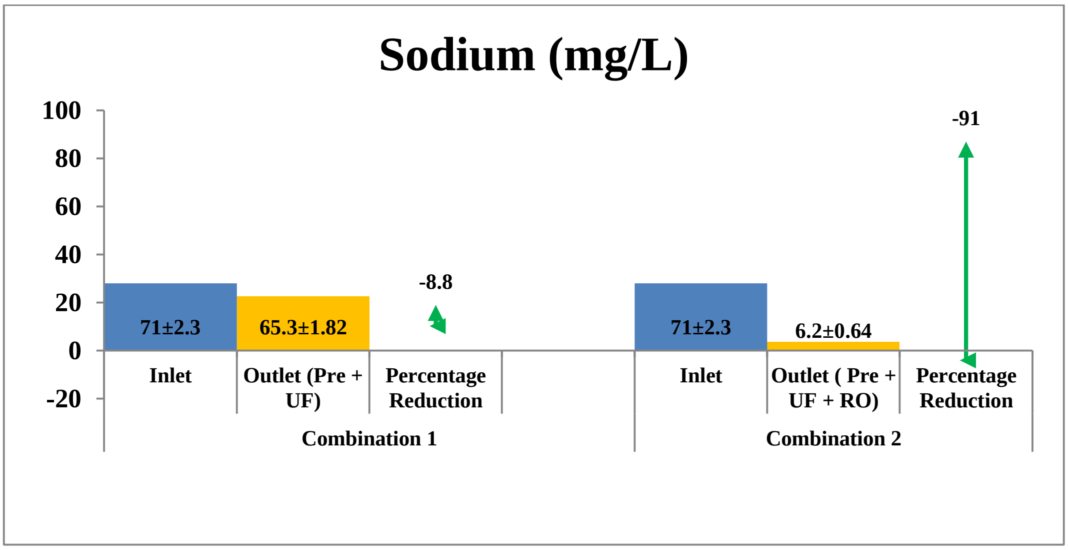


Figure 4.27: Effect of Different treatment setups on removal of Sodium in wood-based industry

The results obtained from our study are in good agreement with various previous studies on removing pollutants from wastewater using both RO and UF membranes. There has been a significant decrease ($P<0.05$) in the concentration of all pollution in both treatment methods (Table 4.23). Membrane filtration at low pressure is reported to be best for bleach effluent treatment in pulp and paper mills (Shukla et. al., 2013). Likewise, it has also been reported that the performance of using RO in the reduction of pulp and paper industry effluent is higher than UF and nanofiltration (Shukla et. al., 2009). In addition, Li et. al., 2004a have recommended the combined RO-UF membrane process for the treatment of wastewater.

4.11.10 Assessment of the reuse of water for recycling in the plant after membrane treatment

The reclamation of water from the effluent treatment plant has been recognized as a practical alleviation of water scarcity (Lazarova et al., 2013). The permeate of both combinations was compared with the feed water characteristics required by the pulp and paper industry listed in Table 4.24. The effluent from the secondary clarifier cannot be reused for the production of paper since none of the parameters fall under the prescribed tolerance limits. The application of pre-treatment and ultra-filtration has, however, reduced the pollution load but except TSS none of the parameters lies in the tolerance regime. However, the permeate from the series combination of pretreatment, Ultrafiltration, and RO unit has been able to achieve the tolerance limits for Color, TDS, and TSS to be reused in the production of all the grades of paper. Although Ultra-filtration is successful in the removal of Total Suspended Solids (TSS), its capacity to provide water that can be reused in the process of paper is highly doubtful. Hence, the addition of RO in the combination can be a practical solution to the problem.

Table 4.27: Change in the various water parameters with different treatment membrane setups in Wood Based Industry

*Significant at P<0.05, Two-tailed independent t-test

	TSS (mg/L)	TDS (mg/L)	COD (mg/L)	BOD (mg/L)	Color (PCU)	Lignin (mg/L)	pH	Conductivity (µs)	Potassium (mg/L)	Na (mg/L)
Inlet (Secondary Clarifier)	36±1.12	1933±87.46	183±11.09	20±0.79	615±23.21	168±8.78	8.12±0.34	2820±91.23	8.8±0.27	71±2.30
Outlet (Pretreatment + UF)	0*	1784±46.23*	146±9.76*	14±0.81*	176±11.20*	18±1.11*	7.89±0.12*	2142.9±84.35*	7.2±0.08*	65.3±1.82*
Outlet (Pretreatment + UF + RO)	0*	36±1.90*	0*	0*	0*	0*	5.12±0.71*	62.9±2.13*	0.2±0*	6.2±0.64*

Table 4.28: Water Quality requirement by Paper and pulp industries in India

	Effluent from different processes			**Tolerance for water for pulp and paper industry (BIS)**			
Water quality parameter	**Effluent from the secondary Clarifier**	**Permeate from pretreatment and Ultrafiltration unit**	**Permeate from pretreatment, Ultrafiltration, and RO unit**	**Ground Wood paper**	**Kraft Paper Bleached**	**Soda and Sulphite Paper**	**High-Grade Paper**
Color	615	176	0	20	15	10	5
TDS	1933	1784	0	500	300	300	300
TSS	36	0	0	25	25	25	10
COD	183	146	0	NS	NS	NS	NS

NS- Not specified

4.11.11 Statistical analysis of the various treatment setups in Star Paper Mill Ltd

The correlation of all the pollution parameters for Star paper mills is shown in Table 4.25. A maximum correlation of 0.99 has been observed between lignin and sodium content in the effluent stream. The least correlation of 0.38 has been observed between Total Solid solids and pH, an indication that there is no correlation between the TSS and the pH of the Wastewater.

The result of hierarchical cluster analysis of all pollution parameters from the effluent of all four sources viz. primary clarifier, secondary clarifier, Pretreatment + Ultrafiltration, and Pretreatment + Ultrafiltration + Reverse Osmosis from Star Paper Mills is shown in figure 4.29 The dendrogram shows two major clusters. Cluster 1 involves the effluent generated from the Plant outlet and the primary clarifier has the same quality of effluent indicating no improvement in water quality after the primary clarifier. Cluster 2 involves the effluent coming out from the Secondary Clarifier, Pretreatment + Ultrafiltration, and Pretreatment + Ultrafiltration + Reverse Osmosis indicating a pronounced reduction in the pollution load using membrane technology.

The screen plot of the Star Paper Mills is shown in Figure 4.30. The screen plot shows the eigenvalue for each individual Principal Component. The component having an Eigenvalue of more than one is selected. The elbow of the curve shows only two components falling above value one.

The agglomeration schedule for Star Paper Mills Pvt Ltd in Table 4.26 is employed to identify the optimal number of clusters. Two optimum clusters were observed. There were four stages of cluster combination obtained and the agglomeration coefficient shows an increase at every stage indicating the clusters having heterogeneous coefficients have been combined.

Table 4.29: Correlation Matrix of the Water Quality Parameters of Star Paper Mills

	pH	TSS	TDS	COD	BOD	Color	Lignin	Conductivity	Na+	K+
pH	1									
TSS	0.382481	1								
TDS	0.935581	0.598451	1							
COD	0.595059	0.858759	0.835567	1						
BOD	0.526238	0.820906	0.791658	0.992573	1					
Color	0.717426	0.790543	0.897516	0.954132	0.929613	1				
Lignin	0.572075	0.782123	0.82317	0.986964	0.993195	0.955677	1			
Conductivity	0.981019	0.477705	0.968369	0.699141	0.638252	0.828098	0.689433	1		
Na+	0.562757	0.762095	0.817351	0.982441	0.992849	0.944605	0.999071	0.678403	1	
K+	0.764734	0.747912	0.942796	0.967404	0.949943	0.963787	0.962476	0.84212	0.959763	1

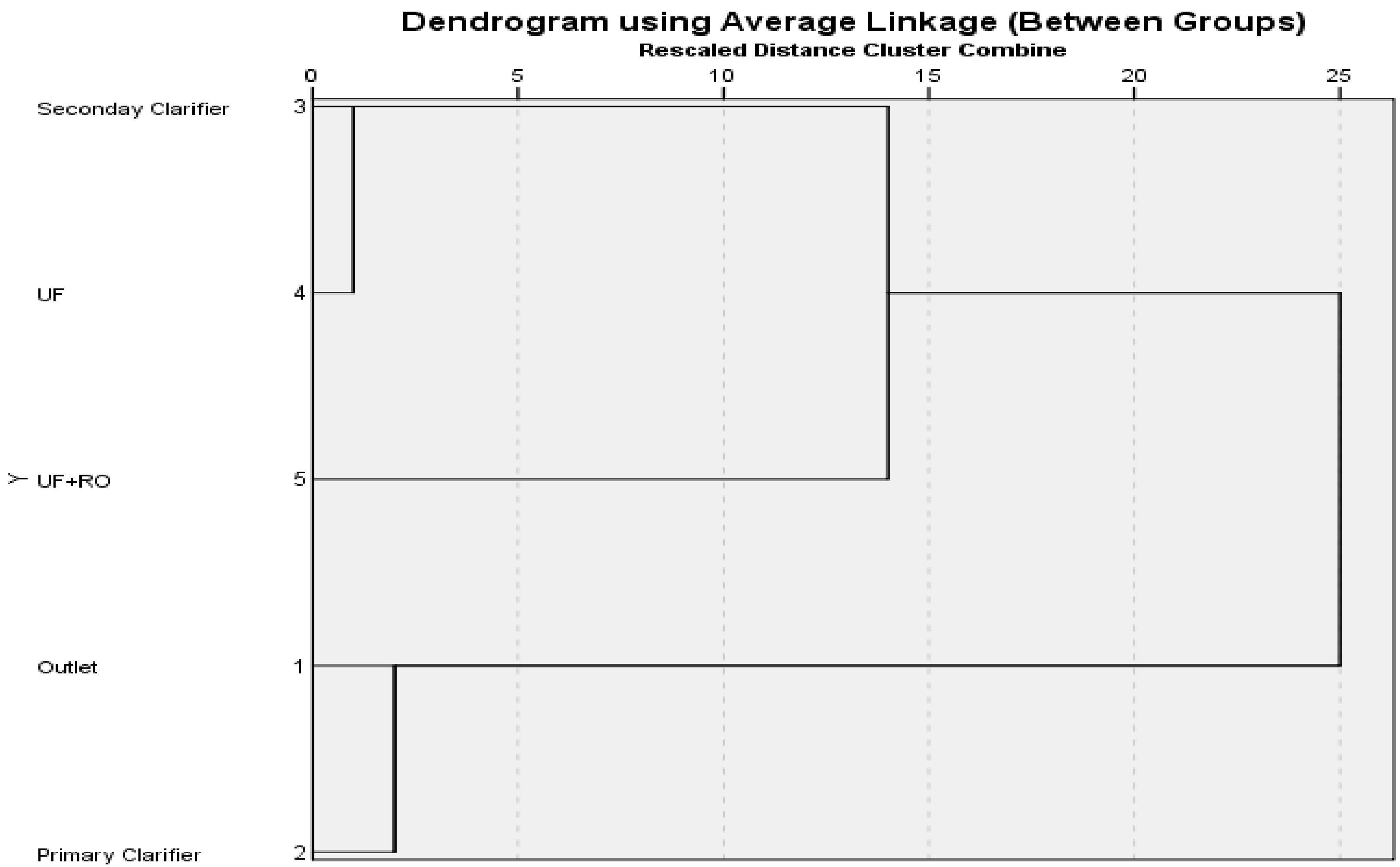


Figure 4.29: Dendrogram showing hierarchical cluster analysis of effluent of different treatments for Star Paper Pvt Ltd.

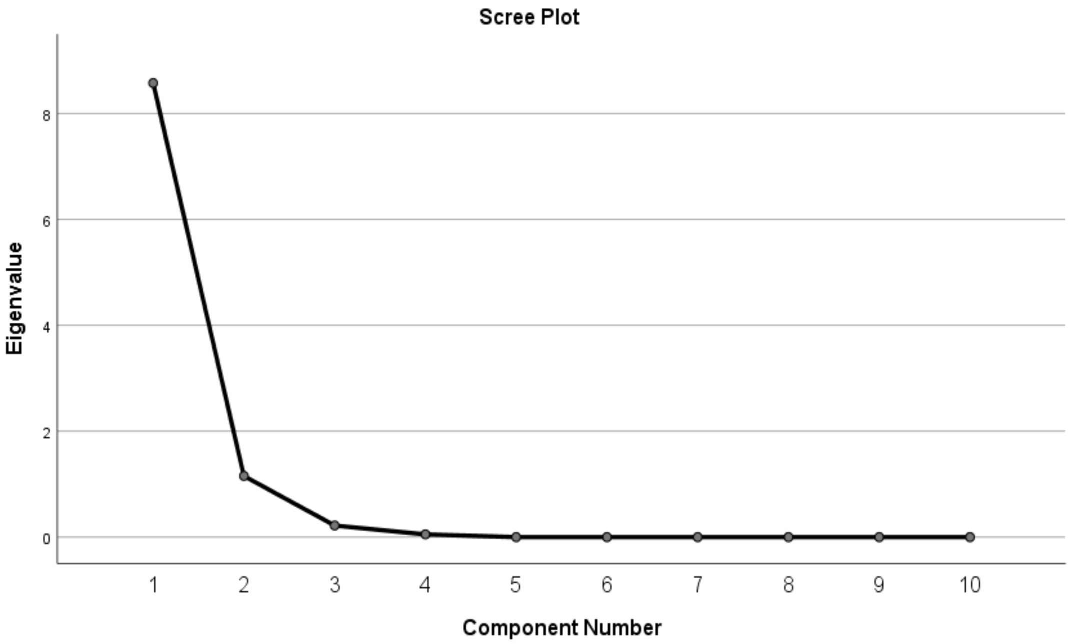


Figure 4.30: Screen Plot of the component analysis for Star Paper Mills Ltd

Table 4.30: Agglomeration coefficient of cluster analysis for Star Paper Mills Ltd

Stage	Cluster Combined		Coefficients
	Cluster-1	Cluster-2	
1	3	4	698615.913
2	1	2	1918463.588
3	3	5	9538778.316
4	1	3	17631923.251

4.12 Economic Evaluation of the membrane technology for pulp and paper industry

With lesser availability of surface water and declining level of groundwater, there has been pressure on the industry for environmental compliance. High Water Cess and high raw water costs have forced the water-intensive industry to find an economic and energy-conserving solution to the problem and has become a widespread goal worldwide to achieve in the wastewater treatment sector. All industries are implementing multilevel strategies and integrated approaches to save energy. In wastewater treatment systems, power is majorly consumed in aeration, pumping systems, sludge dewatering and treatment, and oversizing equipment. Thus, actual sizing of electro-mechanical equipment, optimization of structures, and management of the practices are the foremost and preliminary steps in terms of quality of water discharges and energy saving. It is clear that the consumption of energy and the total cost of the process are subject to the target reuse type as it indicates the water quality and therefore the design of the plant. Evaluation of cost/economics is a critical tool to evaluate and successfully implement plant design and operations. While upgrading existing treatment technology or installing new effluent treatment plants, the emphasis should be given to the reduction of capital as well as operational costs.

Various studies have been conducted to investigate techno-economic performance and consumption of energy, particularly for water treatment and reclamation of water (Mendert et al., 2019; Koutsou et al., 2020; Kehrein et al., 2021). For that three performance criteria have been investigated 1). Recovery rates, 2). Net cost, 3). Energy consumption. In effluent treatment plants the financial expenditure incurred is categorized into capital cost (CAPEX) and the expenditure for operation (OPEX). The cost of wastewater treatment primarily depends on the characteristics of the wastewater, the capacity of the ETP, and so on (Kehrein et al., 2020). The main aim of this analysis is to enhance the efficiency of effluent treatment plants, maximizing the pollution reduction and recycling and reuse of the treated waste-water in the paper-making process. The cost analysis includes two sets of comparisons (UF and RO in hybrid and individual modes) for the purification of water. The ordinary technical presumptions, specifications, and design parameters for cost estimation of water treatment are given in Table 4.28. A discount rate of 5% pertaining to the opportunity cost of time has been applied in the cost analysis by discounting future costs and benefits. Moreover, the cost of the plant is highly dependent on the recovery rate. The recovery rate of the Ultrafiltration unit is 86.43 %; however, the recovery rate of the Reverse osmosis unit is 19.46% only. However, since the ultrafiltration alone cannot reclaim the desired quality of water, hence combination of UF-RO has been used with an average recovery rate of 67.94%. The CAPEX and the OPEX of the lab-scale pilot plant are presented in Table 4.29 and 4.30 respectively. The OPEX includes the entire cost of the process including the membrane replacement cost, annual amortized cost, and the operation and maintenance costs.

Table 4.31: Design parameters and technical assumptions of UF, RO, and Hybrid UF-RO system

Parameter	UF	RO	UF-RO
Recovery %	86.43	19.46	67.94
Feed Flow Rate (LPH)	60	60	60
Water Treated (LPH)	51.85	11.67	40.76
Operation Hours /day	6	6	6
Annual product Volume (m^3/year), P	113.51	25.55	89.06
Life of system (Yrs)	25	25	25
Life of Membrane (Yrs)	3	3	3
Interest rate (%)	5	5	5
Plant Availability (%), f	90	90	90
UF/RO membrane Cost (Rs)	2000	1000	1000

Table 4.32: Capital Cost of UF and RO system in individual and hybrid mode

	UF	RO	UF-RO
High-Pressure Pump	1500	1500	1500
High-Pressure Pipes	500	500	500
Membrane Fitting	250	250	250
Membrane module	1000	1000	2000
Temperature control Device	1000	1000	1000
Total Cost	4250	4250	5250

The total pilot plant cost includes the expense of membrane replacement, the annual amortized expense, and the maintenance and operational expense of the process. The following equation was used to compute the annual amortized cost:

$$A = P \times \frac{r(1+r)^n}{(1+r)^n - 1}$$

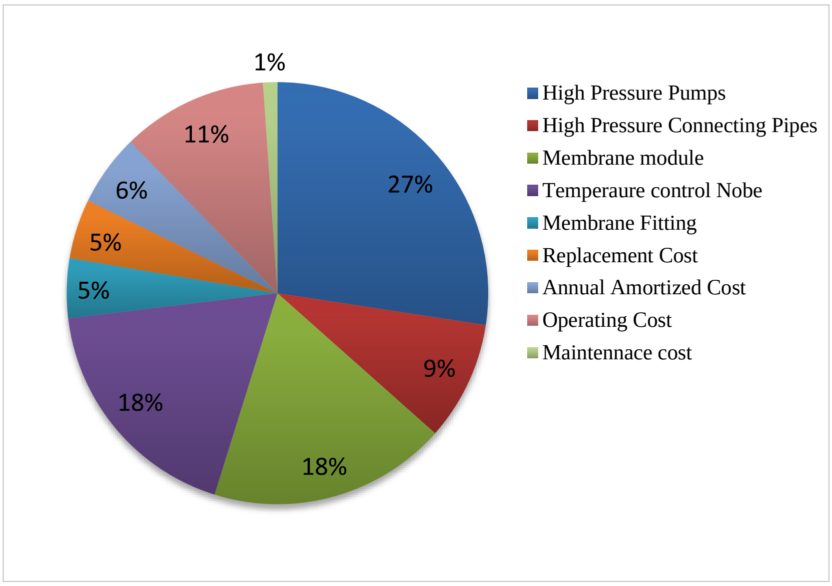
Where P accounts for the Principal Amount, A is the Periodic amortization payment, r represents the period interest rate and n represents the total number of payments.

Table 4.33: Annual Operating and maintenance cost of UF and RO system in individual and hybrid mode

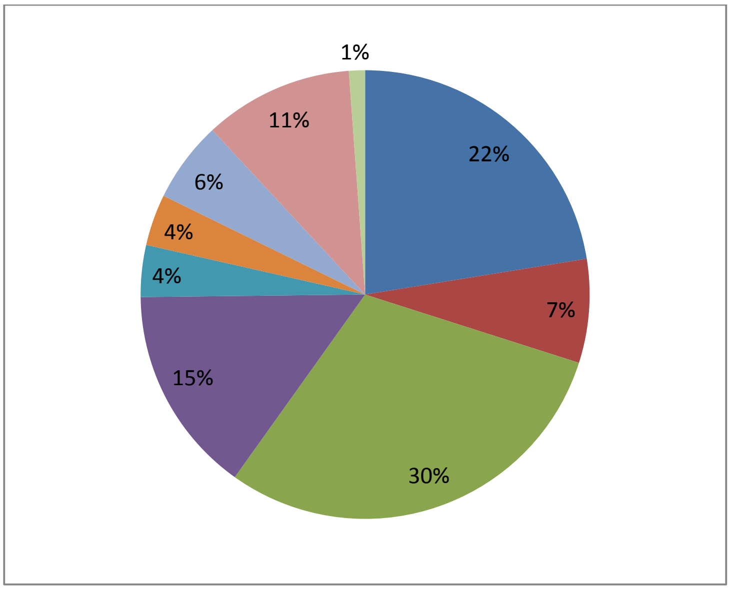
	UF	RO	UF-RO
Membrane Replacement cost, A_1	250	250	250
Annual Amortized Capital Cost, A_2	301.54	301.54	390.25
Operation & Maintenance annual Cost, $A_3 = A_2 \times 0.2$	60.30	60.30	78.05
Annual cost of Operation= ($A_1 + A_2 + A_3$)	611.84	611.84	718.30
Production Cost per unit (Rs/l) = C/ f×P)	0.598	2.66	0.896

The cost of wastewater treatment in UF units was Rs 0.59/L which increases to Rs 2.66/L with RO, since the recovery rate for RO is less than 20%, and hence the cost of water treatment per liter increases. However, the hybrid UF and RO have a recovery rate of around 67.94%, hence the cost of wastewater per liter was Rs 0.896. After economic analysis, it can hence be concluded that the hybrid of UF-RO membrane is a superior alternative to the traditional single UF and RO configuration since the cost of production is lesser and the quantity of water recovered is higher than the individual RO system. On the other hand, the use of UF alone cannot reduce the pollution load of waste water to the desired level so that we can recycle and reuse directly in the pulp industry.

The comparative cost distribution of UF and UF-RO hybrids has been depicted in Figure 4.31 (a) and (b). The CAPEX includes the cost of the membrane module, pumps, and the temperature control knob. The maximum capital cost in the Ultrafiltration unit includes the high-pressure pump followed by a membrane module (Martini and Ang, 2019). However, in the UF-RO hybrid module, the membrane module accounts for the maximum capital cost. The maximum operational cost was contributed by the electric power consumption followed by labor and chemicals respectively. In membrane filtration maximum energy is consumed due to the aeration of the membrane to avoid fouling followed by a pumping system used for permeate and feed pump. However, the power consumption in membrane filtration depends on the characteristics of the wastewater, selection of aeration technique, and pump (Rieger et al., 2012; Magdalena et al., 2019). However, when used as a tertiary treatment technique where the pollution loading is already low, it can come out as an energy-efficient method.



a) **Cost distribution (CAPEX and OPEX) of Ultrafiltration unit**



b) **Cost distribution (CAPEX and OPEX) of UF-RO hybrid unit**

Figure 4.31: Breakdown of the operational cost of wastewater treatment

Economic Evaluation of the membrane filtration for a plant of $0.5m^3$/hr was evaluated and shown in Table 4.31. The effluent coming from the secondary clarifier was used as a feed. The total operating cost (TOPEX) considering the capital cost that includes the membrane module, 2019, and the pumps, fitting, etc. Since the membrane filtration was used as a tertiary treatment, the cost of labor was negligible. The operating cost includes the electricity consumption only. The cost of wastewater treatment was calculated for 3 years, as the membrane has a life of 3 years. The calculated cost of treatment for Ultrafiltration and UF-RO hybrid was 0.292/L and 0.645/L respectively. The treatment operation cost for membrane filtration ranges from 0.26-0.32 Euro/m^3 (Judd et al., 2018). The increased use of membranes and ever-increasing demand may bring down the membrane cost which directly impacts the water treatment cost.

The rates of recovery of each membrane technique are different and the quality of the water, thus a comparison can be based on the amount of reclaimed water. Moreover, the price for reclaimed water may differ significantly. The operation of wastewater treatment as per a study conducted by Solon et al., 2019 consumes 0.45-0.6 kWh/m^3 of energy. The energy consumed by the ultrafiltration for $2700m^3$ water is 7.9kWh/$m^{3.}$ The energy consumption of various municipal treatment plants mentioned in the literature has ranged from 0.7-2.3kWh/m^3 of reclaimed water (Quist-Jensen et al., 2015). However, treatment of 2333.61 m^3 ultrafiltration permeate with reverse osmosis requires 28.6 kWh/m^3, which is somewhat larger than all other operational units. The specific cost for consumption of energy in operations accounts for 0.067/L for ultrafiltration and 0.31/L for hybrid UF-RO units.

The operating cost of the membrane filtration unit in hybrid mode per m^3 recovered water is extensively higher than the total capital cost needed for the initial setup of the plant. The main reason for the same is due to the fact that capital cost (CAPEX) is incurred only once at the initial setting of the plant of an average lifetime of 20 years while the operating costs are due constantly. Therefore, the more important factor in designing the membrane treatment plant is to optimize the operational cost rather than saving the capital cost. And since the major fraction of the operating cost is covered by the energy consumption; the focus should be given to the use of any other alternative source of energy. The solar energy as well as the energy recovered from the aerobic digestion and the biogas produced by the treatment plant can help to resolve the issue (Frijns et al., 2013).

Table 4.34: Economic evaluation of the Lab scale Effluent Treatment model

S. No.	Description	Volume	UF	UF+RO
	Plant Capacity	0.5 m^3/hr		
1.	Electro-mechanical Cost Pumps, Blowers, Diffusers, Instruments, pipes, panels, and Fitting	01 lot	1,32,500.00	1,48,300.00
2.	Membrane Cost + Replacement	01 lot	4,75,000	7,50,000
3.	Operation cost (Power for 03 Years		3.25 KW = 3.25 ×6 = 19.5KW @Rs 8.57/ KW for 3 years= 19.5×365×3×8.57 = 1,82,990.9	15KW Rs. 8,44,573.5
	Total (Rs)		7,90,540	17,42,873.5
	Volume of water treated (0.5m^3/hr × 6 hrs of operation per day × 900 days) Plant Life 3 Years		2700 m^3 0.292/lit 0.067/lit 7.9Kwh/m3	2700 m^3 0.645/lit 0.31/lit 36.5/m3

The total amount of freshwater consumed by Bindal paper and Star paper mills is 8500 KLD-9000 KLD and 11190 KLD. The average rate of the water charged by the Government of UP for industrial supply is 26.40/1000 liter. So, the daily expenditure of the mills towards the water bill is Rs 2, 24, 400 and Rs 2, 95, 416 for Bindal paper mills and Star paper mill respectively. Using the hybrid UF-RO model, the treated water can be sustainably recycled and reused in a closed loop. Moreover, the government charges Cess at a rate of 5p/KL on the consumption of water under The Water (Prevention and Control of Pollution) Cess Act, 1977. This allows the recycling and reuse of treated water and the total fresh water requirement of the plant can be minimized which will ultimately lead to decreased cost of operations. Moreover, a rebate of 25% in Cess is given to the industries treating the wastewater on their premises.

CHAPTER 5
CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

Improving and maintaining the quality of the environment and achieving sustainable goals are the foremost and most critical challenges that the globe is facing currently. The ever-increasing population, urbanization, and industrialization have become a threat to the achievement of the targets making them unconfined and limitless. The sudden urbanization and expanding industries in India have created huge pressure on the water resources and hence, the water demand in the last century has risen four to sevenfold. As per the CPCB report, 70-80% of the wastewater generated is sent back to the ecosystem. The installed sewage treatment capacity is only 6190 MLD against the total generation of 22939 MLD, creating a gap of 78.7%. Moreover, the quality of the waste water recovered after treatment also needs to meet the specified standards (Hugh and Thompson, 2004). This ever-increasing demand for water, the huge production of wastewater, and the inefficiency of treatment facilities have created huge pressure to find an alternative and advanced method of wastewater treatment so as to meet Sustainable Development Goal 6 which focuses on the management of wastewater quality and increasing the water use efficiency.

After many of the advanced treatments like coagulation, and oxidation have one or another drawback, the membrane technology has emerged as a hope for treatment and reclamation of wastewater. Thus, in the present study, the paper and pulp industry wastewater from 2 different industries using different raw materials contributing approximately 60% of the wastewater were chosen as source wastewater. An integrated lab-based model based on a pretreatment filter along with ultrafiltration and reverse osmosis was used with the objective of removing the pollutants (Organic matter, Color, and TDS) and maximizing the water recovery so as to be recycled and reused in the industrial process.

To fulfill the objectives of the work, the physicochemical characterization of the input and output of the effluent treatment plant was done using standard methods. The operating condition of the membrane was optimized to maximize the rate of water recovery and minimize membrane fouling. The permeate coming from the membrane filtration unit was assessed for its potential to be reused in the paper and pulp processing in Industries. Eventually, the cost and the energy consumption were evaluated to mitigate the problem of water scarcity and reach the goal of zero liquid discharge.

Following are the conclusions of the present study.

1) Physio-chemical characterization was done separately for both the chosen paper mills. Bindal Paper Mills Ltd. Muzaffarnagar, UP, is an integrated pulp and paper plant with a capacity of 110 MTons/Annum of writing and printing grade paper using agro waste like bagasse, wheat straw, and hardwood and poplar grown by the farmers. The freshwater consumption in Bindal is approximately 52.3 m^3/T finished paper. The effluent coming out from the various processes has a pH value of 7.39±0.32, BOD value of 653±14.81, COD value of 1465±31.23 mg/L, TSS value of 1911±21.17 mg/L, TDS value of 2967±26.31 mg/L, lignin content of 1004±16.72 mg/L and Color value of 1206±21.12 PCU. The current ETP of Bindal Paper Mill is based on the conventional activated sludge process along with advanced tertiary treatment techniques comprised of the primary clarifier, a secondary clarifier, a Sand filter, an equalization tank, Dissolved Air Floatation, aeration tank, a Direct Membrane filter (DMF), Sludge Bed Drier (SDB) and chemical recovery plant. The mill has also placed an Online Continuous Effluent Monitoring System (OCEMS) at the final output to monitor the quality of the treated effluent 24×7. The pH value of effluent from the secondary clarifier is slightly alkaline (7.46±0.44). The BOD as well and COD values of the outflow coming out of the secondary clarifier were observed in the range of 27±0.56 mg/L and 216±9.11 mg/L respectively. The percentage removal using the conventional

effluent treatment plant including COD, BOD, and TSS was observed as 99.25%, 99.11%, and 99.45% respectively (Leiviskä et al., 2010).

2) Star paper utilizes the wood of eucalyptus and poplar for paper making. The mill has 4 paper machines with a production capacity of 300 Tonnes/day. The mill utilizes traditional chlorine bleaching technology and also involves an oxidative extraction stage which utilizes oxygen and peroxide. Star paper mills utilize around 12000m^3 of freshwater (groundwater) daily, which is abstracted from the bore wells. The effluent coming out from the various processes has a pH value of 8.41±0.29, BOD value of 715±13.68, COD value of 1395±29.72 mg/L, TSS value of 2016±18.21 mg/L, TDS value of 3114±35.14 mg/L, lignin content of 1192±21.67 mg/L and Color value of 1421± 14.23 PCU. The current ETP of Star Paper Pvt ltd is based on the traditional method namely, the activated sludge process, and comprises the primary clarifier with 10087 m^3 capacity and a Side Water Depth of 4m; an aeration tank with 11 aerators of 50HP capacity; A secondary clarifier with a capacity of 6005 m^3. The plant is also installed with a belt press for dewatering the sludge generated in primary and secondary clarifiers. The reported pH of the discharge was found to be in the alkaline range (8.12±0.34) which is within the determined CPCB limits. The TSS from wood-based paper mills (i.e., 36±1.12 mg/L) after secondary treatment is lower than most of the pollutants. The BOD and COD values of the effluent coming out of the secondary clarifier were observed in the range of 20±0.79 mg/L and 183±11.09 mg/L respectively. The percentage of COD, BOD, and TSS was observed as 98.81%, 98.80%, and 98.70% respectively. All the parameters were within the CPCB-prescribed limits.

3) The decision-making for multi-criteria optimization of various parameters viz. Temperature, pH, Flow rate, and Pressure on the efficiency of using Ultra-filtration through grey relational analysis in combination with Taguchi approach concerning Flux Decline due to fouling and COD rejection rate. The percentage contribution of each parameter to achieve minimum fouling was

pH>Pressure>Temperature>flow Rate; and to achieve maximum COD rejection, it was pH>Temperature>Pressure ≥ flow rate.

Taguchi and Grey Relation method-based optimum operating conditions were found to be at pressure 2 bar, pH 8, flow rate of 60 l/hr, 17.1% of the COD rejection rate, and temperature 32°C was obtained under optimum working conditions, that lie within the stipulated range consisting of a 95% confidence interval. Measurement of TDS, TSS, COD, BOD, and Color showed a significant decrease of 11.90%, 99.99%, 17.10%, 22.20%, and 73.70% respectively, showing a substantial reduction in the pollutant loads. The GRG is improved significantly (0.1918) through the setting of optimal parameter combinations. It can be concluded that the Grey-based Taguchi approach for solving multi-attribute decision-making proves to be effective in reducing the number of experiments along with getting reliable results.

4) The purpose of the analysis was to assess the efficacy of membrane technology pollutants removal from secondary clarifier effluent of the paper mill and to check the potential of reuse of the treated wastewater for paper production. For the same purpose a pilot system that incorporates a membrane filtration unit *i.e.*, pre-filtration, UF, RO membrane. Two combinations of membrane filtration were used *viz*, combination 1 consisted of the series assembly of pre-filtration and UF whereas RO membrane is added with pre-filtration and UF in the second combination. The operating conditions were optimized for minimization of the membrane fouling and maximization of the pollution rejection.

5) The pH value of the effluent of the secondary clarifier has an average value of 7.46±0.44.

The pH of the wastewater generated is associated with the microbial activity that impacts the secondary treatment process of the activated sludge process. The reduction of pH of the discharge was noted as 7.19 ±0.12 by combining pre-filter and Ultra-filtration. The secondary clarifier effluent was already low with a TSS value of 56±0.96, indicating that the secondary treatment itself is sufficient to reduce the TSS load. The change in the TSS level with both the combinations of membrane filtration configuration was able to entirely reduce the TSS. The effluent coming from the secondary clarifier has a TDS value of 2215±56.45 mg/L. However, the removal of TDS is not satisfactory with pre-treatment and UF, as only 7.7% (2215±56.45 mg/L to 1951±41.23 mg/L) of TDS could be reduced. The integration of pre-filtration, RO membrane, and UF can reduce 88.26% of TDS for effluent coming from Bindal Paper Mills. When pre-filtration and UF were utilized, reduction potential was noted to be 20.2% (183±11.09 mg/L to 146±9.76 mg/L) respectively in terms of COD. However, the addition of RO membrane along with pre-filtration and UF appeared to reduce 100% of COD and BOD from the effluent of Star Paper Pvt Ltd. The biological Oxygen Demand of the effluent generated from the secondary clarifier has an average value of 20±0.79 mg/L. The pre-filtration and UF in combination have reduced the BOD by 30% (20±0.79 mg/L to 14±0.81 mg/L). However, RO membrane addition along with pre-filtration and UF appeared to reduce 100% of BOD from the effluent.

The effluent generated from the secondary clarifier has a color with an average value of 615±23.21 PCU. Pre-filtration and UF in series could reduce color from 615±23.21 PCU to 176±11.2 PCU *i.e.* by 71.5%. The significant contribution of the pre-filtration and UF membrane in terms of lignin removal from the outflow as 89% lignin reduction (168±8.78 mg/L to 18±1.11 mg/L) was observed. However, the extended RO membrane successfully reduced the lignin content entirely which is 100% lignin elimination from the plant discharge(Leichang et al., 2018).

6) The pre-treatment filter and Ultra-filtration combination satisfactorily reduced the TSS, and lignin, and discharged from both the mill effluents. The positioning of the pretreatment filter helps in the reduction of the pollutant load on the membrane hence decreasing the fouling of the membrane. The pre-treatment filter also reduces the economic cost of membrane cleaning and

replacement due to fouling. However, the removal of TDS, COD, and BOD using pre-treatment and ultra-filtration was not satisfactory (<30%). This may be attributed to the high concentration of LMW compounds in the plant outflow. However, the addition of RO membrane could significantly (>98%) reduce the pollutants from the paper mill effluent. RO membrane exhibited the highest pollutant removal ability when used in conjunction with pre-filtration and UF membranes. So, the application of Pre-treatment, Ultrafiltration, and Reverse Osmosis in series could be an effective measure to help in the recovery of vital and finite water resources and promote their reuse in industries.

7) The permeate of both combinations was compared with the feed water characteristics of the pulp and paper manufacturing industry. It was analyzed that the effluent from the secondary clarifier cannot be reused for the production of paper since none of the parameters fall under the prescribed tolerance limits with a maximum color value of 5 PCU, 300 mg/L TDS, and TSS value of 10mg/L. The application of pre-treatment and ultra-filtration however reduced the pollution load but except TSS none of the parameters lies in the tolerance regime. However, the permeate from the series combination of pretreatment, Ultrafiltration, and RO unit has been able to achieve the tolerance limits for Color, TDS, and TSS to recycle it in the production of all the grades of paper. Ultra-filtration successfully removes Total Suspended Solids (TSS) but its capability for provision of reusable water in the processing of paper is highly doubtful. Hence, the addition of RO in the combination can be a practical solution to the problem.

8) The maximum correlation of 0.99 has been observed between lignin and sodium content in the effluent stream. The least correlation of 0.38 has been observed between Total Solid solids and pH, an indication that there is no correlation between the TSS and the pH of the Wastewater. The dendrogram shows two major clusters. Cluster 1 involves the effluent generated from the Plant outlet and the primary clarifier has the same quality of effluent indicating no improvement in water quality after the primary clarifier. Cluster 2 involves

the effluent coming out from the Secondary Clarifier, Pretreatment + Ultrafiltration, and Pretreatment + Ultrafiltration + Reverse Osmosis indicating a pronounced reduction in the pollution load using membrane technology. The screen plot shows the eigenvalue for each Principal Component. The component having an Eigenvalue of more than one is selected. The elbow of the curve shows only two components falling above value one. Two optimum clusters were observed.

9) Economic assessment was carried out for single-mode Ultra-filtration along with the hybrid (UF- RO system). The results show that the water treatment cost for the Ultrafiltration Unit and Hybrid (UF-RO) was Rs 0.59/liter and Rs 0.89/liter respectively. The cost of waste treatment using the hybrid system was however 33.70% higher than the ultrafiltration unit but the quality of wastewater recovered using the hybrid unit meets the quality standard given by CPCB. However, the cost of treatment for a plant 0.5m^3/hr operating for 6 hours was calculated to be 0.292/lit and 0.64/lit for Ultrafiltration and UF-RO hybrid respectively. The CAPEX covers the cost of the temperature control knob, pumps, and the membrane module. The maximum capital cost in the Ultrafiltration unit includes the high-pressure pump followed by a membrane module. However, in the UF-RO hybrid module, the membrane module accounts for the maximum capital cost. The maximum operational cost was contributed by the electric power consumption followed by labor and chemicals respectively. In membrane filtration maximum energy is consumed due to the aeration of the membrane to avoid fouling followed by a pumping system used for permeate and feed pump. Indicating the cost of treatment can be reduced if the size of the treatment plant can be increased. Moreover, using the membrane technology and reusing and recycling the treated water in a closed loop can reduce daily expenditure.

Membrane technology is an emerging technology for generating high-quality treated water in a single treatment process and nanobubble aeration with closed-loop systems slowly gaining importance for wastewater treatment to save energy consumption, increase membrane life, and reduce fouling of membranes. This study proves the hybrid lab model is cost and energy-efficient which may solve the matter of water scarcity as both treated wastewater can be further reused for industry application.

Recommendations

Membrane technology is an emerging technology for generating high-quality treated water in a single treatment process but several aspects need attention and require further focus which can become the starting point of future research to study feasibility and fouling rate, optimization of membrane bioreactor operation, increase in energy efficiency which leads to improvement of membrane technology.

Implementation of nanobubble aeration systems is slowly gaining importance for wastewater treatment to save energy consumption, increase membrane life, and reduce fouling of membranes which need more precise research work.

While designing a membrane bioreactor, plant layout, effluent characteristics, and waste discharge parameters should be evaluated carefully, and under or oversizing, the wrong selection of equipment can influence the membrane bioreactor's efficiency which increases the energy consumption.

For sustainable development, wastewater treatment could become a blueprint. Wastes are nothing but misplaced resources, thus if wastewater is treated as a valuable resource can become an alternative source that can supplement environmental and economic benefits for human beings.

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PUBLICATIONS

Journal publications

- **Sumit Dagar**, S.K. Singh and Manoj Kumar Gupta, Economics of advanced technologies for wastewater treatment: Evidence from paper and pulp industry, Frontiers in Environmental Science, 2022, 1-18.
- **Sumit Dagar**, S.K. Singh and Manoj Kumar Gupta, Integration of Pre-Treatment with UF/RO Membrane Process for WasteWater Recovery and Reuse in Agro-Based Pulp and Paper Industry, Membranes, 2023, Volume 13, Issue 2, 199

Paper presented in Conferences

- Attended and presented a paper titled **"Evaluating water recycle and reuse efficiency of membrane filtration in Indian pulp and paper mills"** in the International Conference on Climate change and Impacting, Emerging Frontiers in Biological Sciences held on 15-16 July, 2022 organized by Engineers Bhawan, Institution of Engineers, ITO, New Delhi.

- Attended and presented a paper titled **"Integration of Pre-Treatment with UF/RO Membrane Process for Waste Water Recovery and Reuse in Wood-Based Pulp and Paper Industry"** in the International Conference on International perspectives in economics and management for Sustainability "held on 20th December, 2022 organized by Great Lakes Institute of Management, Gurgaon, India.