Preparation of Cellulose acetate blend Green membrane Synthesis and its application studies

Anitha.S

(13PCH002)

Thesis Submitted to

Avinashilingam Institute for Home science and Higher Education for Women,University

Coimbatore-641043

In partial Fulfilment of the Requirements for the

Degree of Master of Science in Chemistry

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J. Thamarai Selvi 27.3.15

Signature of the
Supervisor

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Signature of the
Head of the Department

Acknowledgement

ACKNOWLEDGEMENT

Every work on its backdrop has the blessing of the Lord Almighty.Therefore submitting my reverential gratitude on the feet of the Lord Almighty,I deem it necessary to thank all who rendered their help during the course of the study.

I sincerely thank **Dr.T.S.K. Meenakshi sundaram M.A., M.Phil., Ph.D.,Chancellor,** Avinashilingam Institute for Home Science and Higher Education for Women, Coimbatore-641043, for providing a conducive and congenial ambience for the execution of my research work.

I express my sincere thanks to **Dr. (Tmt).Sheela Ramachandran, M.Sc., P.G.Dip., Ph.D., Vice chancellor,** Avinashilingam Institute for Home Science and Higher Education for Women, Coimbatore-641043, for providing an opportunity to carry out my work in this University.

I record my sincere thanks to **Dr. (Tmt).Venmathi** Registrar in-charge , Avinashilingam Institute for Home Science and Higher Education for Women Coimbatore-641043, for rerendering adequate help required to carry out the work.

I express my grateful thanks to **Dr.(Tmt) Saroja Prabakaran, M.A., Dip.Ed.,(Madras),Ph.d.,(Mother Teresa),** Former Vice-Chancellor, **Director, Hall of residence,** Avinashilingam Institute for Home Science and Higher Education for Women, Coimbatore-641043, for all amenties provided for the conduct of the study.

I express my gratitude to **Dr. (Tmt). A. Parvathi, M.Sc., Dip.Ed., M.Phil., Ph.D., Dean Faculty of Science,** Avinashilingam Institute for Home Science and Higher Education for Women, Coimbatore-641043, for her support and encouragement.

I sincerely thanks to **Dr. (Tmt). R. Shyamala, M.Sc., Dip.Ed., M.Phil., Ph.D., Professor and Head, Department of Chemistry,** Avinashilingam Institute for Home science and Higher education for Women, Coimbatore-641043, for her support in this study.

I expressed **my hearty and sincere thanks to my guide Dr.(Mrs) Thamaraiselvi, M.Sc., M.Phil., Ph.D., Associate Professor (Department of Chemistry)**, Avinashilingam Institute for Home science and Higher education for Women, Coimbatore-641043, for her Exellent support, guidance,Valuable advice, patience, timely suggestion and encouragement throughout my study.

I also thank all the **staff members of the Chemistry Department**, Avinashilingam Institute for Home science and Higher education for Women, Coimbatore-641043, for their help and whenever required to complete my work and co-operation during my study.

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I thank all the non-teaching faculty and research scholars for their help

.

My special thanks to my **beloved parents, family members and my sister. Extented my thanks to lovable sister C.Pradipa** for their constant support and timely help extended whenever, I was in need.

I deeply express my special thanks to all **my friends**, for their constant support during the course of study and throughout the work.

Anitha.S

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LIST OF ABBREVIATION

RO	Reverse Osmosis
UF	Ultra Filtration
NF	Nano Filtration
FAS	Ferrous Ammonium Sulphate
TGA	Thermo Gravimetric Analysis
SEM	Scanning Electron Microscope
FT-IR	Fourier Transformer
X-RD	X-Ray Diffraction
D.O	Dissolved Oxygen
BOD	Biological Oxygen Demand
COD	Chemical Oxygen Demand
EC	Electrical Conductivity
SPADNS	Sulpho Phenyl Azo Dihydroxy Naphthalene Disulphuric acid

Introduction

1. INTRODUCTION

Water is a common chemical substance essential for the survival of almost all known living organisms. Water covers 71% of the earth’s surface, but 97% of this water exists as salt water in oceans. Of all surface water, glaciers and icecaps hold approximately 2%, and freshwater rivers and lakes contain only 1%. Yet many societies around the world do not give consideration and attention to preserving this vital commodity that is in limited supply.

Almost two-billion people in the world, (approximately 25% of the world's population) do not have access to safe drinking water. Particularly important is the prevention of industrial and biological waste-disposal, pollution, and contamination of water sources and air pollution (Wahlqvist *et al.*, 2009)

However, not all contaminants are purely man-made or anthropogenic. Global warming has also affected environmental pollution. Environmental pollution is an unintended outcome of anthropogenic causes and accelerated by human activities. Nevertheless, there are also natural phenomena. Together these enhance the climate-change–induced cyclones, hurricanes, typhoons, droughts, and floods, all of which lead to significant groundwater contamination.(Bhattacharya *et al*., 2001)

Water is one of the basics that support all forms of plant and animal life and it is generally obtained from two principal natural sources; Surface water such as fresh water lakes, rivers, streams and Ground water such as bore hole water and well water. Water is used for so many purposes among which are agricultural uses e.g. irrigation, fishing etc. domestic uses e.g. cooking, bathing, washing, drinking etc. industrial uses e.g. mining, brewing etc. constructional uses e.g. building, road construction etc. One of the most important environmental issues is ground water contamination and between the wide diversity of contaminants affecting water resources (Adetunde et al.,2014).

More than one billion people lack access we develop technologies to provide safe, clean water to the home and developing countries. In these centuries, purification takes major role to make effective delivery of drinking water. In many places clear water project are more expensive but purification process is cheap, simple and practical, purified water is obtained from salt water through desalination. Consequently, water consumption-related deaths (ranging from five to seven million deaths per year) are probably the largest single cause of deaths in the world. It is estimated that in 2020, at the current rate, 75 million people will die each year of preventable water-related deaths.(Gleick *et al.,* 2002, Wimalawansa *et al.,*2013)

Governments in many countries continue to neglect the most vulnerable people who do not have easy access to clean water. This caused, at least in part, by the lack of adequate resources, lack of priority, and/or disregard for the plight of people who do not have a voice, and the lack of safe water and sanitary facilities. To bridge this need, many charitable organizations have stepped in to provide this essential live-saving commodity. During the past two decades, several methodologies were developed to convert contaminated water and brackish water to clean potable water. In the early 1970s at the University of California, Berkley, developed a technology and is relevant for most countries: namely, the reverse osmosis (RO) process**.**(Klumb *et al*.,1975, Nielsen *et al*.,1974, Kunz *et al*.,1973)

Membrane is defined essentially as a barrier,which separates two phases and restricts transport of various chemicals in a selective manner. A membrane can be homogenous or heterogeneous,symmetric or asymmetric in structure, solid or liquid;can carry a positive or negative charge or be neutral or bipolar. Transport through a membrane can be affected by convection or by diffusion of individual molecules, induced by an electric field or concentration, pressure or temperature gradient.The membrane thickness may vary from as small as 10 microns to few hundred micrometers.

Membrane gas separation is attractive because of its simplicity and low energy cost, but it has one major drawback and that is a reverse relationship between selectivity and permeability. Nano composite membranes, in which selectivity and permeability can simultaneously be improved. (Baker *et al*.,2004,porter *et al.,* 1990,Kargari *et al*, 2004, Kargari *et al*., 2004)

Most of the filtration systems used in developing countries based on simple mechanical filtration processes These remove particulate matter by a mechanical process based on physical size. These methods may remove some larger inorganic matter and metals that are in the particulate forms, but not dissolved in the water. Some filters have an additional activated carbon component, which adsorbs some chemicals to the surface of carbon. However, unlike with absorption methods, adsorption depends on the available surface area of the material; and thus the capacity is limited.

The three most common heavy metal contaminants that causing ill health, cadmium, lead, and arsenic in water are in the dissolved form and thus generally cannot be removed by these filtration methods.(Wimalawansa *et al*., 2013**)**

There are several types of filter available and each performing a specialised task.

1.1 ULTRAFILTRATION (UF)

Traditional ultrafiltration technology has provided for relatively clean water that was once suitable for sewer discharge but generally inadequate for reuse. However, recent changes in sewer discharge limits (oil and grease, COD, metals,etc.) have created a need to generate even cleaner water, with an emphasis on reuse. In many locations, ultrafiltration treatment alone does not meet discharge laws. Some companies have resorted to secondary treatment steps in order to comply with the tightening regulations. A related type of membrane technology, nanofiltration (NF), may offer a cost-effective means of separating recyclable or sewerable water from oil. The feasibility of nanofiltration is explored in this paper. Nanofiltration, otherwise known as “loose reverse osmosis,” operates as a water softener rather than as a complete purifier.(Cadotte *et al.,* 1988)

1.2 NANO FILTRATION (NF)

Ultrafiltration (UF) membranes allow almost all soluble material to pass except for large proteins the NF membrane has much smaller pores and is thus able to significantly reject many of the smaller organics that pass through the UF membrane. As a result, nanofiltration can be classified between RO and UF on the filtration spectrum. Nanofiltration has a very strong potential to serve in waste recovery applications like oil/water separation. The NF permeate from oily solutions is cleaner than the UF permeate, therefore allowing more opportunity for in-house recycle. If discharge is preferred, NF permeate can also be clean enough to meet tightening discharge requirements.(van den Berg *et al*., 1987)

1.3 MICROFILTRATION

Microfiltration has limited application in textile wastewater treatment because of its close resemblance to conventional crude filtration processes (Mulder, 1996). Microfiltration membranes usually have pore sizes in the range 0.1 to 10 mm; separation through microfiltration is usually effected at a low pressure differential within 2 bar (Dutta, 2007).

It is mainly used for removal of particles suspension and colloidal dyes from exhausted dye bath and from discarded rinsing bath discharge; microfiltration membranes, however, permit the unconsumed auxiliary chemicals, dissolved organic pollutants and other soluble contaminants to escape with the permeate (Juang *et al*., 2013, Koltuniewicz *et al.,* 2008). Hence, microfiltration is seldom used as an unaided independent treatment technique for remediation of complex industrial wastewater such as textile effluent (Ellouze *et al*.,2012)

The study, thus established the superiority of microfiltration over coagulation flocculation as the pre-treatment step prior to nanofiltration, for treating textile wastewaters. Microfiltration can also be used as a post treatment step while treating industrial effluents(Juang *et al*.,2013)

1.4 ELECTRODIALYSIS

Electrodialysis has been commonly used for treating concentrate, a coupled pellet reactor. (Tran *et al*.,2012) If renewable energy can be used as a power source for electrodialysis, both treatment costs and CO_2 emission could be reduced. In an integrated system consisting of electrodialysis and ozonation, 22.5% of water is returned to the biological treatment unit and 2.5% of water is electrodialysis concentrate, which is discharged to a canal (Zhang *et al*., 2012). In the system, concentrate is mixed with the UF backwashwater and UF concentrate before being discharged to the canal. In order to enhance the recovery rate of the RO system, the electrodialysis-treated effluent is further ozonated before being recycled to the biological treatment unit. The cost of the electrodialysis process used in the system is estimated to be 2.4 times lower compared to the UF or RO treatment cost and 3.7 times lower than the cost of wastewater treatment (Cheng *et al.,* 2008).

1.5 OSMOSIS

Osmosis is defined as the spontaneous passage or passive diffusion of water or a solvent through a semi-permeable membrane due to osmotic pressure. Liquid moves from a dilute to a more concentrated solution across a semi-permeable membrane.

1.6 REVERSE OSMOSIS (RO)

Osmosis is a phenomenon in which water moves from dilute salt solution to a concentrated salt solution through a semi permeable membrane whereas in reverse osmosis, an external force is applied to the water to move from a concentrated salt solution through a semi-permeable membrane. Reverse osmosis treatment removes almost all the salts from the water.

Reverse osmosis membranes are effective in removing most organic and inorganic compounds from water solutions. In recent years, new polymer chemistry and manufacturing processes have brought about dramatic improvements in efficiency, lowering operating pressures and reducing cost. As a result, Reverse osmosis membranes are increasingly used by industry to concentrate or remove chemicals.

Reverse osmosis is increasingly used as a separation technique in chemical and environmental engineering for the removal of organics and organic pollutants present in wastewater. It is seen from literature review that Reverse Osmosis processes have been widely used for separation and concentration recovery of solutes in many fields. The use of Reverse osmosis in the treatment of various effluents of chemical **(**Bodalo-Santoyo et al,2004**)**.Reverse osmosis is mainly used to concentrate all the solutes present in a mixture, while removing water in the process.(Muthukumarappan et al.,2010).

Recently, seawater has been attracted as a promising alternative source for drinking water. Reverse osmosis (RO) is considered as the most efficient technique for seawater desalination. However,RO application in desalination is not free from membrane fouling that adversely affects the performance efficiency in RO plants.

Especially, biofouling will cause a decrease in membrane flux which in turn will result in an increase in operational pressure,and an increase in the frequency of membrane cleanings. This incurs a higher energy demand (Guo et al., 2012; Khan et al.,2011).

Advantages of this membrane are desalting removal of bacteria and viruses from feed water .Also the energy consumption is low when compared to other desalination process, it is also recognized that the membrane should be chemically inert, mechanically strong, and creep resistant. The membrane should be also capable of being fabricated into configuration of high surface to volume ratio. **(**Lonsdale *et al*., 1972)

The process has also been applied to treat tap water. Since conventional tap water processes do not remove dissolved solids, but Reverse osmosis process is used for the removal of dissolved solids.

The allowed level of all the metal ions present in water is shown in the table 1.1. There are several metals present in water.

Table 1.1

Allowed level of metal ions in drinking water(WHO Organisation ,2009)

METALS	**VALUE**
Sodium	20mg/l
Zinc	5mg/l
Chloride	11-77mg/l
Fluoride	1.51
Arsenic	0.01mg/l
Manganese	0.15-2.56mg/l
Potassium	50mg/l
Lead	0.01mg/l
Cadmium	0.003mg/l
Mercury	0.001mg/l
Copper	2.0mg/l
Calcium	50mg/l
Iron	16.11-79.30mg/l

VETIVERU

Figure 1.1 Vetiveru plant

Vetiveria zizanoides belongs to the grass family, vetiver is perrineally growing grass that is indigenous to India. It is also known as khas in north and south india. This grass has tall stem, while the leaves are elongated, slender and somewhat firm.

Figure 1.2 Vetiveru Roots

Various tribes use the different parts of the grass for many of their ailments such as mouth ulcer, fever, boil, epilepsy, burn, snakebite, scorpion sting, rheumatism, fever, headache, etc. Apart from the medicinal uses, the culms along with the panicles form a good broom for sweeping. The culms and leaves are also extensively used by the tribes and villagers for thatching their huts, mud walls, etc. Some tribes (in Kerala) use the mats of the roots and leaves as bed for a cooling effect.

Many harmful elements and disease causing bacteria are present in sewage water, thus, with a lack of proper drainage, this sewage will mix with drain and rain water and flow toward low points which tend to be dams and lakes. It goes without saying that ultimately, this water will reach the sea and cause significant damage on route. The basic Vetiver Grass Technology or Vetiver System comprises a dense vetiver grass (*Vetiveria zizanioides)* hedgerow that is planted across the slope of the land or embankment. The hedgerow traps sediments, spreads out rain-water runoff, and provides through its roots significant reinforcement to the soil, (Grimshaw, 1993). The Vetiver system was first developed by the World Bank for soil and water conservation in India in the 1980's, (Troung, 1998).

The chemical constituents present in the plant are Vetiverol, Vetivone (Rangari *et al*.,)Khusimone, Khusimol, Vetivene, Khositone, Terpenes, Benzoic acid,Tripene-4-ol, ß-Humulene, Epizizianal, vetivenyl vetivenate, iso khusimol, Vetiver oils, vetivazulene(Sheng et al., 2004)Zizaene, prezizaene, b-vetispirene(Karanet al.,2013)Among these, the major active constituents identified are khusimol, vetivone, eudesmol, khusimone, zizaene, and prezizaene(Mitra et al., 2012)which are considered to be the fingerprint of the oil (Anon et al.,1976)(Martinez Et al.,2004)(Champagnat et al.,2006)

PHARMACOLOGICAL ACTIVITIES:

- Antioxidant Activity
- Antifungal Activity
- Antibacterial Activity
- Hepatoprotective Activity
- Antitubercular Activity
- Mosquito Repellent Activity
- Antihyperglycaemic Activity
- Antidepressant Activity

OBJECTIVES

- To prepare Green membrane and to minimize the hardness of water.
- To study the physiochemical parameters of water before and after treatment with membrane.
- Characterisation of the prepared membrane is studied by using FT IR, SEM, XRD, TGA and also Membrane Thickness, Pure water flux, Porosity of the membrane is studied.

Review of Literature

2. REVIEW OF LITERATURE

- In this we can review on different membrane processes and membrane reactors. The application of membrane processes in petrochemical industry, processes such as olefin/paraffin separation, light solvent separation, solvent dewaxing, phenol and aromatic recovery, dehydrogenation, oxidative coupling of methane and steam reforming of methane were discussed in detail. In this study waste water treatment using bioreactor were reviewed. (Ravanchi *et al.,*2009)

- In separation variety of technologies are available, each separation based on physical and chemical properties. Membrane separation technology has major change in processing of diary product. Membrane separations work on the basis of differences in size and shape of the molecules. In the dairy industry we can use reverse Osmosis (RO), Nanofiltration (NF), Ultrafiltration (UF) and Microfiltration (MF) processes and each processes is used for specific application.. Now-a-days wide pore UF process is used to develop enriched protein products, loose NF process is used to recover and purify Oligosaccharides, high pressure UF process is used to replace the conventional NF process used for concentrating dairy product streams. In the present paper, new developments in the application of membrane separation in the dairy industry are presented along with the experimental data from the research conducted by the authors.(Marella *et al*.,2013)

- Samples from mixing ponds which act as activated sludge are collected and being analyze using water analyzer method to obtain parameters such as BOD, COD, suspended solid, turbidity and pH. Wastewater sample from facultative ponds is also being analyzed than mix with activated sludge treated in the bioreactor. Result from lab-scale bioreactor is used in membrane bioreactor pilot plant system to treat the wastewater. Result from bioreactor treatment in pilot plant scale show a decrement 61.2 % of BOD and 58.9% of COD, suspended solid and turbidity is also reducing up 35.3% and 20.4% with pH in range of 5-9. After the wastewater was treated in the ultrafiltration membrane system, high quality water with total of deterioration for all parameter is up to 99.9% and pH up to 7.39. This results show that the membrane bioreactor treatment system is highly effective in treating POME (Hazlan bin haris., 2006)

- The textile industries are major consumers of water, dyes and other toxic chemicals. The effluents generated from each processing step are unutilized resources. The effluents if discharged without prior treatment become several deleterious effects on the environment. These can be observed in various innovative membrane based techniques. The present review paper thus elucidates the contributions of membrane technology towards textile effluent treatment and unexhausted raw materials recovery. Ultrafiltration and Nanofiltration are used to reuse possibilities of water recovered through membrane based techniques. Advantages and bottlenecks, such as membrane fouling associated with each of these techniques have also been highlighted. Finally, various accounts dealing with techno economic evaluation of these membrane based textile wastewater treatment processes have been provided.(Dasgupta *et al*.,2015)

- Nanofiltration (NF) membrane configuration for fluoride removal from groundwater has a key role in controlling operation performances and membrane fouling. The NF membrane surface was negatively charged above its isoelectric point (approximately at pH 5.9–6.4). The raw groundwater was initially adjusted to a neutral or alkaline pH to enhance the effect of electrical charge repulsion and improve the ion removal efficiencies by NF. Membrane in a parallel-linear arrangement was the preferable arrangement to remove fluoride and arsenic from low-salinity groundwater in rural areas. The NF system obtained fluoride and arsenic removal efficiencies of 70~73% and 92~94% respectively at

16◦C. The NF system water product cost are lowest with this optimal arrangement**. (**Xi Beidou *et al.,*2014)

- Reverse osmosis is a water purification technology that uses a semipermeable membrane. This membrane technology is not properly a filtration method. In reverse osmosis, an applied pressure is used to overcome osmotic pressure, a colligative property that is driven by chemical potential, a thermodynamic parameter. Reverse osmosis can remove many types of molecules and ions from solutions, and is used in both industrial processes and the production of potable water. The result is that the solute is retained on the pressurized side of the membrane and the pure solvent is allowed to pass to the other side. To be selective this membrane should not allow large molecules or ions through the pores holes, but should allow smaller components of the solution such as the solvent to pass freely. In the normal osmosis process, the solvent naturally moves from an area of low solute concentration high water potential, through a membrane, to an area of high solute concentration low water potential. The movement of a pure solvent is driven to reduce the free energy of the system by equalizing solute concentrations on each side of a membrane, generating osmotic pressure. Applying an external pressure to reverse the natural flow of pure solvent, thus, is reverse osmosis. The process is similar to other membrane technology applications. However, key differences are found between reverse osmosis and filtration. The main removal mechanism in membrane filtration is straining, or size exclusion, so the process can theoretically achieve perfect exclusion of particles regardless of operational parameters such as influent pressure and concentration. Moreover, reverse osmosis involves a diffusive mechanism, so that separation efficiency is dependent on solute concentration, pressure, and water flux rate (Crittenden., 2005).

- Cellulose acetate-starch membranes were prepared glycerol was added as a plasticizer additive via phase inversion method. Taguchi experimental design effects of total polymer, starch and glycerol concentrations besides the coagulation bath temperature on thickness, contact angle, permeability and biodegradability of the membranes were investigated. Fourier-transform infrared and scanning electron microscopy is used to determine the interactions between polymers and the membranes morphology. Three concentration factors were found to be effective parameters in changing the membranes thickness and permeability. The membranes hydrophilicity was affected by total polymer and starch concentration, while their biodegradability was only influenced by the starch concentration.(Zarei *et al.,*2013)

- In the absence and presence of membrane is prepared and the pore is formed by former by solution blending and ultrafiltration set up was carried out. The application of the characterized CA/PMMA blend membranes for the separation of proteins such as Bovine Serum albumin, Egg Albumin, Pepsin, and Trypsin, and toxic heavy metals such as Cu(II), Ni(II), and Zn(II) using polyethyleneimine as complexing agent have been attempted and the results indicate the efficiency of the ultrafiltration blend membranes.(Vidya *et al.,201*0)

- We can separate clean water from oil/water emulsion can be used to separate by ultrafiltration technique. Recent changes in sewer discharge limits have created a need to generate cleaner water. The various type of membrane filtration are used, all these nanofiltration is more effective recycling water from oil solutions or clean enough water for discharge. Compare both ultrafiltration and nanofiltration concentration tests indicate that nanofiltration can effectively treat industrial machining coolant. Nanofiltration flux was slightly lower at lower pressures,, mostly due to the higher membrane resistance of the nanofilter. At higher operating pressures, the nanofiltration mass transfer coefficient increased while the ultrafiltration transfer coefficient decreased. Nanofilter should observed lower levels of COD and conductivity as compared to the ultrafilter the quality of nanofiltration permeate resembled that of ultrafiltration permeate near the end of the concentration cycle.(Park *et al*.,2001)

- Approximately 25% of the world’s population has no access to clean and safe drinking water. Many of these water sources are contaminated by natural means or through human activity in most parts of the world. In industries need clean water for product development and machinery operation. Due to industry expansion freshwater supplies are being contaminated and scarce the demand for potable water is increasing. Water contamination in modern farming like agrochemicals artificial chemical fertilizers, pesticides, fungicides, and herbicides. Other areas where the groundwater contaminated with fluorides, arsenic, and radioactive material occur in the soil. The liver or kidneys, or both organs may fail due to the human body is detoxify and excrete toxic chemicals. With continual consumption of polluted water body conditions are unfavourable, depending on the type of pollutants and toxin, liver, cardiac, brain, or renal failure. Water purification and ultrafiltration are used to remove the toxic chemicals for most of the household filters. We are use of reverse osmosis technology and ion exchange methods now a days reverse osmosis should remove more than 95% of toxic chemicals. (Wimalawansa,2013)

- Usually for human consumption water purification is the removal of contaminants from untreated water to produce drinking water. In these process suspended solids, bacteria, algae, viruses, fungi, minerals such as iron, manganese and sulphur, and other chemical pollutants are removed. Water contains many types of dissolved salt (mainly NaCl) in it and the process which used to remove these salt and the amount of salt present in water is measured in term of salinity it is measured in ppm.. Fresh water, brackish water and sea water of salinity are divided. Only fresh water is fit for drinking and other purposes. Reverse Osmosis is best used method for conversion of brackish and sea water into fresh water.(Kaushik., 2012)

- More than one billion people lack access to safe drinking water. In India most of disease caused by water. Many people are conscious about contaminated drinking water. The paper is a primary data based, collected from Warangal, to assess the saleability of Pure it brand Water Purifier. (kumar and rao.,2012)

- Cooling water plays an important role in many industrial processes. To maintain proper cooling water quality, fresh water must be added to the circulating system and to compensate for water loss. We evaluated the feasibility of forward osmosis using rainwater as the makeup water source for the cooling water. Although the changes in pH had a small direct effect on the water and the membrane flux increased approximately and increased the temperature from 3°C to 50°C 10 times. During the operation, no decreases in flux were observed as a result of membrane fouling.(Wang *et al*.,2014)

- Ground water discharge to a stream has important implication for ground water flow, especially with respect to contaminant transport. Measurements of ground water discharge were completed using current meter measurements, stream temperature and heat transport modeling of measured temperature. An area-averaged estimate of ground water flow is studied in first two techniques. Stream temperature and stream flow surveys were combined using a simple heat-balance to yield a higher-resolution practically it is obtained with current meters alone. This a cost-effective method of quantifying ground water discharge in streams.(Becker *et al.,*2004)

- When membrane performance and applicability was investigated for reverse osmosis (RO) desalination of saline water. In this process many synthetic membrane were prepared and characterized for the desalination process. Different multilayer composite membranes of reverse osmosis parameters were evaluated compared to a reference membrane of desalination. Values of both salt rejection and water flux were measure of membrane efficiency. In this work, samples of CA/PEG and PVA/CA/PEG membranes were used for the reverse osmosis process of different feed concentrations of groundwater, brackish, highly saline and also extremely saline water and the concentrations were determined by conductivity measurements. We are prepared synthetic membranes, the antimicrobial sustainability was also evaluated where prospective function against gram +ve and gram -ve was depicted.(Hassanien *et al.,*2013)

- Transport of phenol through a flat sheet supported liquid membrane containing cooking oil was investigated.. It was found that these parameters strongly influence phenol removal efficiency grape seed oil as liquid membrane, feed pH of 2.0, initial phenol concentration of 100 mg/L, stirring speed of 350 rpm, and 0.2M sodium hydroxide as effective stripping agent were found as the best conditions for greater phenol transport. phenol was completely removed from the feed phase to strip phase after 10.5 h. According to stability experiments, it was observed that the supported liquid membrane is stable after 22 h. Thus, the use of cheap, nontoxic, and naturally oil as a novel and green membrane for recovery of phenol from wastewater was demonstrated.(peydayesh *et al*.,2014)

- Membrane application in surface water treatment provides many advantages. Increases in hydraulic resistances, operational and maintenance costs, deterioration of productivity and frequency of membrane regeneration problems. We are discussing natural organic matter (NOM) and its components as the major membrane foulants that occur during the water filtration process the current techniques used to characterized fouling mechanisms, fouling mechanisms relating to reversible and irreversible of natural organic matter.(Zularisam et al., 2006)

- Microfiltration membrane has been prepared by blending polymer and Cellulose acetate (CA) was blended with polyethyleneimine (PEI), in a mixture of solvents. PEI can supply coupling sites for ligands and it should be used as ligands for metal chelating and these effects of the time were induced by water vapor,and crosslinking agent on membrane were investigated. Specific surface area of the prepared blend membranes are 12.04–24.11m2/g and pure water flux (PWF) of 10–50 ml/cm2 min with porosity of 63–75%. Cu2+ ion on the blend membrane have maximum adsorption capacity 7.42 mg/g dry membrane.(Chen *et al.,*2004)

- Cellulose acetate is used to prepare membrane, in these have LiCl and $CaCl_2$ as porogens and methyl(S)lactate as a solvent. In these membrane are biodegradable, non-toxic and non-volatile organic compounds. Phase inversion technique is used to prepare flat sheet ultrafiltration membrane. In water treatment industry these parameters are ideal range. Improvement of pollutant degree and ecotoxicity of the process was evaluated by ‘green’ metrics by the P (pollutants, persistent and bioaccumulative) and E (ecotoxicity) parameters. This study represents a step ahead towards the production of ultrafiltration polymeric membranes by a‘greener’ process than current methods.(Gonzalez *et al*.,2011)

- In most cases waste water treatment is used because depletion of fresh water to focus water recovery, reuse, and recycling this process is mainly based on this technique.
  Compare with biological treatment processes, membrane technology has gained wide range of applicability and these have been established by various investigations. Parameters and effluent quality are used to evaluate primary findings on these technologies. Factors such as loading rates, retention time, cross-flow velocities, membrane types, membrane fouling, and backwashing, etc. (Visvanathan *et al*.,2000)

- Membrane foulants have external foulants and internal foulants these are systematically characterized in a full-scale membrane bioreactor (MBR) for supermarket wastewater treatment in this study. For this purpose we can use these technique to characterised the membrane such as three dimensional excitation emission matrix (EEM) fluorescence spectroscopy, gel filtration chromatography(GFC), Fourier transform infrared (FT-IR) spectroscopy, scanning electron microscopy (SEM) and energy diffusive X-ray. Both external and internal foulants were identified as protein-like substances and soluble microbial by-product. FT-IR analysis is used to identify the oil substance on the fouled membranes. SEM and EDX analyses indicated the that not only organic substances but also inorganic elements including Mg, Ca, Na, Al, K, and Si.(Zhu *et al*.,2011)

- Now a days removing solids is an major task when recirculating water an aquaculture system. Dissolved solids production directly from particulate solids as well as by fish is a function of time. These contaminants can indirectly affect the fish both biologically and physically. So we can use new technology for membrane processes is used to remove fine particles and dissolved matter.(chiam and sarbatly.,2011)

- Submerged membrane adsorption bio-reactors were investigated as a new pre-treatment for seawater reverse osmosis desalination. These are very useful and these were tested with different doses of powder activated carbon. Without any membrane damage the biofilm formation on membrane was reduced. Powder activated carbon also helped to remove low molecular weight. A small amount of powder activated carbon (2.4–8.0 g of PAC/m3 of seawater) was sufficient to reduce biofouling. (Jeong *et al*.,2014)

- Water is essential for human survival and these can be different purity in different surface of the earth. Different types of contaminants are present in the earth. These can be minimized with the help of membrane processes like microfiltration, ultrafiltration, nanofiltration and reverse osmosis these are all advanced physical treatment of water for drinking purposes as well as for agro industrial sectors. Characterisation methods play an important role for the selection of the more appropriate membrane for the above applications.(Bottino *et al*.,2009)

- Reverse osmosis process for purification of Tehran refinery oily wastewater is presented. The effects of different operating parameters on permeation flux and TDS rejection were investigated. Taguchi method was used initially to plan a minimum number of experiments. Analysis of variance was applied to calculate sum of square, variance, ratio of factor variance to error variance and contribution percentage of each factor on response. The results showed that TMP and temperature have significant effects on the response. Permeation flux was found to increase with increasing Tran's membrane pressure, cross flow velocity and feed temperature at constant feed concentration but rejection slightly decreases. The p^H effects were found to be complex. By increasing acidic and basic nature of the feed, permeation flux was found to increase but rejection decrease. At original oily wastewater composition, high rejection of TDS (87%), COD (95%),BOD (95%), TOC (90%), turbidity (82%) and oil and grease content (87%) along with complete rejection of colour, free oil and TSS were achieved with a reasonably high permeation flux of 50 L/m2 h. Also, the results show that cake filtration model can well predict the flux decline (Salahi *et al*.,2012).

- A report of physico-chemical study of the water samples taken from the region presented here. Twenty five water samples are subjected to analysis like odour, taste, transparency, pH, TDS, hardness, and conductivity. The calcium, magnesium, potassium and sodium count obtained from the study of these samples has given the information regarding the suitability of the water for drinking purpose. The drop in mineral contents after reverse osmosis treatment is critically examined and the pros and cons are identified. While the pros are appreciated, remedies to cons are also suggested possible (Bhatt *et al*.,2011).

- Tunisian norms for drinking water tolerates a maximum TDS of 1.5 g/L and the domestic water presents usually salinity greater than 500 mg/L. In the last years, several small capacity reverse osmosis desalination prototypes have been marketed. The reverse osmosis membrane unit used in this research produces 10–15 L/h of treated water with a recovery rate between 25 and 40% and salt rejection in the order of 90%. The salinity of the tested domestic water is between 0.4 and 1.4 g/L. Water pre-treatment is composed of three filtration operations cartridge filter, granulate active carbon filter, and 5 lm cartridge filter. Pre-treated water is pumped through reverse osmosis membrane membrane with a maximum pressure of 6 bars. At the 4th year, the reverse osmosis membrane unit's performance was substantially decreased. The recovery rate and salt rejection fall down about 50 and 100%, respectively, and the pressure drop increases from 1 to 2.1 bars. An autopsy of the used reverse osmosis membrane membrane was done by different analysis techniques such as SEM/EDX, AFM, XRD, and FTIR spectroscopy.

  The analysis of the membrane surface shows a 2 lm deposit film indicating a fouling phenomenon. The SEM photos show deterioration on the active layer of the membrane which seems to be attacked by the tap water chlorine. X-ray diffraction and FTIR show that the deposit collected on the used membrane contains organic and mineral Gypsum, SiO2, and clays materials. The desalinated water cost TDS < 100 mg/L is less than 0.02 US$/L. It is about one-seventh of the mineral water price (Hassanien *et al.,* 2013).

- Research has shown biological filtration can be a successful treatment for manganese (Mn) removal from groundwater and surface water. In this study, bench-scale direct bio filtration was used to remove Mn and dissolved organic carbon from a p^H 6 surface water source in Halifax, Canada. The removal of Mn in pH 6 surface water was significantly (α = 0.05) removed with 200-300 µg/L phosphorus (P), and 500 µg/L hydrogen peroxide (H_2O_2). DOC removal was significantly (α = 0.05) improved with granular activated carbon (GAC) media, P enhancement at 200-300 µg/L, and H_2O_2 enhancement at 500 µg/L. Mn was likely removed by biological oxidation and physical adsorption to biogenic Mn and iron (Fe) oxides. These results show direct bio filtration of surface water at pH 6 can remove Mn below the 50 µg/L aesthetic guideline from a Mn loading of over 1 mg/L. Further research is required to verify the microbial mechanism of Mn removal (Granger.,2013).

Aim and Scope

AIM AND SCOPE

The aim of this study is to prepare a Green membrane by low cost and Eco friendly method.

This prepared membrane can be used for the water purification.

Materials and Methods

3. MATERIALS AND METHODS

3.1 PREPARATION OF MEMBRANE

Chemicals required:

Celluloseacetate, Dimethylacetamide, Vetiveru, Diethyl succinate(Biosolvent), BDH Indicator, ammonium chloride and 1.25g of magnesium salt of EDTA,0.01N KCl, Eriochrome black-T indicator, $CaCO_{3,}$ Murexide indicator, Sodium hydroxide, phosphate buffer, magnesium sulphate, calcium chloride , ferric chloride, potassium dichromate 0.250m, Standard ferrous ammonium sulphate (FAS) 0.25N, Ferroin indicator, con sulphuric acid, Silver sulphate crystals ,Mercuric sulphate crystals , Potassium chromate indicator, Silver nitrate solution (0.014N), sodium fluoride, Zirconyl chloride octahydrate, Zirconyl acid-SPADNS reagent.

3.2 MEMBRANE PREPARATION (I)

1g of cellulose acetate was mixed with 6ml of dimethyl acetamide followed by 50mg of vetiveru and then was subjected to vigorous stirring for 40mins using a magnetic stirrer at 30^{o}c.The solution was completely dissolved by stirring and then coated with flat tiles. Then the coated solution was immersed into water for 2 to 3 mins. Afterwards membrane were peeled out and then dried for half an hour at room temperature in open air atmosphere. The membrane thickness was 0.55mm.

3.3 MEMBRANE PREPARATION (II)

1g of cellulose acetate was mixed with 6ml of diethyl succinate (biosolvent) followed by vigorous stirring with the help of magnetic stirrer at 25^{o}c. After 40mins the solution was completely dissolved and then coated with flat tiles. Then the coated solution was immersed into water for 5mins.Afterwards membrane were peeled out and then dried for half an hour at room temperature in open air atmosphere. The membrane thickness was 0.76mm.

The prepared membrane prepared were characterised by the following methods.

3.4 COMPACTION

The thickness of the cast membrane was measured using WIRA Digital thickness tester (WIRA Instrument, UK).The thickness of the membrane used in this study was 0.55±0.2mm. The prepared membrane were cut into effective membrane area 38.465 cm^2 and it was initially pressured with distilled water at 250mmHg for 2min 45sec These pre-pressurized membrane were used in subsequent water filtration process.

3.5 PURE WATER REFLUX

Membrane after compaction was subjected to pure water flux studies at a trans-membrane pressure of 250mm/Hg. The pure water flux is determined by

$$J_W = Q/A\Delta V \qquad \rightarrow \quad (1)$$

Where, J_W-water flux ($ml/cm^2 min$)

Q-quality of water permeate (ml)

A-Membrane area (cm^2)

T- Sampling time (hours)

3.6 WATER UPTAKE

Percent water content of the membranes was obtained after soaking membranes in water for 24hrs and the membranes were weighed followed by mapping it with filter paper. The wet membranes were placed in opened air for 1hr and the dry weights of the membranes were determined. From the wet and dry weights, percentage water content was determined by

%Water content=wet sampleweight-Drysample weight/wet sample weight×100

→ (2

3.7 THERMAL STABILITY PROFILE (TGA)

Thermal gravimetric analyser were used to detect the weight loss and the model using (Ex star SII TG/DTA).The heating rate of the thermal stability analyser was adjusted between 30^{o}c(room temperature) to 210^{o}c.

3.8 SCANNING ELECTRON MICROSCOPE (SEM)

The surface morphology of the prepared membrane (I and II) was studied using Scanning Electron Microscope (SEM) XL 20 PHILPS. Photographs were taken in 200nm and these are 25.00 magnification.

3.9 FT-IR SPECTROSCOPY

To study the membrane structure FT-IR spectra of the membrane is also studied (Model: IR Affinity –I) It was used to characterised the major functional groups of the sample.

3.10 X-RAY DIFFRACTION

X ray diffraction studies were carried out using (pan analytical) for the prepared membrane.

3.11 FILTRATION OF MEMBRANE

The water was filtered using carbon rod covered with prepared membrane(I & II). The sample water was added drop wise to it. So that the water is filtered through the membrane. The filter water is collected and analysed the physio- chemical parameters.

3.12 WATER ANALYSIS

The parameters analysed to assess the water quality are

pH, Electrical Conductivity Total Dissolved Solids,Total Hardness, Calcium Hardness, Magnesium Hardness, Dissolved Oxygen (D.O), Biological Oxygen Demand (BOD), Chemical Oxygen Demand (COD),Carbonate, Bicarbonate, Chlorides, Fluoride, Sodium, Bacteria and Fungi.

- **pH:**

The effect of pH on the chemical and biological properties of liquids makes its determination very important. It is one of the most important parameter in water chemistry and is defined as -log [H^+], and measured as intensity of acidity or alkalinity on a scale ranging from 0-14. If free H^+ are more it is expressed acidic (i.e. pH<7), while more OH^- ions is expressed as alkaline (i.e. pH> 7).

In natural waters pH is governed by the equilibrium between carbon dioxide/bicarbonate/carbonate ions and ranges between 4.5 and 8.5 although mostly basic. It tends to increase during day largely due to the photosynthetic activity (consumption of carbon-di-oxide) and decreases during night due to respiratory activity. Waste water and polluted natural waters have pH values lower or higher than 7 based on the nature of the pollutant.

Apparatus required:

- **pH indicator (BDH) method:** BDH Indicator (Universal Indicator) and test tubes.
- **Electrometric method:** Glass electrode, reference electrode (mercury/calomel or silver/silver chloride) and pH meter. The colorimetric indicator method can be used only for approximate pH values.

Procedure:

- **Colorimetric method:** About 10ml of the sample is taken in a wide mouth test tube, 0.2ml of BDH indicator is added, and shaken gently. The colour developed is matched with the chart and the pH is noted.

- **ELECTRICAL CONDUCTIVITY (EC) :**

Principle:

Electrical conductivity (EC) is used to determine the cell constant value.

Apparatus:

EC meter and Thermometer

Reagent:

0.01N Potassium chloride solution: Dry a small quantity of A.R. grade Potassium chloride at 60oC for 2 hours. Weigh 0.7456 gm of it and dissolve in freshly prepared distilled water and make to one litre. This solution gives and electrical conductivity of 1411.8 x 10-3 i.e.1.41 dS/m at 25°C.

Procedure:

- Calibrate the conductivity cell with the help of standard KCL solution and determine the cell constant.
- Dip the conductivity cell assembly in water sample taken in a 50 or 100 ml beaker and record the conductivity. If the value is too low, change the adjustment accordingly. Record the temperature of water during the test.
- Observed values of EC are multiplied by the cell constant (usually given on conductivity cell) and a temperature factor to express results at 25°C.
- Remove the cell from sample water, clean with distilled water and dip into a beaker of distilled water. EC is expressed as dS/m.
- Keep the conductivity cell in distilled water when not in use.

Calculations:

The cell constant K is given by Cell constant (K) =ECw25 = ECT x K x ft

Where,

ECw25 is the conductivity of the water at 25°C.

ECT is apparent conductivity of water as measured.

K is the cell constant

Ft is temperature correction factor.

- **TOTAL HARDNESS**

Hardness is predominantly caused by divalent cations such as calcium, magnesium, alkaline earth metal such as iron, manganese, strontium, etc. The total hardness is defined as the sum of calcium and magnesium concentrations, both expressed as $CaCO_3$ in mg/L. Carbonates and bicarbonates of calcium and magnesium cause temporary hardness. Sulphates and chlorides cause permanent hardness.

Principle:

In alkaline conditions EDTA (Ethylene diamine tetra acetic acid) and its sodium salts react with cations forming a soluble chelated complex when added to a solution. If a small amount of dye such as Eriochrome black-T is added to aqueous solution containing calcium and magnesium ions at alkaline pH of 10.0 ± 0.1, it forms wine red colour. When EDTA is added as a titrant, all the calcium and magnesium ions in the solution get complexed resulting in a sharp colour change from wine red to blue, marking the end point of the titration. Hardness of water prevents lather formation with soap rendering the water unsuitable for bathing and washing. It forms scales in boilers, making it unsuitable for industrial usage. At higher pH>12.0, Mg^{++} ion precipitates with only Ca^{++} in solution. At this pH, murexide indicator forms a pink colour with Ca^{++} ion. When EDTA is added Ca^{++} gets complexed resulting in a change from pink to purple indicating end point of the reaction.

Apparatus required

Burette, pipette, conical flask, beakers etc.

Reagents:

- **Buffer solution**: 16.9 g of ammonium chloride and 1.25g of magnesium salt of EDTA is dissolved in 143ml of concentrated ammonium hydroxide and diluted to 250ml with distilled water.
- **Eriochrome black-T indicator**: 0.5 g of Eriochrome black-T indicator is dissolved in 100g of triethanolamine.

- **Standard EDTA titrant:** 0.01M or Na AR grade EDTA is dissolved in distilled water and diluted to 1000ml and is standardised against standard calcium solution, 1ml = 1mg $CaCO_3$.
- **Standard Calcium Solution:** 1.0g of AR grade $CaCO_3$ is weighed into a 250ml conical flask, to which 1:1HCl is added till all $CaCO_3$ is dissolved completely. 200ml of distilled water is added and boiled to expel carbon-di-oxide, and diluted to 1000ml. 1ml = 1mg $CaCO_3$

Procedure:

Exactly 50ml of the well-mixed sample is pipette into a conical flask, to which 1ml of ammonium buffer and 2-3 drops of Eriochrome black -T indicator is added. The mixture is titrated against standard 0.01M EDTA until the wine red colour of the solution turns pale blue at the end point.

Calculation:

$$\text{Total Hardness} = \frac{\text{Volume of titrant x 1 x 1000}}{\text{Volume of Sample taken}}$$

- **CALCIUM HARDNESS**

The presence of calcium (fifth most abundant) in water results from passage through or over deposits of limestone, dolomite, gypsum and such other calcium bearing rocks. Calcium contributes to the total hardness of water and is an important micro-nutrient in aquatic environment and is especially needed in large quantities by molluscs and vertebrates. It is measured by EDTA titrimetric method. Small concentration of calcium carbonate prevents corrosion of metal pipes by laying down a protective coating. But increased amount of calcium precipitates on heating to form harmful scales in boilers, pipes and utensils.

Principle:

When EDTA (Ethylene-diamine tetra acetic acid) is added to the water containing calcium and magnesium, it combines first with calcium. Calcium can be determined directly with EDTA when pH is made sufficiently high such that the magnesium is largely precipitated as hydroxyl compound (by adding NaOH and isopropyl alcohol). When murexide indicator is added to the solution containing calcium, all the calcium gets complexed by the EDTA at pH 12-13. The end point is indicated from a colour change from pink to purple.

Apparatus required:

Burettes, pipette, conical flask, beakers and droppers.

Reagents:

- **Sodium hydroxide (8%):**8g of sodium hydroxide is dissolved in 100ml of distilled water.
- **Murexide indicator (ammonium purpurate):**0.2 g of murexide is ground well with 100g of sodium chloride thoroughly.
- **Standard EDTA titrant, 0.01M:**3.723 g of EDTA (disodium salt) is dissolved in distilled water and made up to 100ml with the same.

Procedure:

A known volume (50ml) of the sample is pipetted into a clean conical flask, to which 1ml of sodium hydroxide and 1ml of isopropyl alcohol is added. A pinch of murexide indicator is added to this mixture and titrated against EDTA until the pink colour turns purple.

Calculation:

$$\text{Calcium as calcium carbonate} = \frac{\text{Volume of titrant x 1 x 1000}}{\text{Volume of Sample taken}}$$

- **MAGNESIUM HARDNESS**

Magnesium is a relatively abundant element in the earth's crust, ranking eighth in abundance among the elements. It is found in all natural waters and its source lies in rocks, generally present in lower concentration than calcium. It is also an important element contributing to hardness and a necessary constituent of chlorophyll. Its concentration of water greater than 125 mg/L can influence cathartic and diuretic actions.

Principle:

Magnesium hardness can be calculated from the determined total hardness and calcium hardness.

Calculation:

$$\text{Magnesium hardness} = (\text{Total hardness} - \text{Calcium hardness})$$

High concentration of magnesium proves to be diuretic and laxative, and reduces the utility of water for domestic use while a concentration above 500 mg/L imparts an unpleasant taste to water and renders it unfit for drinking. Chemical softening, reverse osmosis and electro dialysis or ion exchange reduces the magnesium hardness to acceptable levels.

- **BIOLOGICAL OXYGEN DEMAND (BOD):**

Biological Oxygen Demand is the amount of oxygen required by microorganisms for stabilizing biologically decomposable organic matter (carbonaceous) in water under aerobic conditions. The test is used to determine the pollution load of wastewater, the degree of pollution and the efficiency of wastewater treatment methods. 5-Day BOD test being a bioassay procedure (involving measurement of oxygen consumed by bacteria for degrading the organic matter under aerobic conditions) requires the addition of nutrients and maintaining the standard conditions of pH and temperature and absence of microbial growth inhibiting substances.

Principle:

The method consists of filling the samples in airtight bottles of specified size and incubating them at specified temperature (20 °C) for 5 days. The difference in the dissolved oxygen measured initially and after incubation gives the BOD of the sample.

Apparatus required:

BOD bottles - 300ml capacity, air incubator - to be controlled at 20^0C-\+ 1^0C, oximeter and magnetic stirrer.

Reagents:

Preparation of dilution water:

To 1000ml of water, 1ml each of phosphate buffer, magnesium sulphate, calcium chloride and ferric chloride solution is added, before bringing it to 20°C and aerating it thoroughly.

Procedure:

The sample having a pH of 7 is determined for first day DO. Various dilutions (at least 3) are prepared to obtain about 50% depletion of D.O. using sample and dilution water. The samples are incubated at 20^0C for 5 days and the 5th day D.O is noted using the oximeter. A reagent blank is also prepared in a similar manner.

Calculation:

$$\text{BOD} = \frac{(D_1 - D_2) - (B_1 - B_2) \times f}{p}$$

D_1 - 1st day D.O of diluted sample

D_2 - 5th day D.O of diluted sample

P - decimal volumetric fraction of sample used.

B_1 - 1st day D.O of control

B_2 - 5th day D.O of control

- **CHEMICAL OXYGEN DEMAND (COD):**

Chemical oxygen demand (COD) is the measure of oxygen equivalent to the organic content of the sample that is susceptible to oxidation by a strong chemical oxidant. The intrinsic limitation of the test lies in its ability to differentiate between the biologically ox disable and inert material. It is measured by the open reflux method.

Principle:

The organic matter in the sample gets oxidized completely by strong oxidizing agents such as potassium dichromate in the presence of conc. sulphuric acid to produce carbon-di-oxide and water. The excess potassium dichromate remaining after the reaction is titrated with Ferrous Ammonium Sulphate (FAS) using ferroin indicator to determine the COD. The dichromate consumed gives the oxygen required for the oxidation of the organic matter.

Apparatus required:

Reflux apparatus, Nessler's tube, Erlenmeyer flasks, hot plate and lab glassware.

Reagents:

- **Standard potassium dichromate solution (0.250M):** 12.25g of potassium dichromate dried at 103°C for about 2 hours is dissolved in distilled water and made up to 1000ml.
- **Standard ferrous ammonium sulphate (FAS) 0.25N:**98g of FAS is dissolved in minimum distilled water to which 20ml of conc. sulphuric acid is added and made up to 1000ml using distilled water to give 0.25N of ferrous ammonium sulphate.

- **Ferroin indicator:** 1.485g of 1, 10-Phenanthroline monohydrate and 695mg of ferrous sulphate is dissolved in 100ml of distilled water.
- **Conc. sulphuric acid**
- **Silver sulphate crystals**
- **Mercuric sulphate crystals**

Procedure:

15ml of conc. sulphuric acid with 0.3g of mercuric sulphate and a pinch of silver sulphate along with 5ml of 0.025M potassium dichromate is taken into a Nessler's tube. 10ml of sample (thoroughly shaken) is pipetted out into this mixture and kept for about 90 minutes on the hot plate for digestion. 40ml of distilled water is added to the cooled mixture (to make up to 50ml) and titrated against 0.25M FAS using ferroin indicator, till the colour turns from blue green to wine red indicating the end point. A reagent blank is also carried out using 10ml of distilled water.

Calculation:

$$\text{COD} = \frac{(\text{Blank reading - Sample reading}) \text{ X N X F X } 1000}{\text{Volume of Sample taken}}$$

To calculate F,

$$F = \frac{10000}{\text{Titrant value of blank.}}$$

- **CHLORIDES**

The presence of chlorides in natural waters can mainly be attributed to dissolution of salt deposits in the form of ions (Cl^-). Otherwise, high concentrations may indicate pollution by sewage, industrial wastes, intrusion of seawater or other saline water. It is the major form of inorganic anions in water for aquatic life. High chloride content has a deleterious effect on metallic pipes and structures, as well as agricultural plants. They are calculated by Argentometric method.

Principle:

In alkaline or neutral solution, potassium chromate indicates the endpoint of the silver nitrate titration of chlorides. Silver chloride is quantitatively precipitated before the red silver chromate is formed.

Apparatus required:

Burette, pipette, conical flask, beakers etc

Reagents:

- **Potassium chromate indicator solution:** 50g of potassium chromate is dissolved in minimum amount of distilled water and silver nitrate is added drop wise till a red precipitate is formed. The mixture is allowed to stand for about 12 hours and diluted to 1000ml with distilled water.
- **Silver nitrate solution (0.014N):**2.395g of silver nitrate is dissolved in distilled water and made up to 1000ml.

Procedure:

A known volume of filtered sample (50ml) is taken in a conical flask, to which about 0.5ml of potassium chromate indicator is added and titrated against standard silver nitrate till silver dichromate ($AgCrO_4$) starts precipitating.

Calculation:

$$\text{Sodium chloride} = \frac{\text{Volume of Silver nitrate x normality}}{\text{Volume of Sample taken}}$$

- **FLUORIDES**

Fluorides have dual significance in water supplies. High concentration causes dental fluorosis and lower concentration (<0.8 mg/L) causes dental caries. A fluoride concentration of approximately 1mg/L in drinking water is recommended. They are frequently found in certain industrial processes resulting in fluoride rich wastewaters. Significant sources of fluoride are found in coke, glass and ceramic, electronics, pesticide and fertiliser manufacturing, steel and aluminium processing and electroplating industries. It is calculated by spadns method.

Principle:

The colorimetric method of estimating fluoride is based on the reaction of fluorides (HF) with zirconium SPADNS solution and the 'lake' (colour of SPADNS reagent), which is greatly influenced by the acidity of the reaction mixture. Fluoride reacts with the dye ‘lake’, dissociating (bleaching) the dye into a colourless complex anion (ZrF_6^{2-}). As the amount of fluoride increases, the colour produced becomes progressively higher or of different hue.

Apparatus required:

Spectrophotometer and lab glassware.

Reagents:

Standard Fluoride Solution:

- **Stock:**221.0mg of AR grade sodium fluoride was dissolved in distilled water and made up to 1000ml to give 1ml = 100 μ g of F^-
- **Working Standard:** 100ml of the stock fluoride was diluted to 1000ml to give 1ml = 10 μg of fluoride.
- **SPADNS Solution:** 958mg of SPADNS is dissolved in 500ml of distilled water.

- **Zirconyl-acid reagent:** 133mg Zirconyl chloride octahydrate ($ZrOCl_2 .8H_2O$) was dissolved in about 25ml of distilled water. 350ml of conc. HCl was added and diluted to 500ml with distilled water.
- **Zirconyl acid-SPADNS reagent:** Equal volume of SPADNS and zirconyl acid reagent was mixed.

Procedure:

A standard graph is prepared by using fluoride concentrations ranging from 0.005 mg/L to 0.150 mg/L at 570nm. A reference solution is prepared by adding 4ml of acid zirconyl-SPADNS reagent to 21ml of distilled water. A known volume of filtered sample (21ml) is taken in a test tube,4ml of acid zirconyl-SPADNS reagent is added to the sample along with a reference solution. The mixture is left for about 30 min for complete colour development and the optical density is read at 570nm.

Calculation:

$$\text{Fluoride} = \frac{(\text{O.D sample})(\text{Conc. of the Standard})(1000)}{(\text{O.D Standard})\ (\text{Sample taken})}$$

CARBONATES AND BICARBONATES:

Principle :

Carbonate and bicarbonate ions in the sample can be determined by titrating it with against standard sulphuric acid (H_2SO_4) using phenolphthalein and methyl orange as indicators. Addition of phenolphthalein gives pink red colour in the presence of carbonates and titration with H_2SO_4 converts these CO_3 into HCO_3 and decolourises the red colour.

Thus the carbonates neutralization is only half way. These carbonates along with the already present ones are then determined by continuing the titration using methyl orange indicator which gives yellow colour in presence of bicarbonates. On complete neutralization of bicarbonates the yellow colour will change to red.

Obviously the bicarbonate titre value will be less if carbonates were not present. (absence of pink colour). In such a situation, either the same aliquot is used for bicarbonate titration or a fresh sample is analyzed for this. If carbonates are present and neutralized, the volume of H_2SO_4 used in the first phase (carbonate titration) is to be doubled to get the actual volume needed for complete neutralization of the carbonates.

Reagents :

- Saturated H_2SO_4 (0.01N): Carefully add 2.8 ml of conc. H_2SO_4 to one litre volumetric flask and dilute to one litre with distilled water, the strength will be approximately 0.1N H_2SO_4. Dilute 100 ml of this solution to 1 litre to obtain 0.01N H_2SO_4. Standardize it against primary standard, Na_2CO_3.

- Standard Na2CO3 (0.01N): Dissolve 5.3 gm of A.R. grade Na_2CO_3 in one litre volumetric flask with distilled water, the strength will be 0.1N Na_2CO_3. Dilute 100 ml of this solution to get 0.01N. This may be used for standardization of 0.01N H_2SO_4.

- Phenolphthalein (0.25%): Dissolve 25 gm of pure Phenolphthalein powder in 100 ml of 60% ethyl alcohol.

- Methyl Orange (0.50%): Dissolve 0.5 gm of dry methyl orange powder in 100 ml of 95.0% ethyl alcohol.

Procedure:

- Transfer 25 ml of water sample to a 150 ml conical flask. Add 2-3 drops of Phenolphthalein.

- If pink red colour appears, titrate it against standard H_2SO_4 till colour disappears. The burette reading (volume used) is designated as Y ml.

- To this colourless solution or in original sample (25 ml) add 2- 3 drops of methyl orange. This will develop the yellow colour.

- Again titrate with standard H_2SO_4 till colour changes from yellow to rosy red. Record the volume of H_2SO_4 as Z ml. This volume corresponds to initial carbonate changed to bicarbonates plus initial bicarbonates present in irrigation water.

- Run a blank (25 ml distilled water) and subtract from the titre value to avoid error due to any impurity of chemicals.

Results and Discussion

FT-IR spectrum of cellulose Acetate

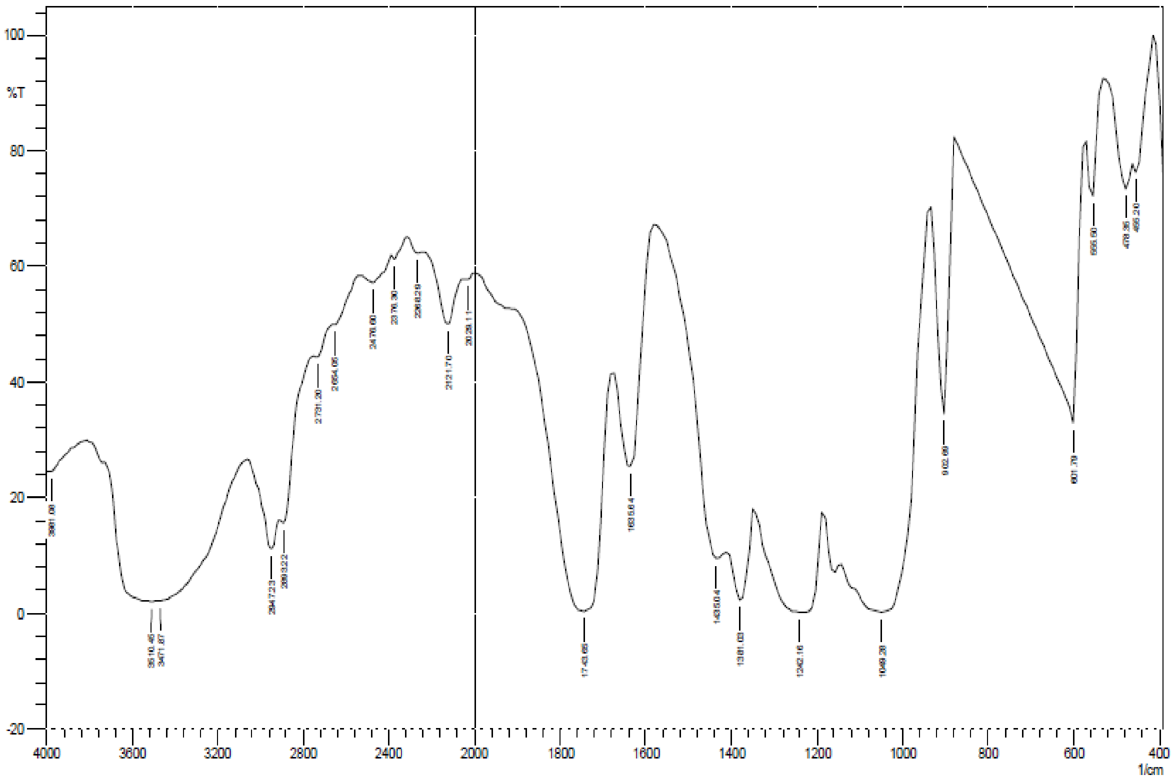

Figure 5.1 FTIR Spectrum of cellulose Acetate

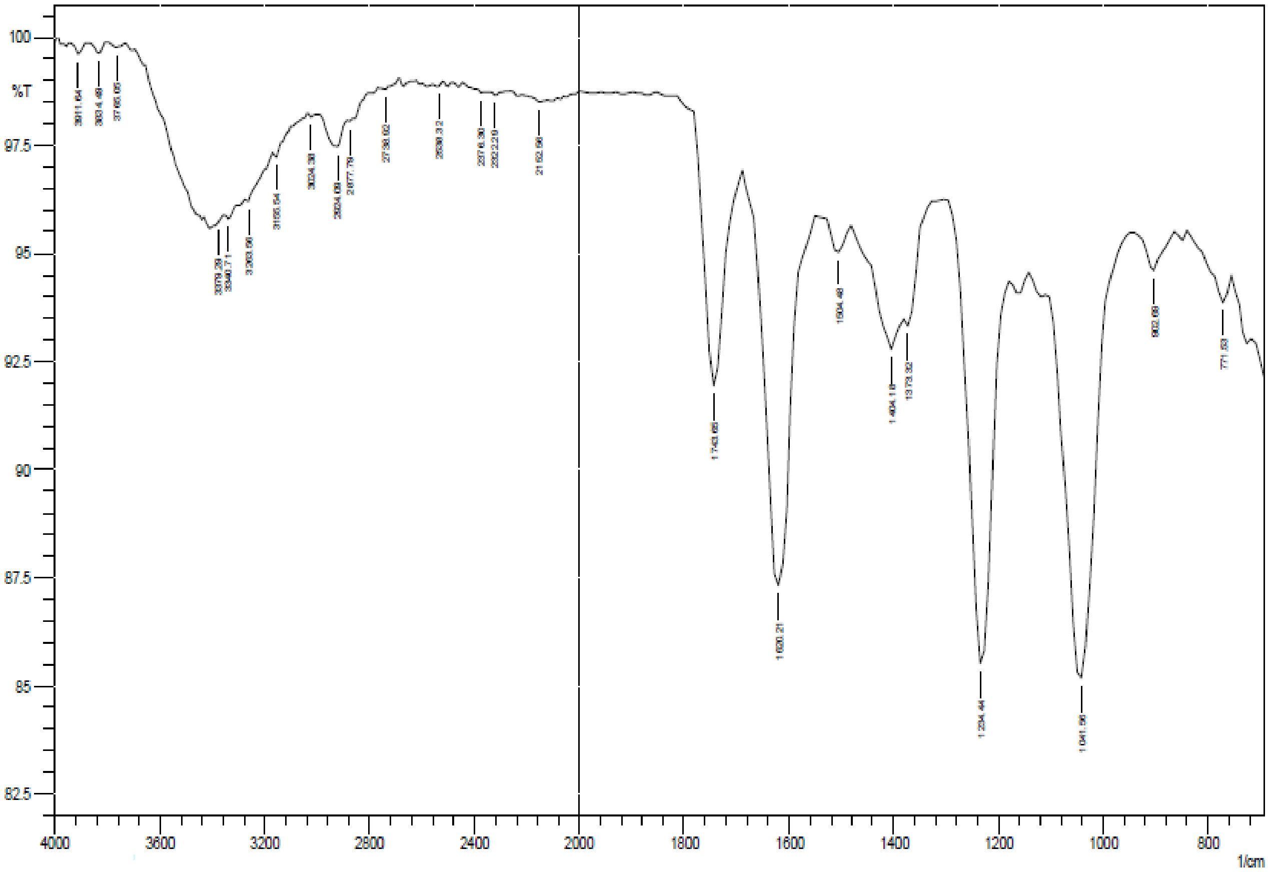

Figure 5.2 FTIR Spectrum of Membrane(I)

The peak corresponding to 2376.30, 1743.65 and 902.69 is found to be common peak for membrane(I) and cellulose acetate. The peak for 2376.30 indicate N-H (ammonium ion)(multiple bond peak) (ketone) stretching and for 902.69 C-H (bending)(strong).

The additional peak found for membrane include

Table 5.1

FTIR Spectrum of Membrane(I)

Sl.N0	Frequency (cm^{-1})	Stretching
1	3911.64	X-H(Halogen groups)
2	3834.49	N-H(Amines)
3	3765.05	N-H(Amines)
4	3379.29	O-H(H-bonded)(stretch free strong) (alcohol phenol) N-H(Primary amines)
5	3340.71	O-H (stretch free),(strong bond) N-H(primary amines)
6	3263.56	N-H(primary amines) O-H(stretch free),(strong bond)
7	3155.54	N-H(primary amines), C-H(Alkene),(strong)
8	3024.38	C-H(alkene)(strong) O-H(dimer)(broad) COOH(alcohol-phenol)
9	2924.09	C-H(alkane)(strong), O-H(dimer),(COOH),(broad)
10	2877.79	C-H(Alkane),(strong) O-H(Dimer)(broad)(COOH)
11	2738.32	C-H(Alkane)(strong), O-H(broad)(COOH)
12	2538.32	N-H(ammonium ion),(multiple broad peak) O-H(broad)(COOH)
13	2376.30	C=C(Alkenes)
14	2322.29	C≡C (Alkynes)

15	2152.56	C≡C(Alkynes), N=C=O,N=C=S,N=C=N,C=C=O(isocynates,isothiocynates,dimide,ketones)
16	1620.21	C=C(aromatic bending) N-H(primary amine) C-H(Phenyl ring)(substitution overtones)
17	1504.48	C=C(Aromatic bending) NO_2(asymmetrical stretch)
18	1404.18	C=C(Aromatic bending, medium-weak) CH_2(Bending) C-H(Alkanes)
19	1373.32	C-H(Alkanes,Scissoring and bending) C-F(alkyl halide, strong) NO_2(Nitrocompounds,symmetrical) CH_2(rocking)
20	1234.44	C-F(Alkyl halide) C-N(Medium-weak) C-0(Ether, weak)
21	1041.56	C-N(Amine) C-O(Ether) N-H(Primary amine)(strong, broad)
22	171.53	O-H(Stretch, free)

FT-IR spectrum of membrane (II):

The peak corresponding to 1381.03 is found to be common peak for membrane (II) and cellulose acetate. The peak for 1381.03 indicate C-F (Alkyl halide),C-H(Alkane,bending),N-O(Nitro, strong band).

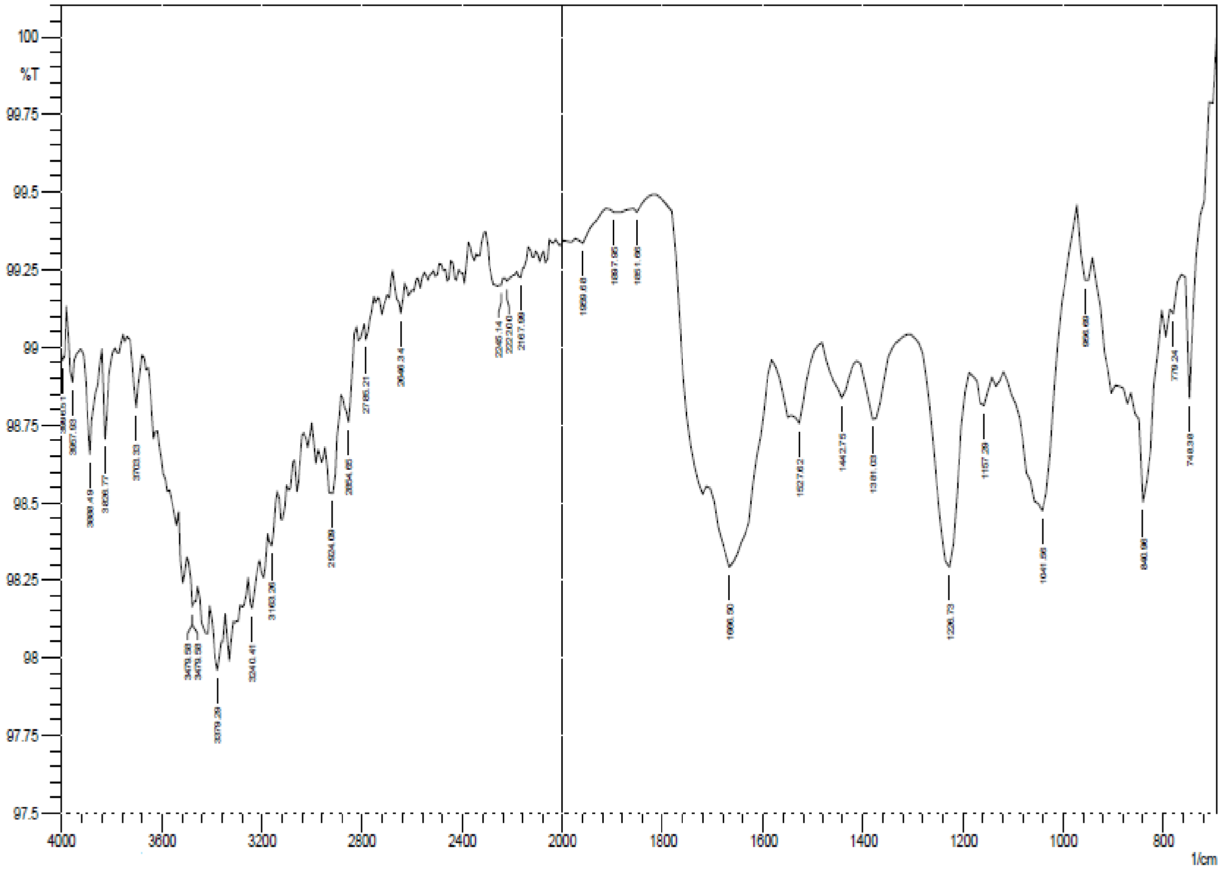

Figure 5.3 FTIR Spectrum of Membrane (II)

The additional peak found for membrane include

Table 5.2

FTIR Spectrum of Membrane (II)

Sl.N0	Frequency(cm^{-1})	Stretching
1	3990.51	X-H(Halogen groups)
2	3957.93	X-H(Halogen groups)
3	3888.49	O-H(H-bonded)(stretch free strong) (alcohol phenol)
4	3826.77	O-H(H-bonded)(stretch free strong) (alcohol phenol)
5	3703.33	N-H(amide)
6	3479.58	N-H(primary amines) O-H(alcohol)(phenol) C=0(Aldehyde,ester,ketone,carboxylic acid)
7	3379.29	N-H(primary amines), C=0(Aldehyde,ester,ketone,carboxylic acid) C-H(Alkane) O-H(alcohol, phenol)
8	3240.41	C=0(Aldehyde,ester,ketone,carboxylic acid) O-H(Alcohol,phenol)
9	3163.26	C=0(Aldehyde,ester,ketone,carboxylic acid) O-H(Alcohol,phenol)
10	2924.09	C-H(Alkane),(strong) O-H(Carboxylic acid)
11	2854.65	C-H(Alkane)(strong), O-H(broad)(COOH)
12	2785.21	O-H(Carboxylic acid)
13	2646.34	O-H(Carboxylic acid)
14	2245.14	.C=N(Nitrile)
15	2222.00	C=C(Nitrile)

16	2167.99	C≡ $C(Alkyne)$
17	1959.68	C-H(Phenyl substitution overtones)
18	1897.95	C-H(Phenyl substitution overtones)
19	1851.66	C-H(Phenyl substitution overtones)
20	1666.50	C=C(Alkenes) C=O(Amide)
21	1527.62	N-H(Amine)(Scissoring, bending)
22	1442.75	C-H(Alkanes) CH_2(Bending)
23	1226.73	C-O(Aldehyde,ketone.carboxylic acid,ester) C-N(Amine)
24	1157.29	C-O(Aldehyde,ketone,carboxylic acid,ester)
25	1041.56	C-N(Amine) C-O(Aldehyde,ketone.carboxylic acid, ester)
26	956.69	C-H(Alkenes)
27	840.96	C-H(Alkenes)(Arenes,C-H bending and ring puckering)
28	779.24	CH_2(Bending) C-H(Alkenes)(Arenes C-H Bending and ring puckering)
29	748.38	C-H(Alkenes)(Arenes C-H Bending and ring puckering)

SCANNING ELECTRON MICROSCOPE:

SEM images of the membrane (I)

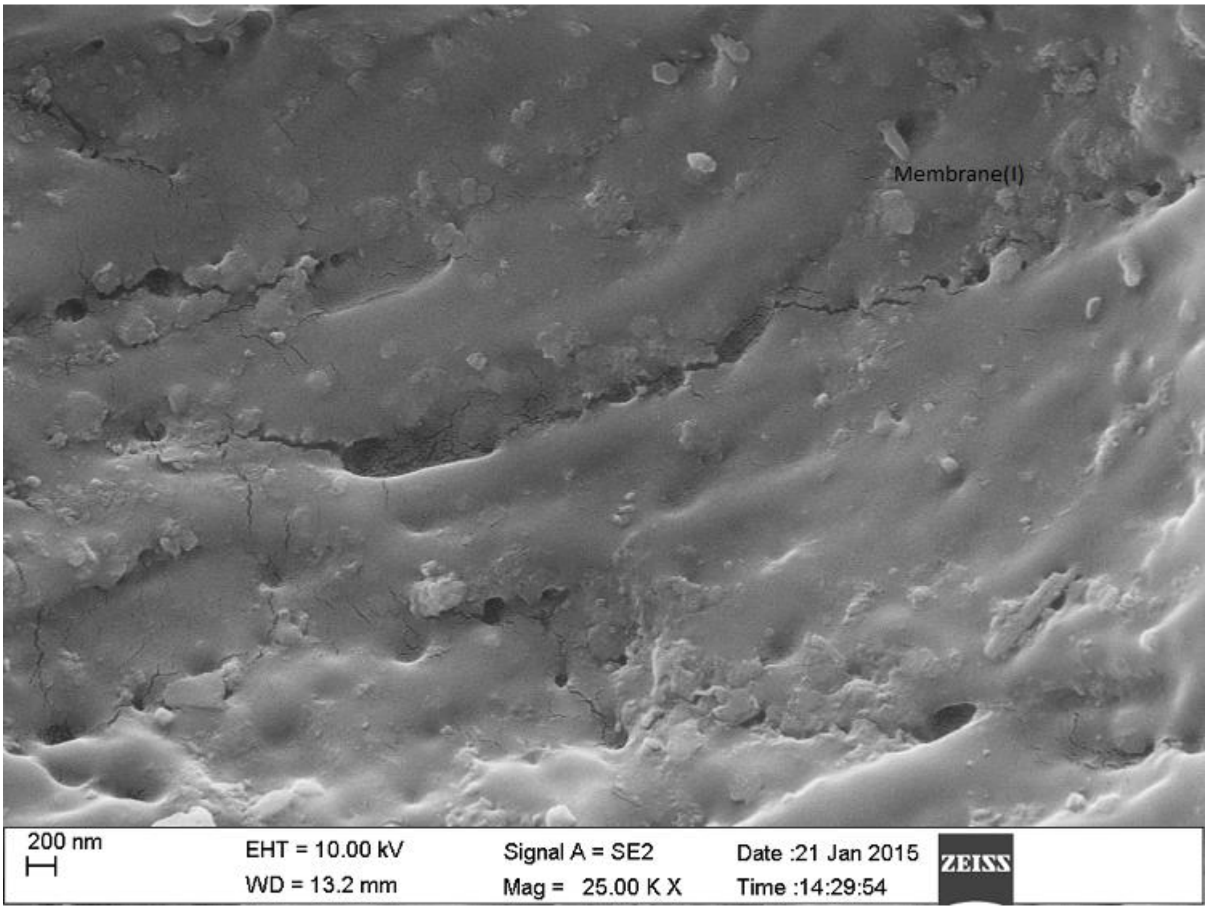

Figure 5.4 SEM images of Membrane (I)

SEM images of the membrane (II)

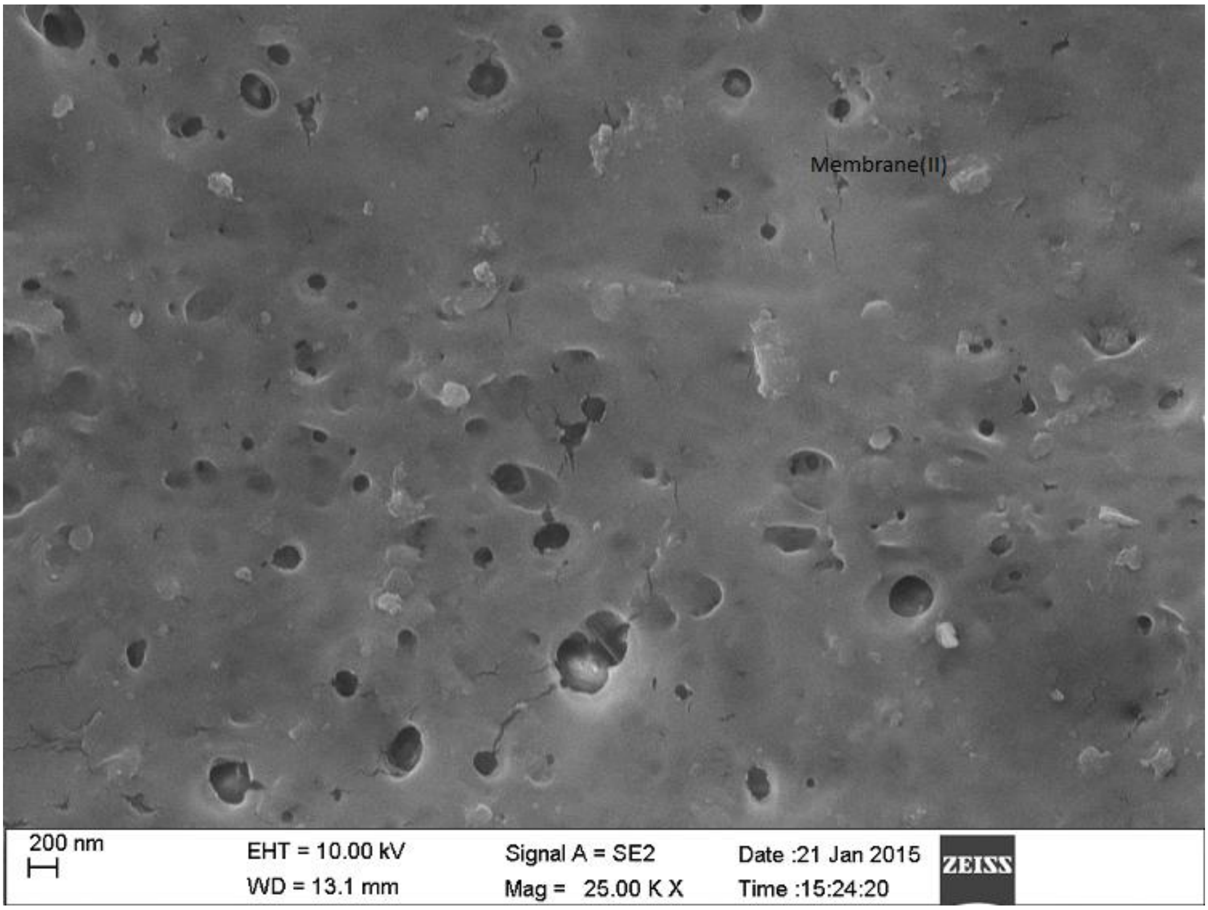

Figure 5.5 SEM images of Membrane (II)

THERMAL GRAVIMETRIC ANALYSIS:

The weight loss of the membrane was analysed by the thermogravimetric analysis and it was measured from the temperature of 30°C to 210°C .

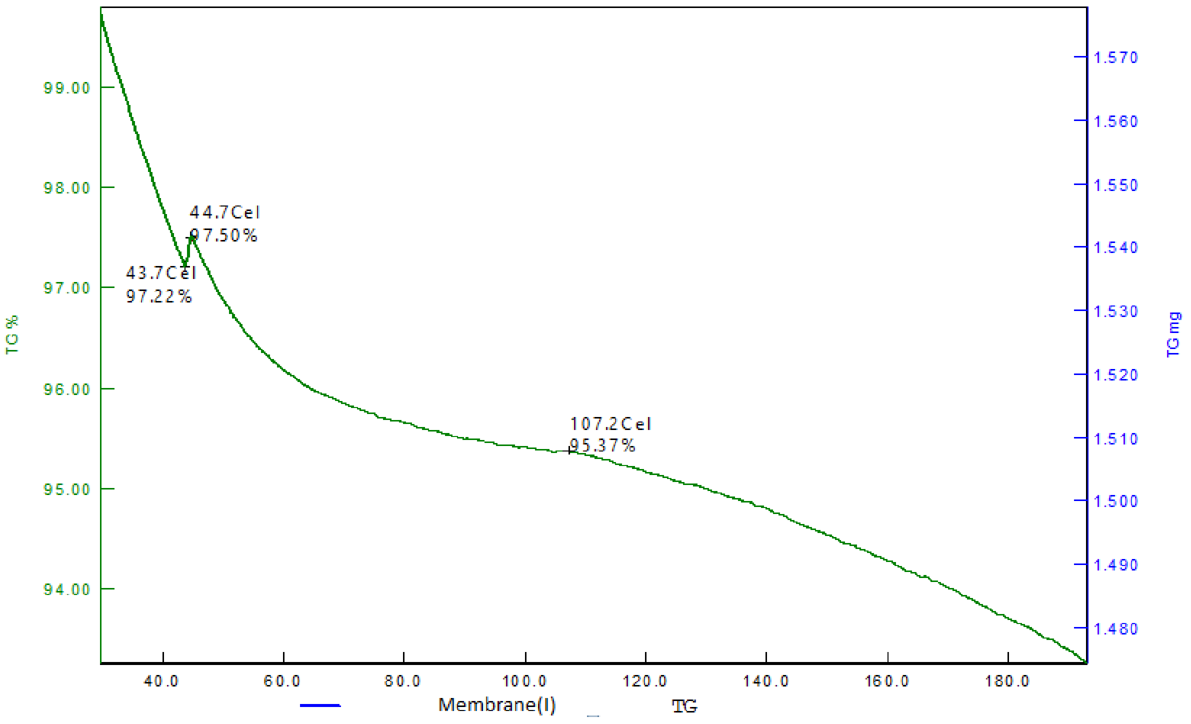

Figure 5.6 TGA images of Membrane (I)

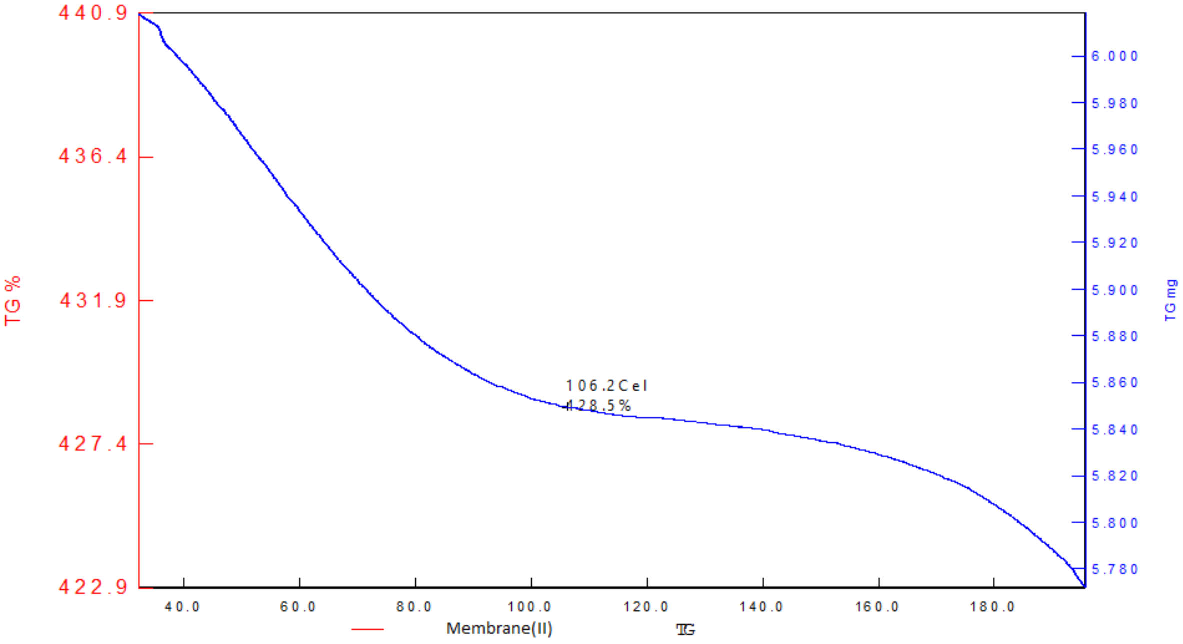

Figure 5.7 TGA images of Membrane (I)

X-RAY DIFFRACTION:

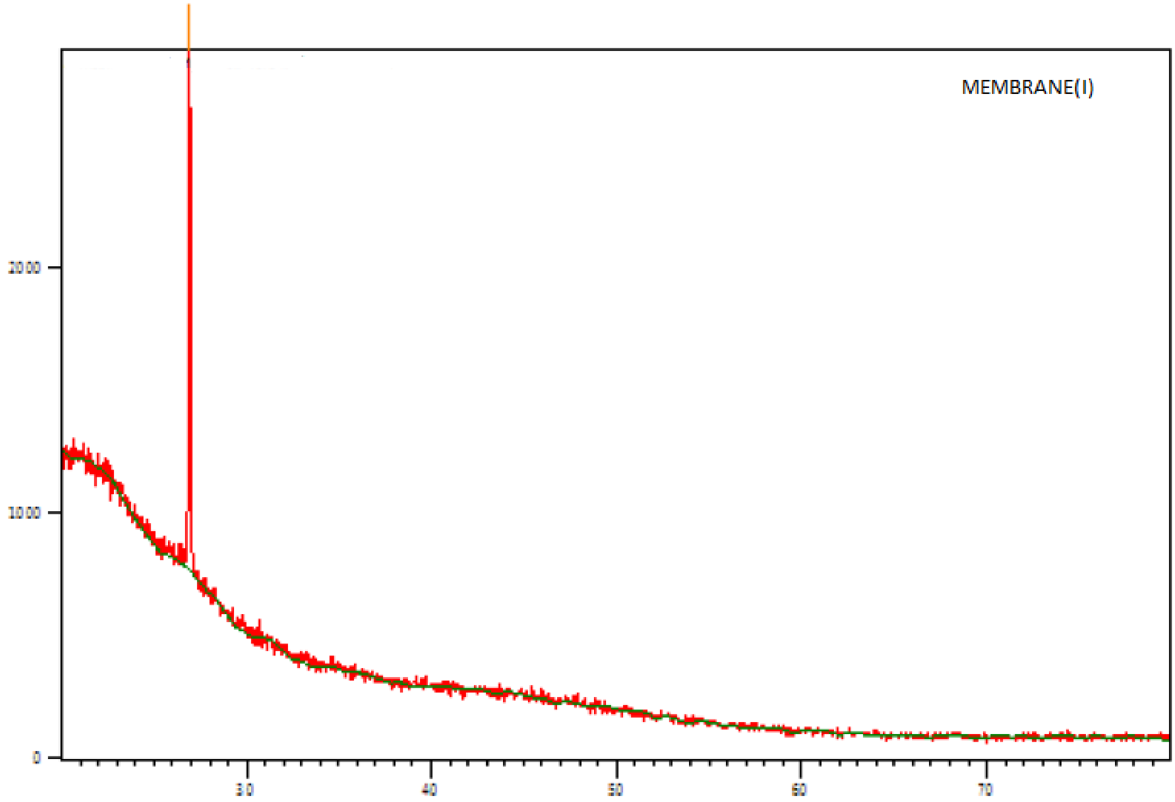

Figure 5.8 X-RD images of Membrane (I)

Using X- ray diffraction particle size was obtained in the range 65.6872nm.

PURE WATER FLUX:

Pure water flux was calculated by using formula

$$J_W=Q/A\Delta V \qquad \rightarrow` \qquad (1)$$

Membrane (I):

$Q=19$; $A=\pi r^2$; $\pi=3.14$; $r=3.5$;

$$J_W = 19/3.14\times3.5\times3.5\times0.033$$

$$= 14.8344$$

Membrane (II):

$Q=19$; $A=\pi r^2$; $\pi=3.14$; $r=3.5$;

$$J_W = 19/3.14\times3.5\times3.5\times0.0500$$

$$= 9.8793$$

Porosity:

Porosity is calculated by using formula

$$=[W1-W2/d_{water}]\times100/v$$

Membrane (I):

$W_1=0.8634$; $W_2=0.4053$;dwater=1.33 ; $v=\pi r^3$

$$= [0.5301/1.33]\times100/3.14\times4.2\times4.2\times4.2$$

$$= 0.0427$$

Membrane (II):

$W_1 = 2.6944$; $W_2 = 2.0424$; $d_{water} = 1.33$; $v = \pi r^3$

$= [0.6520/1.33] \times 100/3.14 \times 3.5 \times 3.5 \times 3.5$

$= 0.0909$

Table 5.3

Physio-chemical parameters of sample water & Filter water using membrane (I) & (II)

S.NO	PARAMETER	SAMPLE WATER	MEMBRANE(I)	MEMBRANE(II)
1	PH	8.81	7.96	7.58
2	EC (ds/m)	1.21	1.19	1.19
3	Calcium(mg/L)	52.80	43.2	38.4
4	Magnesium(mg/L)	59.41	50.78	36.2
5	Sodium(mg/L)	143.06	75.21	53.59
6	Potassium(mg/L)	98.66	14.04	9.36
7	Carbonate(mg/L)	2.40	1.60	1.60
8	Bicarbonate(mg/L)	3.60	3.20	3.20
9	Chloride(mg/L)	9.58	9.52	8.16
10	BOD	5.4	4.6	3.8
11	COD	16.5	15	12.1
12	Dissolved oxygen	7.9	6	5.2
13	Bacteria	10.5×10^5	9×10^5	4.5×10^5
14	Fungi	6×10^3	5.5×10^3	1.5×10^3

Table 5.4

Hardness measurement of sample water and Filtered water using membrane (I) & (II)

S.NO	PARAMETER	SAMPLE WATER	MEMBRANE(I)	MEMBRANE(II)
1	Calcium carbonate	2.16	1.60	1.60
2	Magnesium carbonate	0.24	0.24	-
3	Magnesium bicarbonate	3.28	2.92	2.16
4	Sodium bicarbonate	0.28	0.28	-
5	Sodium chloride	2.40	0.09	1.90

pH:

pH is an important indicator which indicates acidic and alkaline nature of water. pH should be in the level of 6.6 to 8.4(World Health Organisation).The pH of the sample water was found to be 8.81.The sample water was filtered using membrane (I) and tested for pH. It was found that the pH of sample water was found to be 7.96.

The sample water was filtered using Membrane (II) and tested for pH. It was observed that the pH of sample water was found to be 7.81.

The pH of the sample water after filtration using membrane(I) and (II), the pH were obtained 7.96 and 7.81.

The obtained values were compared with the standard value of World Health Organisation and it was found that the values were in good agreement.

Electrical conductivity (EC):

Electrical conductivity (EC) is used to determine the cell constant value. The EC of the sample water was found to be (1.21).Th e sample water was filtered using membrane (I) and tested EC. It was observed that the EC of sample water was found to be (1.19)

The sample water was filtered using Membrane (II) and tested for EC. It was observed that the EC of sample water was found to be (1.19).

The EC of the sample water after filtration using membrane (I) and (II), the EC were obtained 1.19 and 1.19.

Calcium Hardness:

In drinking water, Calcium hardness should be in the level of 75mg/L(World Health Organisation).The calcium hardness of sample water was found to be 52.80mg/L. The sample water was filtered using Membrane (I) and tested for Calcium Hardness. It was observed that the Calcium hardness of sample water was found to be 43.2 mg/L.

The sample water was filtered using Membrane (II) and tested for Calcium hardness. It was observed that the Calcium hardness of sample water was found to be 38.4 mg/L.

The Calcium hardness of the sample water after filtration using membrane (I) & (II), the Calcium hardness obtained are 43.2 mg/L& 38.4 mg/L respectively.

The obtained results were compared with the standard value of world health organisation and it was found that the values were in good agreement.

Magnesium Hardness:

In drinking-water, Magnesium hardness should be in the level of 150mg/L (world health organization 2003).The Magnesium hardness of sample water was found to be 59.4mg/L. The sample water was filtered using Membrane (I) and tested for Magnesium hardness. It was observed that the Magnesium hardness of sample water was found to be 50.7 mg/L..

The sample water was filtered using Membrane (II) and tested for Magnesium hardness. It was observed that the Magnesium hardness of sample water was found to be 36.2mg/L.

The Magnesium hardness of the sample water before filtration using membrane (I) & (II), the magnesium hardness obtained are 50.7mg/L & 36.2mg/L respectively.

The obtained results were compared with the standard value of world health organisation and it was found that the values were in good agreement.

Potassium hardness:

In drinking-water, potassium hardness should be in the level of 82mg/L (world health organization 2003).The potassium hardness of sample water was found to be 98.6mg/L. The sample water was filtered using Membrane (I) and tested for potassium hardness. It was observed that the potassium hardness of sample water was found to be 14.04mg/L.

The sample water was filtered using Membrane (II) and tested for potassium hardness. It was observed that the potassium hardness of sample water was found to be 9.36 mg/L.

The potassium hardness of the sample water before filtration using membrane (I) & (II), the potassium hardness obtained are 14.04 mg/L &9.36mg/L respectively.

The obtained results were compared with the standard value of world health organisation and it was found that the values were in good agreement.

Sodium:

In drinking-water, sodium hardness should be in the level of 200mg/L (world health organization 2003).The sodium hardness of sample water was found to be 143.06mg/L. The sample water was filtered using Membrane (I) and tested for sodium hardness. It was observed that the sodium hardness of sample water was found to be 75.21mg/L.

The sample water was filtered using Membrane (II) and tested for sodium hardness. It was observed that the sodium hardness of sample water was found to be 53.59mg/L.

The sodium hardness of the sample water before filtration using membrane (I) & (II), the sodium hardness obtained are 75.21 mg/L &53.59 mg/L respectively.

The obtained results were compared with the standard value of world health organisation and it was found that the values were in good agreement.

Carbonate:

In drinking water, Carbonate hardness should be in the level of 500mg/l (World Health Organisation).The carbonate hardness of sample water was found to be 2.40 mg/L. The sample water was filtered using Membrane (I) and tested for carbonate Hardness. It was observed that the carbonate hardness of sample water was found to be 1.60 mg/L.

The sample water was filtered using Membrane (II) and tested for carbonate hardness. It was observed that the carbonate hardness of sample water was found to be 1.60 mg/L.

The carbonate hardness of the sample water after filtration using membrane (I) & (II), the carbonate hardness obtained are 1.60 mg/L& 1.60 mg/L respectively.

The obtained results were compared with the standard value of world health organisation and it was found that the values were in good agreement.

Bicarbonate:

In drinking water, Bicarbonate hardness should be in the 500mg/l (World Health Organisation).The bicarbonate hardness of sample water was found to be 3.60 mg/L. The sample water was filtered using Membrane(I) and tested for bicarbonate Hardness. It was observed that the bicarbonate hardness of sample water was found to be 3.20 mg/L.

The sample water was filtered using Membrane (II) and tested for Calcium hardness. It was observed that the bicarbonate hardness of sample water was found to be 3.20 mg/L.

The bicarbonate hardness of the sample water after filtration using membrane (I) & (II), the bicarbonate hardness obtained are 3.20mg/L& 3.20 mg/L respectively.

The obtained results were compared with the standard value of world health organisation and it was found that the values were in good agreement.

Dissolved oxygen:

In drinking-water, Dissolved oxygen should be in the level of 30mg/L (World health organzation2003).The Dissolved oxygen of sample water was found to be 7.9mg/L. The sample water was filtered using Membrane (I) and tested for Dissolved oxygen. It was observed that the Dissolved oxygen of sample water was found to be 6mg/L.

The sample water was filtered using Membrane (II) and tested for Dissolved oxygen. It was observed that the Dissolved oxygen of sample water was found to be 5.2mg/L.

The Dissolved oxygen of the sample water after filtration using membrane (I) & (II), the Dissolved oxygen obtained are 6mg/L & 5.2 mg/IL respectively.

The obtained results were compared with the standard value of world health organisation and it was found that the values were in good agreement.

Biological oxygen demand:

Biological oxygen demand is the measure of degradation of organic matter present in water. The Biological oxygen demand refers to the oxygen used by the micro-organisms in the aerobic oxidation of organic matter. The Biological oxygen demand of sample water was found to be 3.8 mg/L. The membrane I was filtered water and tested for Biological oxygen demand of water, it is observed that 5.4mg/L. The membrane II was tested for Biological oxygen demand of water, it was observed that 4.6mg/L.

The Biological oxygen demand of the sample water after filtration using membrane (I) & (II), the Biological oxygen demand obtained are 5.4 mg/L & 4.6 mg/L respectively.

The obtained results were compared with the standard value of world health organisation and it was found that the values were in good agreement.

Chemical oxygen demand:

Chemical Oxygen Demand is another measure of organic material contamination in water specified in mg/L. Chemical Oxygen Demand is the amount of dissolved oxygen required to cause chemical oxidation of the organic material in water. The Chemical Oxygen Demand of sample water was found to be 12.1 mg/L. The sample water was filtered using membrane(I) and tested for Chemical Oxygen Demand of water, it was observed that 16.5 mg/L.The membrane II was tested for Chemical Oxygen Demand of water, it was observed that 15 mg/L.

The Chemical oxygen demand of the sample water before filtration using membrane (I) & (II), the Chemical oxygen demand obtained are 16.5mg/L & 15mg/L respectively.

The obtained results were compared with the standard value of world health organisation and it was found that the values were in good agreement.

Chloride:

Chloride is an important parameter in accessing the water quality. The values of chlorides in normal drinking water are range 250 mg/L (world Health organization 2003). The sample water has the chloride value of 9.52mg/L. The membrane (I) was filtered and tested for Chloride of water, it is observed that 8.16mg/L.

The membrane II was tested for Chloride of water, it is observed that 9.5mg/L.

The Chloride value of the sample water after filtration using membrane (I) & (II), the Chloride obtained are 8.16 mg/L & 9.5mg/L respectively.

The obtained results were compared with the standard value of world health organisation and it was found that the values were in good agreement.

Bacteria:

Bacteria are an important parameter in accessing the water quality. The sample water has the bacteria value of 10.5×10^5mg/L. The membrane (I) was filtered and tested for bacteria of water, it is observed that 9×10^5mg/L.

The membrane II was tested for bacteria of water, it is observed that 4.5×10^5mg/L.

The bacteria value of the sample water after filtration using membrane (I) & (II), the bacteria obtained are 9×10^5 mg/L &4.5×10^5 mg/L respectively.

Fungi:

Fungi are an important parameter in accessing the water quality. The sample water has the fungi value of 6.3×10^3mg/L. The membrane (I) was filtered and tested for fungi of water, it is observed that 5.5×10^3mg/L.

The membrane II was tested for fungi of water, it is observed that 1.5×10^3mg/L.

The fungi value of the sample water after filtration using membrane (I) & (II), the fungi obtained are 5.5×10^3 mg/L &1.5×10^3 mg/L respectively.

Summary and Conclusion

SUMMARY AND CONCLUSION

Cellulose Acetate can be dissolved directly in a dimethyl Acetamide/diethyl succinate solution.

The solvent system is of low toxicity and higher solubility. Cellulose Acetate and vetiveru powder are directly dissolved in the solvent.

The aim of this study to prepare the Green membrane synthesis using low toxicity solvent.

The physio-chemical parameter values of the filtered water using the prepared membranes (I) & (II). Compared with the values described by the World Health Organisation and the values mostly which is in good agreement.

Compared with the values obtained using membrane (I) &(II) the datas so obtained which are in good agreement are membrane(I) which may be due to the presence of vetiveru powder.

The membrane preparation is an Eco-friendly method and it is easy to handle and it is low cost method also.

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