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TOPIC:

**Water treatment station's pursuing  
in TABUK PHARMACEUTICALS**

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## Dedication

It is with great honor that I dedicate this modest work to my dear parents, before all and for all, whom I adore and wish to always see them by my side:

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## **Abbreviations List:**

**AFM** :atomic force microscopy

**CA**: cellulose acetate

**CODB**: Biodegradable dissolved organic carbon

**COD**: dissolved organic carbon

**DMSO**: dimethyl sulfoxide

**FO** :forward osmosis

**K**: Potassium

**MD**: molecular dynamics

**MF**: microfiltration

**MPD**: metaphenylene diamine

**NF**: nanofiltration

**UF** : ultrafiltration

**ED** : Electro dialysis

**EDR** : Electro dialysis reversal

**PA**: polyamide

**PALS** :positron annihilation spectroscopy

**PRO**: pressure retarded osmosis

**PS-CF**: preferential sorption-capillary flow

**RO**: reverse osmosis

**SANS** :small-angle neutron scattering

**S-D model**: solution-diffusion model

**SEM**: scanning electron microscope

**SWCNTs**: single walled carbon nanotubes

**TFC** :thin film composite

**TMC** :trimesoyl chloride

**TOC**: total organic carbon

**MES: Suspended Materials**

**MO: Organic materials**

**Na +: Sodium**

**NH<sub>4</sub> +: Ammonium**

**NO<sub>2</sub>-: Nitrite**

**NO<sub>3</sub>-: Nitrate**

**NTU: Nephelometric Unit**

**pH: Hydrogen potential.**

**PO<sub>4</sub>-3: Phosphate**

**SO<sub>4</sub>: sulphates**

**μS / cm: Micro cimmence per centimetre**

**PH : Hydrogen potential.**

**Ph. Eur : European Pharmacopoeia.**

**Ppb : parts per trillion.**

**Ppm : part per million.**

**TA: Alkalimetric Title**

**TAC: Complete alkalimetric titer**

**TH: Hydrometric Title**

**Turb EB: turbidity of raw water.**

**Turb ET: turbidity of the treated water**

**SPC: chlorination by-products**

**THMs: trihalomethanes.**

**SO<sub>2</sub>- : Sulphates**

**T : temperature**

**UFC :Colony forming unit.**

**UI : international unit. (Unit of measure for the quantity of a substance).**

**USP : united states pharmacopy**

**UV : ultraviolet**

**"Nothing is lost, nothing is created, everything  
is transformed ..."**

**This principle also applies to water: source of  
life,**

**valuable but difficult to access resource,  
which can also be a source of death if not  
properly managed.**

## **Introduction :**

Water affects all facets of life for humans, animals, plants, and the environment. Without it, there would be no vegetation on land, no oxygen for animals to breathe, and no humans to survive. Water is an essential component of everything we eat and drink now and into the future. Even though there are a variety of water sources, such as rivers, lakes, and groundwater, fresh drinking water only constitutes 3% of the total global water available. [1]

Safe drinking water production is an ancient art while establishing standards is relatively new ,the treatment of raw water by removing such pollutants compounds (organic or/and inorganic), however, is neither cheap nor easy to do. There is therefore a clear and urgent need for more intensive research in wastewater treatment that is practical and achievable at the lowest possible cost.

The advent of membranes, particularly hollow fiber based, has made a major impact as brackish water and seawater can be converted at room temperature into treated water. It appears that even purified water can be made via membrane processes.

These technologies, with all types of membrane processes, such as microfiltration, ultrafiltration, Nano filtration (NF) and reverse osmosis have implications in terms of large energy savings. The growth of RO is impressive, as all unwanted organic impurities, including antibiotics, agrochemicals, dyes, and so on, can be so easily and effectively removed.

reverse osmosis (RO) is considered as a well-established separation process, ever since the first cellulose acetate membrane was announced more than half a century has passed. RO is still continuing its steady growth both in the commercial market and as a popular research topic.

the purpose of our work is to describe the performance qualification process of the Purified water production station in order to verify that all the components of the system linked together can operate efficiently and reproducibly

Eventually our researcher's report would come across the four divided parts , which are the following:

-The first chapter introducing all of the different membranes presses , their applications into eliminate of impurities such as ( nitrates , heavy metals , inorganic contaminants ..ect) , and their configuration.

-in the 2<sup>nd</sup> chapter are mentioned briefly water sources supplies in addition to defining multiple sorts of pharmaceutical water including their chemical specifications .

- the 3<sup>rd</sup> chapter study Tabuk Pharmaceuticals's water station , going through 9 checkpoints after every filtration step highlighting materials and methods used in our water treatments and passing by frequencies, parameters and control standards at each sampling point.

-In our final 4<sup>th</sup> chapter our work results are represented , discussing how we managed to disinfect contaminants using ozone and why .

## ملخص

الماء عنصر طبيعي ضروري للحياة. إنه أصل ضروري لأي نشاط بشري ، وهو تراث أمة. الهدف الرئيسي من هذه الدراسة هو تقييم الجودة الفيزيائية والكيميائية لمياه الشرب التي تزود صناعة الأدوية تبوك ، وكذلك الجودة الفيزيائية والكيميائية والميكروبيولوجية للمياه النقية التي تنتجها الصناعة نفسها من نفس مياه الشرب المذكورة أعلاه.

في هذه الدراسة ، تم أخذ عينات من المياه النقية ومياه الشرب التي تمر عبر نقاط مختلفة من محطة المعالجة ، وقد تم أخذ هذه الأخيرة يوميًا لمدة أربعة أسابيع متتالية.

أظهرت النتائج التي تم الحصول عليها أن مياه الشرب تتميز بتمعدن كبير ومستويات منخفضة جدًا من النترات والرصاص. مياه الشرب لا تحتوي على أي مادة قابلة للتأكسد.

تتميز المياه النقية بموصلية كهربائية منخفضة للغاية ، وغياب شبه كامل للنترات والرصاص والمواد القابلة للأكسدة، فيما يتعلق بالتحليل الميكروبيولوجي ، فإن عدد الجراثيم الهوائية لم يتجاوز أبدًا حد التنبيه وهو 10 UFC /100 مل .

أخيرًا ، أوضحت الدراسة أن المياه النقية في شركة تبوك للأدوية كانت ذات جودة فيزيائية وكيميائية وميكروبيولوجية جيدة ومع ذلك ، تظل هذه الجودة مرتبطة بالتشغيل السلس للتنقية.

**Abstract:**

Water is a natural element essential for life. It is a necessary asset for any activity human, and is the heritage of a nation. The main objective of this study is assess the physicochemical quality of drinking water supplying the TABUK pharmaceuticals industry, , as well as the physicochemical and microbiological quality of the purified water produced by the industry itself from the same drinking water mentioned above.

In the present study, a sampling of the purified water and of the drinking water passing through the different points of the treatment plant, the latter were taken daily for four successive weeks.

The results obtained show that drinking water is characterized by significant mineralization and very low levels of nitrates and lead. Drinking water does not contain any oxidisable substance.

Purified water is characterized by very low electrical conductivity, the almost total absence of nitrates, lead and oxidizable substances.

Regarding the microbiological analysis, the count of total aerobic germs has never exceeded the alert threshold which is 100 CFU / 10ml.

Finally, the study revealed that the purified water atTabuk pharmaceuticals was of good physicochemical and microbiological quality. However, this quality remains linked to the smooth running of the purification.

## **Résumé:**

L'eau est un élément naturel essentiel à la vie. C'est un atout nécessaire à toute activité humaine, et c'est l'héritage d'une nation. L'objectif principal de cette étude est d'évaluer la qualité physico-chimique de l'eau potable alimentant l'industrie pharmaceutique TABUK, ainsi que la qualité physico-chimique et microbiologique de l'eau purifiée produite par l'industrie elle-même à partir de la même eau potable mentionnée ci-dessus.

Dans la présente étude, un prélèvement de l'eau épurée et de l'eau potable passant par les différents points de la station d'épuration, ces dernières ont été prélevés quotidiennement pendant quatre semaines successives.

Les résultats obtenus montrent que l'eau potable est caractérisée par une minéralisation importante et de très faibles niveaux de nitrates et de plomb. L'eau potable ne contient aucune substance oxydable. L'eau purifiée se caractérise par une très faible conductivité électrique, l'absence presque totale de nitrates, de plomb et de substances oxydables.

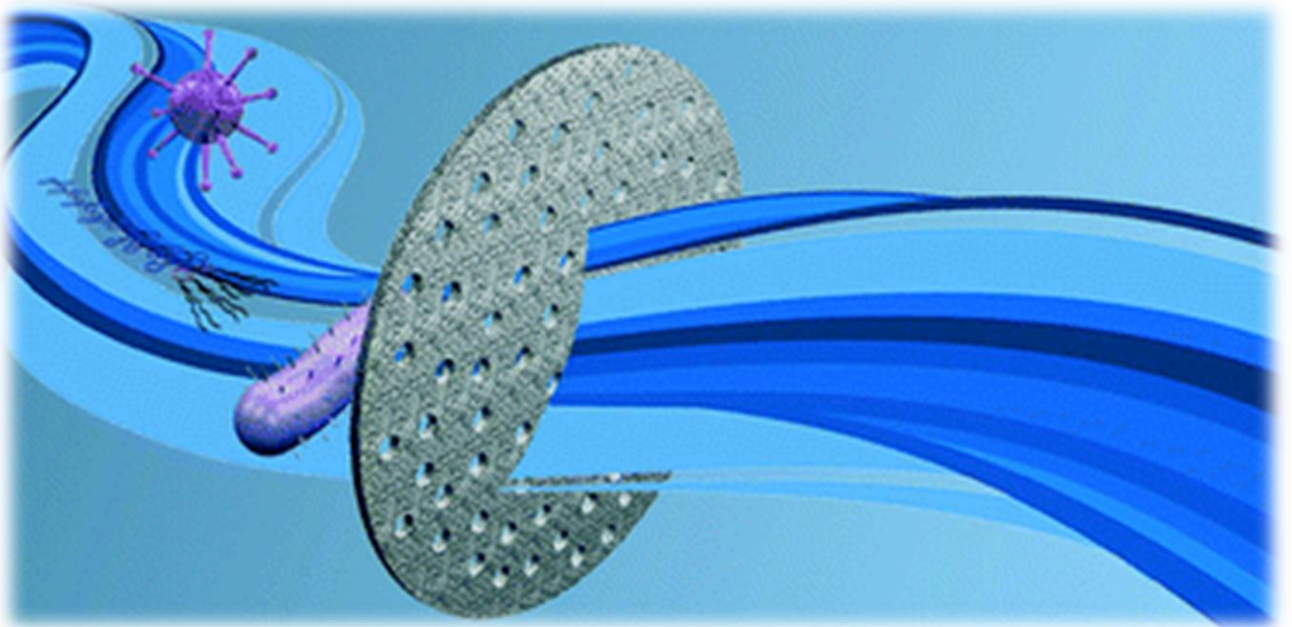
Concernant l'analyse microbiologique, le nombre de germes aérobies totaux n'a jamais dépassé le seuil d'alerte qui est de 100 UFC/10ml.

Enfin, l'étude a révélé que l'eau purifiée de Tabuk Pharmaceuticals était de bonne qualité physico-chimique et microbiologique. Cependant, cette qualité reste liée au bon déroulement de l'épuration.



# Chapter I :

## Membranes processes and water treatment



### **I.1. Introduction**

Only certain so-called mineral or spring waters may be marketed and consumed without treatment. However, for spring water sometimes treatments such as aeration, settling and filtration can be applied to them. All the others so-called "raw" natural waters need treatment before being consumed, in order to meet the standards in force defining water good for consumption human. They are then transported to a drinking water production plant. The treatment of raw water depends on its quality, linked to its origin, the main treated water being surface water and groundwater [1.3]

### **I.2. Purpose of the treatment**

The treatment should be adjusted to the chemical composition of the collected water. It therefore varies from site to another. If, for certain waters, a partial or simple treatment such as filtration rapid and disinfection of the water is sufficient, others require a complete treatment more or less complex, or even specific treatments to remove specific pollutants. [1]

### **I.3.Pretreatment of water:**

Preserving membrane integrity through proper pre-treatment of source water is essential to maximize the efficiency and longevity of an RO or NF membrane system. This section provides a discussion of the various methods of pre-treatment that prevent chemical alteration of the membrane polymer and fouling accumulation and scaling on the membrane surface . [4]

### **I.4.Filtration :**

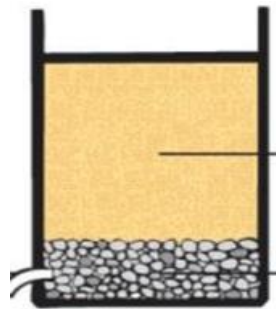
The filtration system is composed of filters with different sizes and consist of the Mostly gravel, sand and charcoal. There are two types of filtration of sand basis: slow sand filtration and fast sand filtration. Filtration slow sand is a biological process because it uses bacteria to treat the water. Bacteria forms a layer on the top of the sand and cleans the water as it passes through, digesting contaminants in the water. The bacteria layer is called the biofilm. Rapid sand filtration is a physical process that removes the suspended solids in water. This rapid filtration is much more common because that fast sand filters have fairly high flow rates and require little space to function. During filtration, the water flow rate can be up to 20 meters per hour. The filters are generally cleaned twice a day.

The particles that are removed from the water during filtration depend on the size of the filters that are used. Slow filtration removes bacteria, viruses and protozoa and mainly produces clean water. It is recommended to use a disinfectant as a precautionary measure. Fast filtration removes suspended particles, such as bacteria, viruses and protozoa. In treatment plants, filtration removes a large number of contaminants, but still requires disinfection to produce safe drinking water. Even though rapid filtration cannot remove all bacteria and viruses, this is an important step in the treatment process. Coagulation and filtration can remove suspended and dissolved matter, so that the disinfection is more successful with a reduced amount of chlorine. The principle of the filter under gravel is to mimic nature's scrub cycle using the

sandy bottom of the aquarium. Water laden with organic impurities (mainly solid waste, substances ammonia and nitrites) is carried by a slow downward current through gravel thickness. The water leaves its solid impurities in the gravel. The charcoal is a substance that has long been used to adsorb impurities. The activated carbon is a carbon that has a slight positive electric charge making it attractive chemicals and impurities. As the water passes through the activated carbon, the ions Negative contaminants are attracted to the surface of the carbon granules. Filters activated carbon remove / reduce a lot of volatile organic chemicals, pesticides and herbicides, such as chlorine, benzene, radon, solvents and others synthetic chemicals found in tap water. [6.7]

#### **I.4.1 .Sand filtration:**

This pre-treatment filtration is very easy to implement , It consists of filling a container, often a simple concrete tank with a tap or a means of draining, a layer of rather fine enough gravel and a layer of heavy more sand on which the water to be treated is poured than it suffices to collect at the bottom of the container.

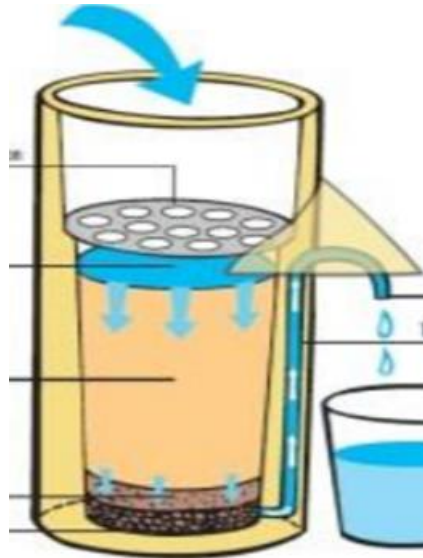


**Figure I.1:** sand filter. [7]

Sand filter is effective when used as a pre-treatment for other methods of disinfection (by solar radiation, by chlorination or by boiling) when raw water available is a little cloudy but not enough on its own. [6.7]

#### **I.4.2.Biological sand filter:**

Biological sand filters provide comprehensive and highly efficient water treatment. due to a constant water level inside the filter, there is installation of a biological activity that eliminates pathogenic germs. The filter is not effective for the first uses because the biological filter has not yet had time to form. It takes about 3 weeks for the bio layer to fully mature.



**Figure I.2:** Biological sand filter. [8]

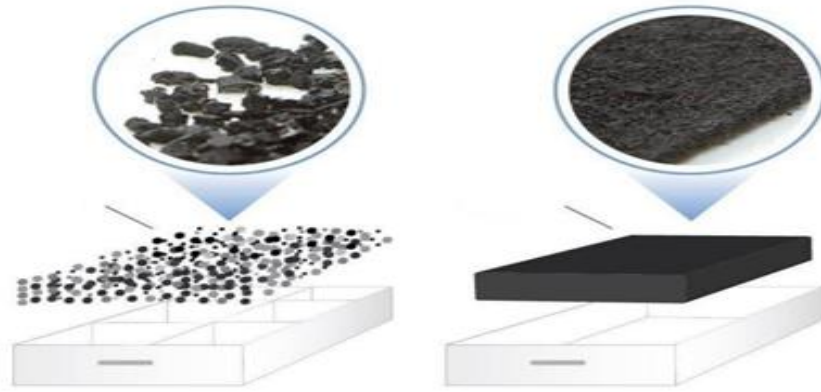
The filter has a diffusion plate to avoid digging up the sand when poured into it the water. The water flows through fine sand. The bottom of the filter is made up of a layer of coarse sand and a layer of gravel. A PVC pipe goes up to the height of the top of the sand layer, in order to keep the water level constant inside the filter. [4]

The main disadvantage of this filter is that the water flows slower and slower as the and as you use it. When the treatment becomes too slow, the sand must be replaced. [4]

#### **I.4.3.Activated carbon adsorption :**

Activated carbon water adsorption has been used for centuries to filter water intended for human consumption. All our water fountains are equipped with a activated carbon water adsorption system that guarantees cleaner and more pleasant water to drink only when it enters the machine. Activated carbon filters affect quality organoleptic of water by removing chlorine, contaminants, particles and others undesirable taste and odor factors. Drinking filtered water is a first step in pure water approach. And the good news is that essential nutrients are preserved. The natural minerals pass through the filter to bring you all their benefits.

Firms, are now commonly used, except in applications in which the better chlorine tolerance of cellulosic membranes is desired. [8]



**Figure I.3:** Activated carbon types. [8]

#### **1.4.4. Softening techniques**

The softening techniques use three processes, namely the decarbonation which eliminates only the calcium and magnesium ions linked to the bicarbonates, this is then a partial softening, and the total softening which eliminates the whole. calcium ions and magnesium, but without changing the alkalinity of the water. The third process is represented by nanofiltration which partially removes calcium, magnesium and bicarbonate ions. Among the decarbonation processes, we find:

- Lime decarbonation;
- Decarbonation with soda;
- Decarbonation on ion exchange resins of the carboxylic type, regenerated by an acid;
- electro-decarbonation.

#### **1.4.5. Softening advantage:**

The choice of the softening technique best suited to a given water is made on the basis of many criteria, namely:

- Presence or absence of suspended matter, iron.
- Nature and composition of hardness.
- Cost of reagents.
- Possibilities of evacuating the eluates.
- Cost of dehydration.
- Possibilities of landfill in CET, spreading, discharge into the sewerage network or reuse of by-products.
- Operating conditions.

### **I .5.Different membranes processes used in water treatment :**

The five membrane processes commonly used in the production of drinking water are RO, NF, ultrafiltration (UF), microfiltration (MF), and electro dialysis/electro dialysis reversal (ED/EDR). Although all five are classified as membrane processes, the technologies and applications are very different in some cases. In general, there are three groups of similar membrane processes: MF/UF, RO/NF, and ED/EDR. [35.36]

Four primary factors distinguish these three groups of membrane processes from each other: the type of membrane, the mechanism of contaminant removal, the process driving force, and the primary application. MF and UF are pressure-driven membrane processes that use microporous membranes to remove particulate matter (including turbidity and microorganisms), via a sieving mechanism, on the basis of size exclusion. These two processes do not remove ions or other dissolved constituents. Although there are some UF membranes that are used in industrial applications to separate high molecular weight organic molecules from solutions, these membranes are not commercially available for municipal drinking water treatment. In some cases, however, as with conventional media filters, MF and UF processes may be used to reduce levels of dissolved organic material (i.e., total organic carbon [TOC] when applied to coagulated water). [35.36]

**Table I.1:** Membrane processes and target contaminants. [35.36]

Membrane Technology	Target Contaminants Removed
MF	<ul style="list-style-type: none"><li>• <i>Giardia</i></li><li>• <i>Cryptosporidium</i></li><li>• Bacteria</li><li>• Turbidity/particulate matter</li><li>• Coagulated organic matter</li><li>• Inorganic precipitates</li></ul>
UF	<ul style="list-style-type: none"><li>• All contaminants removed by MF, <i>plus</i></li><li>• Viruses</li><li>• Large organic macromolecules</li></ul>
NF	<ul style="list-style-type: none"><li>• Divalent ions/hardness</li><li>• Limited monovalent ions</li><li>• Dissolved organic carbon</li><li>• Color</li></ul>
RO	<ul style="list-style-type: none"><li>• All contaminants removed by NF, <i>plus</i></li><li>• Monovalent ions</li></ul>
ED/EDR	<ul style="list-style-type: none"><li>• Dissolved ions</li></ul>

### **I.5.1. Microfiltration and Ultrafiltration MF/UF:**

MF and UF membranes may be manufactured from a number of different materials, including cellulose acetate and synthetic polymers such as polyvinylidene fluoride, polypropylene, polysulfone, polyether sulfone, and polyacrylonitrile. [1] For modern water treatment applications, these MF/UF membrane materials are commonly configured into hollow fibers, although other configurations may be available. [27]

### **I.5.2. Reverse osmosis and Nano filtration :**

RO and NF are also pressure-driven processes; however, these technologies utilize semipermeable membranes to primarily target the removal of dissolved contaminants via a diffusion-controlled separation process. While RO and NF also remove particulate matter, the nonporous, semipermeable membranes can rapidly foul when subjected to significant particulate loading. When high pressure in excess of the natural osmotic gradient of the system is applied to the feed side of the membrane, water is forced through the molecular structure of the membrane surface Courtesy of Black & Veatch while the dissolved solids 0.(i.e., the solutes) are largely rejected. Although solutes can also diffuse through the semipermeable membranes, the rate of mass transfer of these constituents is much slower than that of the water. Consequently, the water that passes through the membrane (i.e., the permeate) contains fewer dissolved solids than does water entering the system (i.e., the feed). [9]

The amount of energy (hydraulic pressure) required to drive the feed water across the membrane depends on the membrane material and thickness, as well as the osmotic pressure of the feed. The osmotic pressure is the pressure on the membrane created by the naturally occurring process of water flowing from a dilute solution (i.e., lower dissolved solids concentration) across a semipermeable membrane to a more concentrated solution (i.e., higher dissolved solids concentration). Thus, energy in the form of hydraulic pressure is required to overcome both the physical resistance of the membrane itself and the osmotic pressure of the system. Because this pressure is applied to force water against the natural osmotic gradient to produce less saline water from more concentrated water, the treatment process is called reverse osmosis. [10]

### **I.5.3.RO AND NF MEMBRANE APPLICATIONS :**

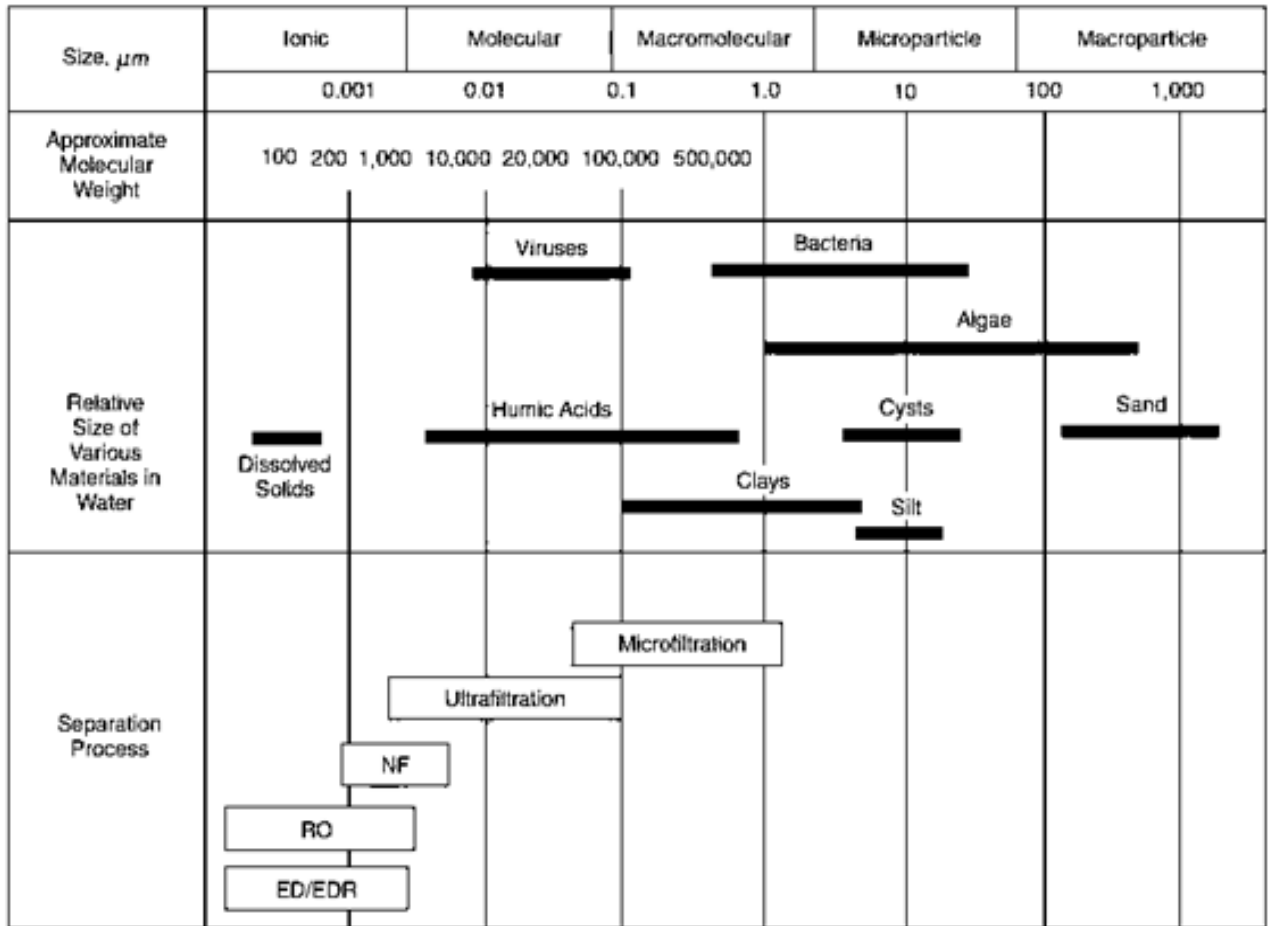
This section describes the major current applications of RO and NF membrane processes, including: desalting, the removal/reduction of DBP precursors, hardness, color, inorganic contaminants (e.g., nitrate, fluoride, arsenic, heavy metals, radionuclides, etc.), synthetic and volatile organic compounds, pathogens, and indirect potable reuse. A short discussion of emerging applications is also provided. Note that RO and NF can also remove suspended solids/particulate matter; however, because the semipermeable membranes are not porous (and therefore not able to be backwashed), any significant particulate loading can rapidly and sometimes irreversibly foul the membranes. Thus, although RO and NF will reduce particulate matter levels (i.e., turbidity, particle counts, etc.), the technology is not applied specifically for this purpose, and pre-treatment to remove particulate matter upstream of the membranes is almost always employed. [11.12]

A flowchart for selecting an appropriate membrane process (including MF, UF, and ED/EDR) is shown in Figure 1-4. Note that this is a very general guideline and does not take into account cost, site-, or application-specific considerations. The figure is primarily intended to serve as an illustrative tool to distinguish the various types of membrane processes on the basis of treatment application . [24]

### **I.5.4.Electrodialysis/Electro dialysis reversal (ED/EDR):**

ED/EDR are electrically driven membrane processes that remove dissolved solids using cation- and anion-selective membranes. However, unlike RO and NF, ED/EDR does not provide a barrier to pathogens and does not remove suspended solids or non-charged, non-ionic constituents. In RO and NF processes, product water is filtered while passing through the membrane. By contrast, with ED/EDR the demineralized product water passes along the membrane surface in a tangential pattern while charged ions are transported through the membrane and concentrated into the brine stream; thus, the product water does not pass through a membrane barrier. ED/EDR has been used primarily to desalinate brackish waters and applied in specialty applications, such as the removal of fluoride or radionuclides. In addition, because ED/EDR does not affect silica concentrations, it may be advantageous in cases in which silica removal is not needed. [15.16]





**Figure I.4:** Membrane and conventional process overview[27.15]

Figure I.4 illustrates the removal abilities of the various types of membrane technology for their respective target drinking water contaminants, based on size of the removed compounds.

Table 1-1 summarizes some of this same information in tabular form, including the various membrane process and target contaminants.

Note that both Figure I.4 and Table 1-1 focus on the target contaminants, not all the contaminants that the various membrane technologies are capable of removing. For example, while RO and NF processes will remove particulate matter, these technologies are generally not applied specifically for this purpose because the membranes will foul rapidly and in many cases irreversibly. [22.21]

**I.6.History of Development :**

One of the first applications for membrane technology was the conversion of seawater to drinking water through the use of the RO process. Early generation membranes were manufactured with cellulose acetate and were much less permeable than those currently used. The disadvantages of early membranes included the high pressure required and the low recovery rate—only 10 to 25 percent of the source water was converted to desalinated permeate. These factors resulted in extensive and cost prohibitive energy requirements. [26]

The first commercial application of RO membranes for brackish water desalting began in the early 1960s using the spiral-wound configuration developed in 1967, by General Atomics. In 1969, E.I. DuPont de Nemours, Inc. (DuPont) introduced the polyamide hollow fine-fiber membrane in the form of the B-9 permeator for brackish water desalting. These brackish water modules generally operated in the pressure range of 300 to 400 psi. The first municipal brackish water RO plant was located at Key Largo, Florida's, Ocean Reef Club. The plant began operation in October 1971 with an initial operating pressure of 600 psi and a capacity of 0.6 mgd, which was later expanded to 0.93 mgd. [13]

In 1974, DuPont introduced the hollow fine-fiber B-10 permeator, the first RO membrane capable of producing potable water from typical seawater in a single pass at operating pressures of 800 to 1,000 psi. Spiral-wound, thin-film composite RO membranes developed for both seawater and brackish water desalting were introduced in the mid- to late 1970s. Feed pressures for the early composite membranes were approximately the same as for the cellulosic and polyamide hollow fine-fiber modules. Dow Chemical Company's introduction of the low-pressure Dowex™ hollow fine-fiber RO membrane led to a major reduction in the cost of brackish water RO facility operation. The first plant to use the new membrane began operation in 1981, at Venice, Fla., with a 1 mgd capacity. The Dowex™ membrane provided salt rejection and fluxes comparable to the standard pressure cellulosic and polyamide membranes at roughly one half the operating. [19]

Low-pressure, thin-film composite, spiral-wound modules were first introduced in the early 1980s by FilmTec Corporation (now part of Dow Chemical Company) and Fluid Systems (now part of Koch Membrane Systems). These composite membranes, currently available from a number of supplier [16.18]

## **I.7.Contaminants removed by RO and NF :**

### **I.7.1.Desalting (TDS Removal) :**

The primary application of RO and NF membranes is desalting (i.e., TDS removal) from saline surface water, brackish groundwater, seawater, tertiary treated wastewater, or industrial process water. The rejection capabilities of different commercially available products can vary significantly, and in many cases particular membranes are selected specifically for a target TDS range. Because incremental increases in TDS reduction boosts the required pressure, translating to higher energy costs, it can be significant in some cases to ensure that TDS is only reduced to the extent such that the desired treatment objective is satisfied. For example, for saline surface waters that may be relatively low in TDS but high enough to adversely impact the taste of the water for a utility's customers, a low-pressure/low-rejection RO membrane may be used with less efficient rejection characteristics. By contrast, in high purity applications that are common in industry or seawater desalination, RO membranes with much higher rejection of TDS are necessary. For potable water applications, the United States Environmental Protection Agency (USEPA) has established a secondary maximum contaminant level (SMCL) for TDS of 500 mg/L. While this federal SMCL is nonenforceable and established for aesthetic quality, this benchmark is often used as a target for treated water quality, particularly when it may be significantly more expensive to further reduce the TDS.

(Note that water quality regulations can vary from state to state, and in some states, the federal SMCLs are enforceable by state mandate.) [30]

Because the removal of TDS by RO and NF is nonselective and relatively high rejections are achieved, the permeate produced by these processes is typically very corrosive and characterized by low alkalinity and minimal buffering capacity. If acid is used as pretreatment to control scaling, the pH may also be low, further compounding the aggressive nature of the permeate. However, RO and NF systems can be designed with appropriate posttreatment processes to produce water that is both low in TDS and well buffered with sufficient alkalinity to help reduce the potential for pipe corrosion in the distribution system . [30]

#### **I.7.2.DBP Precursors :**

Because DBPs are a significant regulatory concern, RO and NF membranes are increasingly applied to remove DBP precursors such as natural organic matter (NOM)/TOC, which can react with various disinfectants used in the water treatment process to form potential carcinogens. These DBPs include total trihalomethanes (TTHMs) and the sum of five haloacetic acids (HAA5), both of which are strictly regulated in the parts per billion range by the Stage 1 and Stage 2 Disinfectants and Disinfection By products Rules. As a result of these low maximum contaminant levels (MCLs), NOM removal is a significant water treatment objective for many utilities. RO or NF as a stand-alone process has been shown in many cases to reduce TOC to less than 0.5 mg/L. RO can also remove TTHMs and HAAs, albeit less efficiently than their precursor material; however, it is uncommon to apply these membranes for DBP reduction after the disinfection process in water treatment plants as a result of the susceptibility of most such membranes to damage from chemical disinfectants. [30]

#### **I.7.3.Hardness:**

NF has become a significant alternative to lime softening for reducing the level of calcium and magnesium ions in naturally hard waters where TDS reduction is not a primary treatment goal. Although RO membranes are also capable of reducing hardness, NF membranes have lower rejection characteristics for monovalent ions, allowing them to be operated at lower pressures while still efficiently removing the divalent ions that contribute to hardness, resulting in energy cost savings. Typically, NF membranes used for softening applications remove more than 95 percent of total hardness. [30]

#### **I.7.4. Color:**

NF is also more effective than lime softening in removing naturally occurring color and DBP precursors, both comprised primarily of organic carbon, and can often be operated more efficiently than RO. NF is generally capable of removing more than 95 percent of color.

#### **I.7.5. Inorganic Contaminants:**

The USEPA currently recognizes RO as the best available technology (BAT) for removing most inorganic compounds (IOCs) regulated under the Safe Drinking Water Act), including radionuclides and arsenic, among many others. This classification reflects the broad-spectrum removal capability of the RO process. The ability of NF to remove IOCs is determined to a large extent by the specific dissolved solids character of the water. NF rejection of specific multivalent cations is a function of solution pH and the speciation of other ionic constituents

present to a greater degree than for the RO process. One of the more common applications of RO for treating a specific inorganic contaminant is nitrate removal. RO is considered an effective nitrate removal process for groundwater supplies polluted by the agricultural use of nitrate-containing fertilizers or septic tank discharges. Rejection of nitrate by some RO membranes is significant; composite polyamide low-pressure brackish water membranes typically exhibit sodium nitrate rejection in the range of 93 to 97 percent . Several RO plants are currently in operation in southern California treating groundwater contaminated with high concentrations of nitrate from past agricultural practices, including those in the cities of Riverside and Tustin, as well as several facilities operated by the Chino Basin Desalter Authority. Note that NF is generally not applied for nitrate removal as a result of its relatively low rejection of this anion compared to RO. [34]

Another inorganic contaminant to which RO is often applied is fluoride. Many groundwater sources in the United States contain elevated levels of naturally occurring fluoride. The USEPA has established a fluoride MCL of 4 mg/L to protect against skeletal fluorosis and a recommended SMCL of 2 mg/L to prevent tooth discoloration. Because levels of naturally occurring fluoride are about the same order of magnitude as the MCL, it is generally not necessary to achieve extremely high rejection, particularly considering that 0.8 to 1.2 mg/L of fluoride in drinking water is recommended for dental health. As a result, in many cases treatment costs can be reduced through the use of split treatment, in which a portion of adequately treated source water is bypassed around the membrane system and blended with the RO permeate. [34]

In general, for water quality constituents or specific inorganic contaminants that are relatively common, RO/NF membrane manufacturers have modeling software that can predict permeate quality fairly accurately. However, for the removal of less common inorganic contaminants for which RO and NF have not been as frequently utilized, rejections are typically based on manufacturer, utility, or independent, third party experience and research. Although increasingly uncommon for many well-known inorganic contaminants, pilot testing can be conducted to quantify or verify rejection levels, if desired. [34]

#### **I.7.6.Synthetic and Volatile Organic Chemicals:**

Many of the synthetic organic compounds (SOCs) regulated by the USEPA in drinking water supplies are pesticide residuals. Pilot testing has been conducted in a municipality–USEPA partnership to evaluate the pesticide removal efficiency of a number of different types of RO membranes for treatment of groundwater contaminated by various agricultural chemicals. This study found that removals were greatest for the polyamide thin-film composite membranes (67 to 95 percent), and it concluded that RO should be considered as a water treatment process for this application. Other studies have assessed the capability of a wide range of NF membranes to remove commonly occurring pesticides to below the 0.1 µg/L . Theoretically, specific SOC rejection is primarily a function of molecular size and degree of ionization. This theory was corroborated by a pilot study demonstrating that the degree of rejection is proportional to the molecular weight. Synthetic organic chemicals with a molecular weight greater than 300 Daltons were completely rejected by one type of NF membrane, while those with molecular weights less than 300 Daltons were only partially

rejected. For these studies, the degree of rejection was proportional to the molecular weight [38].

It is less common for RO and NF to be applied for the removal of volatile organic compounds (VOCs), such as trichloroethylene and tetrachloroethylene, among others, because rejection is generally inefficient (albeit varying by specific compound). In addition, many VOCs are solvents that at higher concentrations may dissolve the glue lines on the membrane elements or damage the membranes themselves. It has also been reported that some VOCs may adsorb onto the membrane, potentially reducing permeability or desorbing into the permeate in concentrations higher than the feed until steady state is achieved [37].

#### **I.7.7.Pathogens:**

Because semipermeable RO and NF membranes are not porous, they have the ability to screen microorganisms and particulate matter in the feedwater. This ability has been verified in a number of studies, such as one that demonstrated that RO membranes provide between 4- and 5-log (i.e., 99.99 to 99.999 percent) removal of viruses normally associated with waterborne disease .

RO is listed as an alternate filtration technology that is effective for the removal of Giardia and viruses (USEPA 1990), such that unlike many other alternate technologies, no piloting or other studies are necessary to demonstrate that the RO process can achieve 3.0-log (i.e., 99.9 percent) Giardia and 4.0-log virus removal when combined with disinfection. The proposed Ground Water Rule also notes the demonstrated ability of RO and NF to achieve 4.0-log virus removal. In addition, under the Long Term 2 Enhanced Surface Water Treatment Rule, both NF and RO are specifically listed as membrane filtration technologies that can be applied to achieve significant Cryptosporidium removal credit [40].

However, it is important to note that RO and NF are not necessarily absolute barriers. RO and NF membranes are primarily designed for the removal of TDS rather than particulate matter, and thus the elimination of all small seal leaks that have only a nominal impact on the salt rejection characteristics is not the primary focus of the manufacturing process. Consequently, RO and NF spiral-wound elements are not intended to be sterilizing membranes and some passage of particulate matter, including pathogens, may occur despite the absence of pores in the membrane. [41].

#### **I.7.8.Indirect Potable Reuse :**

Both RO and NF are being increasingly used in the reclamation of municipal wastewaters serving indirectly as future potable water supplies and other reuse applications. In some of these applications, RO and NF remove many contaminants, including nitrogen, heavy metals, TOC, and pathogens, and subsequently the high-quality permeate is injected into groundwater aquifers for recharge. The underground strata serve as an additional filtration step to achieve natural attenuation of the groundwater supply, in some cases over many years, before it is pumped to the surface again for further treatment and distribution. RO technology is often a critical component for groundwater recharge with reclaimed water. The state of California, for example, currently requires all recycled water to be treated via RO prior to injection. The oldest and most widely known groundwater recharge project is the Orange County Water

District's Ground Water Replenishment System in Fountain Valley, Calif., which originated as Water Factory 21, and has been in service since 1976. Numerous other utilities in California, Arizona, and other states are also practicing, planning, or studying indirect potable reuse using RO. [39]

### **I.8. Emerging Applications:**

Because RO and NF achieve significant rejection/removal of a wide assortment of potable water contaminants, these processes are often among the first treatment technologies considered for a variety of emerging applications. For example, perchlorate—an inorganic anion—is one such emerging contaminant that is only effectively removed by a limited range of technologies, such as RO and ion exchange. RO and NF are also likely to be among the BATs for removing contaminants such as endocrine disruptors and pharmaceutically active compounds, two broad classes of contaminants that are just beginning to be studied and quantified in drinking water sources. As improved analytical techniques continue to reveal previously unknown contaminants, it is likely that the number of applications for RO and NF will likewise increase. [39]

### **I.9. Prevent Membrane Damage :**

If an RO or NF membrane is chemically altered by substances in the source water, the membrane performance efficiency will decrease (such as a decline in salt rejection or loss of productivity). Chemical attacks on membranes generally occur via oxidation or hydrolysis, both of which are easily controlled. The susceptibility to chemical attack is a function of the chemical composition of the membrane polymer. [43]

Cellulose acetate polymers are subject to a loss of rejection properties because of a gradual hydrolysis of the acetyl groups on the polysaccharide backbone. The rate of hydrolysis is a function of pH. Mineral acid is generally required to adjust the pH of most source waters into the 5.5 to 6 range to maximize cellulosic membrane life. Polyamide membranes are capable of operating over a rather broad pH range: from pH 2 to 11 on a continuous basis and up to pH 12 for short-term cleaning. When a polyamide membrane is used for processing water at the higher pH (>8) range, careful consideration must be given to controlling calcium carbonate scaling, as discussed in greater detail in this section. [44]

Although polyamide membranes are not particularly pH sensitive, all varieties of these membranes are intolerant of chlorine and certain other strong oxidizing agents to some extent.

Dechlorinating through the addition of sodium metabisulfite in excess of stoichiometric requirements should protect the more tolerant polyamide membranes from chlorine attack. For the most susceptible polyamide compositions, chlorine should be totally avoided as a part of pre-treatment. Cellulosic membranes, on the other hand, will tolerate chlorine at levels used to provide disinfection of potable water supplies, typically 1 mg/L Cl<sub>2</sub> or less. However, over long membrane service life, the chlorine will facilitate oxidation of the membrane material. In some cases, such as municipal wastewater tertiary treatment, chloramines have been successfully used with composite polyamide membranes for biological control. [29]

#### **I.9.1. Mitigate Membrane Fouling :**

An accumulation of one or more foreign substances on the surface of a membrane will result in a loss of productivity. Higher operating pressures will then be required to maintain water production (flux) and quality. Membrane fouling generally occurs by one of the following mechanisms:

- Deposition of silt or other suspended solids that were inadequately removed by the pre-treatment
- Inorganic scale deposits formed because of precipitation of sparingly soluble salts or silica
- Biological fouling caused by excessive microbial growth
- Interaction of organics with the membrane

In some instances, pre-treatment is required to remove algae; certain metals, such as iron and manganese; or other particulate matter. The need for such treatment is dictated by the source water quality, the permeate standards, and the need to protect the membranes. Only the most commonly encountered contaminants are discussed in this manual. [29]

#### **1.10. Chemical Disinfection by ozone :**

Initially, ozone was used extensively in Europe to purify water. Ozone, a molecule composed of three atoms of oxygen rather than two, is formed by exposing air or oxygen to a high-voltage electric arc. Ozone is much more effective as a disinfectant than chlorine, but no residual levels of disinfectant exist after ozone turns back into O<sub>2</sub>. Ozone was always expected to see increased use in the United States as a way to avoid the production of trihalomethanes due to the usage of chlorine. While ozone did break down organic molecules, sometimes this was a disadvantage, as ozone treatment produced higher levels of smaller (lower molecular weight) molecules that provide an energy source for microorganisms or higher levels of toxicity. If no residual disinfectant is present (as would happen if ozone were used as the only treatment method), these microorganisms cause the water quality to deteriorate rapidly during the storage and distribution. Ozone also changes the surface charges of the dissolved organics and colloiddally suspended particles. This causes micro flocculation of the dissolved organics and coagulation of the colloidal particles. [17]

Ozone (O<sub>3</sub>) is generated on-site at water treatment facilities by passing dry oxygen or air through a system of high-voltage electrodes. Ozone is one of the strongest oxidants and disinfectants available. Its high reactivity and low solubility, however, make it difficult to apply and control. Contact chambers are fully contained, and non-absorbed ozone must be destroyed or recycled prior to its release into the atmosphere to avoid corrosive and toxic conditions. Ozone is particularly effective against spores and cysts. Extensive studies on various factors affecting *Giardia muris* cyst inactivation by ozone are reported. These encompass factors like temperature, turbidity, pH, ozone dose, and contact time. It was found that residual and utilised ozone both had important influences in *G. muris* cyst inactivation. It was more difficult to achieve 2 or 3 log inactivation of *G. muris* cysts in the natural waters at 22°C than at 5°C . *Encephalitozoon intestinalis*, a microsporidian pathogen for humans and animals that is detected in surface water, has been listed as a major potential emerging waterborne pathogen by the USEPA. Ozone has been very effective in rendering these spores

inactive, and its CXT values were an order of magnitude lower than the CXT values of chlorine in a comparative study .

Ozone has several advantages, such as being one of the strongest oxidants, not producing chlorinated THMs or HAAs, and being effective against *Cryptosporidium*, to name some. On the other hand, its limitations are also numerous. Its process operation and maintenance requires a high level of technical competence, it provides no protective residual, and it reacts with bromine-containing compounds and forms brominated by-products like bromate and brominated organics, and forms non-halogenated by-products such as ketenes, organic acids, and aldehydes, which could be more toxic than the original contaminants. Ozone breaks down more complex organic matter, and smaller compounds can enhance microbial regrowth in distribution systems and increase disinfectant by-product (DBP) formation during secondary disinfection processes. Its higher operating and capital costs than chlorination are also a major limitation. Difficulty to control and monitor, particularly under variable microbial and organic load conditions, also adds to the list of ozone's demerits as a disinfectant. [17]

The mechanism of action of ozone on microbes has been studied extensively. The primary attack of ozone occurs on the double bonds of the fatty acids (lipid layers) in the cell wall and membrane, and there is a consequent change in the cell wall permeability and cell contents leak out, causing death. Attack on nucleic acids inside the cells is yet another mechanism of cell death. Thymine was more sensitive to ozone attack than were cytosine and uracil . In the case of viruses, a complete loss of viral proteins was responsible for the death of viruses , as a result of an ozone attack. [17]

## **1.11.SOURCE WATER SUPPLY :**

### **1.11.1Surface Water:**

Surface waters present numerous technical problems for pretreatment systems for membrane treatment processes. RO and NF membranes are very sensitive to the concentration of particulates in the water. A surface water source may require construction of comprehensive pretreatment facilities to prevent particulate material from plugging the membranes. Facilities for the coagulation/removal of organic compounds, such as humic acids, may also be required to mitigate membrane fouling. Beach or shallow wells are sometimes used to avoid direct intakes of surface water. The quality of the surface water supply source can be monitored for many years to assess needed changes in a treatment facility's design.

Fresh water. Intakes for most fresh surface water treatment facilities, regardless of the treatment process, are submerged pipes in rivers, lakes, or reservoirs. A fixed-position intake pipe is commonly found where sufficient data are available to determine that the pipe will always be submerged regardless of climatic conditions. However, many utilities prefer multiple-depth openings to optimize source water quality.

The elevation of the intake pipe should be at least a few feet below the water surface to avoid inflow of floating debris and higher concentrations of aquatic plants, which tend to stay in the photic zone (i.e., those areas of water that are penetrated by light). The intake pipe must also



be placed as far from the bottom as possible to avoid collection of sediments. Intakes possessing the ability to withdraw water from multiple depths are recommended in waterways that experience seasonal anaerobic conditions in the benthic region to avoid membrane fouling resulting from increased manganese and iron concentrations. Additional details are contained in Water Treatment Plant Design 4th ed. [7]

The end of the intake should be screened to avoid the entrainment of large debris. The diameter of the intake pipe should be sized to adequately to permit the required flow, while maintaining acceptable approach velocities to prevent sediment entrainment and the scouring of vegetative growth from the pipe. The presence of zebra mussels in many waterways in North America also requires additional considerations in the design of intake systems. Provision must be provided for the maintenance of the intake screens and pipe work, including disinfection and physical removal of zebra mussels from the facilities. In many cases, provision of two parallel intake facilities is recommended.

Seawater or brackish water. Intakes from tidal surface water bodies feeding RO water treatment facilities share many common problems with freshwater systems. However, a number of other issues, such as the susceptibility of a facility to corrosion, storm damage, and marine organism growth, must be considered.

A number of existing seawater membrane treatment facilities use surface intake systems. Many of these systems are located in the Middle East or on islands, both natural and artificial (i.e., oil platforms). Most large-scale membrane facilities using direct surface water intakes are located adjacent to large tidal water bodies, such as the Atlantic Ocean or the Arabian (Persian) Gulf. Some critical problems in the design of intakes in these areas are how far offshore and how deep the intake should be placed and what type of terminal should be used.

Oceanographic investigations for intakes are required to assess storm impact potential, potential growth rates of attaching benthic marine organisms (such as corals and sponges), and near-shore water quality fluctuations. The terminal must be below a water depth affected by damaging orbital storm wave motion and yet far enough offshore to avoid the near-shore sediment transport area where storms can cause suspension of large quantities of sediment. Water quality changes must also be considered in the establishment of the intake point. In the Arabian Gulf, water quality and temperature vary significantly. In some instances, the final intake point may lie several miles offshore. Alternative designs should be considered before a surface intake is installed into any tidal water body.

Similar to fresh water surface sources, the end of the intake should be designed to limit entrainment and impingement. Marine growth may also require additional considerations in the design of the intake system. Provisions for intake screen and pipework must be provided, including disinfection and physical removal of marine growth from the facilities. In many cases, provision of two parallel intake facilities is recommended.

### **1.11.2.Groundwater :**

Under steady-state conditions, groundwater normally provides a chemically stable, low turbidity source of water over a long time. However, in coastal areas with high groundwater

withdrawal quantities, groundwater quality can vary dramatically. Over pumping can cause saltwater intrusion, which in turn may cause a membrane treatment plant to have problems unless it is designed to accommodate the increased concentration of dissolved solids.

In the development of a groundwater source, the aquifer must be carefully tested and modelled to determine the anticipated short- and long-term changes in water quality. Groundwater quality can change depending on the nature of the aquifer used or the presence of over pumping. The potential for significant water quality changes over time should be considered in the design of the membrane treatment system.

#### **I.12. PHARMACEUTICAL WATERS:**

Water and steam used in the pharmaceutical industry and related disciplines are classified by various pharmacopeia's. The U.S. Pharmacopeia (USP) classifies compendia waters as follows:

- Water for Injection
- Bacteriostatic Water for Injection
- Sterile Water for Inhalation
- Sterile Water for Injection
- Sterile Water for Irrigation
- Purified Water
- Sterile Purified Water
- Water for Haemodialysis
- Pure Steam
- Drinking Water (indirectly)

With the exception of Drinking Water, USP Purified Water, USP Water for Injection, and USP Pure Steam, the classifications listed above refer to “packaged water” (USP, 2010(a)). Drinking Water, USP Purified Water, and USP Water for Injection are the primary waters used for most pharmaceutical applications, and are the primary topic of this book. Validation is required for all compendial water systems producing USP Purified Water or USP Water for Injection, with the exception of Drinking Water. Drinking Water used in a specific application generally requires “commissioning/ qualification” to an “internal” specification, verifying that the quality of the product water, from both a chemical and microbiological standpoint, does not vary from established internal specifications with time. This qualification process is often used not only to maintain control of product water but also to document the nature of the system by preparing and executing documents similar to those used for compendial water systems. Obviously, the internal specifications established for a qualified system may parallel a particular USP official monograph specification, such as that for Purified Water. Finally, certain applications may expand the USP requirements for a particular grade of water. As an example, many biotechnology water specifications require “low bacterial endotoxin” Purified Water. For such application, the biotechnology company would validate the system as a USP Purified Water system and incorporate an internal bacterial endotoxin specification. Chemical, bacteria, bacterial endotoxin, and other parameters associated with each of the pharmaceutical grades of water identified above are addressed individually in this chapter.

USP is prepared and published by The United States Pharmacopeia Convention, a private organization. The material within USP is established by “Expert Committees,” circulated to the general public for comment and review, and revised after acceptance. The Expert Committees as well as the review processes include U.S. Food and Drug Administration comment, review, and approval. Since new volumes of USP are published periodically, it is suggested that reference to USP states the number of the most recent addition and/or most recent edition including all “Supplements.” [7]

### **1.13. DEFINITION OF PHARMACEUTICAL WATERS—EP, JP, BP, etc**

As indicated, water and steam used in the pharmaceutical industry and related disciplines are also classified by other pharmacopeia’s, including the European Pharmacopeia (EP), Japanese Pharmacopeia (JP), and the British Pharmacopeia (BP). Over the past several years, there have been many attempts to “harmonization” descriptions, specifications, and method of production for compendial waters. While significant progress has been achieved, specific differences of importance will be addressed within this chapter. [19]

### **1.14. CHEMICAL SPECIFICATIONS :**

#### **1.14.1 Drinking Water:**

From a chemical standpoint, water classified as Drinking Water, for applications such as some initial rinsing operations and active pharmaceutical ingredient manufacturing operations, must meet the U.S. Environmental Protection Agency’s (EPA) National Primary Drinking Water Regulations (NPDWR), or comparable regulations of the European Union, Japan, and/or World Health Organization, as applicable, for “Drinking Water.” This would include but not be limited to the parameters presented in Table 1.1 for U.S. EPA Drinking Water (EPA, 2010). It is important to note that the NPDWR will change with time, incorporating additional parameters or changing regulated item concentrations. It should be emphasized that all validated USP systems, as well as systems using Drinking Water, should have access to correspondence identifying changes to these regulations.

As discussed further in subsequent chapters of this book, it is highly recommended that supplemental analysis for Drinking Water, including feedwater to a USP Purified Water or USP Water for Injection system, be considered. The nature and type of analyses are dictated by the intended use of the Drinking Water. For example, if groundwater is used for an initial rinsing step during applications such as “clean-in-place” (CIP) or the production of an active pharmaceutical ingredient, it may be appropriate to treat the water through a particulate removal filter and/or water softening system. If water softening is used, the presence of high molecular weight multivalent cations, such as barium, strontium, and aluminum, in the feedwater should be identified. As discussed in chapter 3, these compounds will affect the Standard Operating Procedures (SOPs), specifically the regeneration salt dosing and concentration, during regeneration of the water softening system. Multivalent cations, such as calcium and magnesium, are not included in the NPDWR, but affect the performance of the system.

Other specific components are critical to different water purification unit operations. Another example is the level of naturally occurring organic material (NOM) in a surface water supply

to a USP Purified Water system. Both anion resin and reverse osmosis (RO) membranes will foul with organic material. The level of the NOM in feedwater will not only dictate the nature of pretreatment equipment but also establish an analytical monitoring program clearly demonstrating that the selected pretreatment operations “protect” the anion resin within the ion exchange system or RO membranes from fouling. [1]

#### **1.14.2.Purified Water :**

Chemical specifications for USP Purified Water are outlined in the Official Monograph by referencing Physical Tests chapters for conductivity and total organic carbon (TOC). Physical Tests Section provides the TOC specification, capability of the TOC analyzer, “system suitability” requirements, and calibration requirements. The section does not set forth requirements for online measurement versus “grab” sampling and laboratory analysis. Further, the section does not state the frequency of analysis. The TOC limit for USP Purified Water is 0.50 mg/L. The specification agrees with the current EP specification.

USP Physical Tests Section outlines the specification for conductivity, method of determination, instrument (meter and probe), calibration requirements, etc. This section outlines a three-stage test method that compensates for the presence of carbon dioxide and pH. The most restrictive specification, “Stage 1,” is 1.3 mS/cm at 25°C or 1.1 mS/cm at 20°C, in agreement with the EP specification. The section does not set forth requirements for online measurement versus grab sampling and laboratory analysis. Further, the section does not state the frequency of analysis.

Other pharmacopeias may have additional testing requirements. As an example, the EP contains a nitrate specification of 0.2 mg/L, maximum. It is important to review the various pharmacopeial requirements for countries where products will be sold.

As indicated previously, certain systems may require supplemental sampling and monitoring for important contaminants not addressed specifically within the pharmacopeia. As an example, for a Purified Water system using ozone for microbial control (storage and distribution system), control of residual disinfecting by-products such as trihalomethanes may be critical. Ozone will oxidize trihalomethane compounds to carbon dioxide, which will react with water yielding the hydronium and bicarbonate ion, increasing the conductivity of Purified Water.

#### **1.14.3.USP Sterile Purified Water :**

USP Sterile Purified Water is USP Purified Water that is sterilized and suitably packaged. It contains no antimicrobial agents. The chemical specifications for USP Sterile Purified Water are defined in the Official Monograph and currently include the following:

- Oxidizable substances
- Conductivity
- Unlike Drinking Water, USP Purified Water, or USP Water for Injection, packaged waters must meet laboratory-type chemical tests. [19]

#### **1.14.4.USP Water for Injection:**

The chemical specifications for USP Water for Injection are identical to the chemical specifications for USP Purified Water

The EP chemical specification for Water for Injection includes a nitrate specification (0.2 mg/L, maximum) (EP, 2010(a))

#### **1.14.5.USP Sterile Water for Injection :**

USP Sterile Water for Injection is USP Water for Injection that has been sterilized and suitably packaged. It contains no antimicrobial agents or added substances. It must pass the physical and chemical tests set forth in the current edition of USP with all supplements.

#### **1.14.5. USP Bacteriostatic Water for Injection:**

Bacteriostatic Water for Injection is USP Water for Injection sterilized and suitably packaged. It contains one or more suitable antimicrobial agents. It must pass the physical and chemical tests set forth in the current edition of USP with all supplements.

#### **1.14.6.USP Sterile Water for Irrigation :**

USP Sterile Water for Irrigation is USP Water for Injection sterilized and suitably packaged. It contains no antimicrobial agents or other added substances. It must pass the physical and chemical tests set forth in the current edition of USP with all supplements

#### **1.14.7.USP Sterile Water for Inhalation :**

USP Sterile Water for Inhalation is USP Water for Injection sterilized and suitably packaged. It contains no antimicrobial agents, except when used in humidifiers or other similar devices that may be liable to contamination over a period of time, or other added substances. It must pass the physical and chemical tests set forth in the current edition of USP with all supplements . [19]

**CHAPTER II:**  
**TABUK**  
**PHARMACEUTICALS**  
**WATER TREATMENT**  
**STATION**

### **II.1.References and related documents :**

- European pharmacopeia in vigueur
- Documents on qualification of water treatment system
- journal official Algerian edition 1998
- Microbiological standards for drinking water
- Potable water prelevement , purified SQP-CQ-001
- Control procedure of potable and purified water SOP-CQ-002
- Purified water Procedure control SOP-CQ-003

### **II.2.System conception :**

we represent below Tabuk Pharmaceuticals water station illustration that gathers all of the pretreatment system for filtration and softening of raw water and a purification system used in the industry .





### **II.3. Pretreatment system:**

this system is used for a filtration and softening of raw water to produce fresh drinking water and its mainly composed of:



**Figure II.2 :** Microfiltration process membranes

**II.3.1. Sand filter:** this treatment reduces the rate of silica and also other large particles suspended in the water, it sets up a physical barrier to the passage particles

**II.3.2 .Two softeners :** Softeners reduce the hardness of the water. they operate in parallel with the staggered regeneration cycles. the fresh water generated by the softener is stored in a storage tank to provide the necessary buffer required for daily use, a minimum level of chlorine(NMT0.25ppm) is maintained during the process to minimize bacterial growth in the softener bed

**II.3.3. A dechlorinating station On activated carbon and micro filtration:** the activated carbon eliminates the chlorine present in the water, its presence can destroy the reverse osmosis membranes, water will then go through filtration cartridges of 1u to remove particles which are likely to clomerate the Bi-osmosis.

### **II.4. Production system for purifying water:**

in our station, we have a purification system for a membrane filtration right after the pretreatment,



**Figure II.3:** reverse bi-osmosis

the system is composed of the following parts:

**II.4.1. Bi-osmosis :** reverse osmosis is a process of removing molecules by infiltration through semi-permeable membranes, It also removes suspended particles and reduces organic carbon total present in water .

**II.4.2. UV lamp:** An ultraviolet system and set up for microbial decontamination and ozone removal

**II.4.3 - Storage of distribution systems :**

mainly composed of:

**II.4.3.1. water storage tank purify:** The purified water produced by the water system is introduced into a tank before distribution to user points

**II.4.3.2. distribution loop:** the purified water stored in the tank is supplied to various points of use in the loop installation via a piping / distribution network which is a closed loop system.

## II.5. Purified water 's production / distribution process diagram :

the following diagram represent the water treatment chain highlighting different filters used during our process .

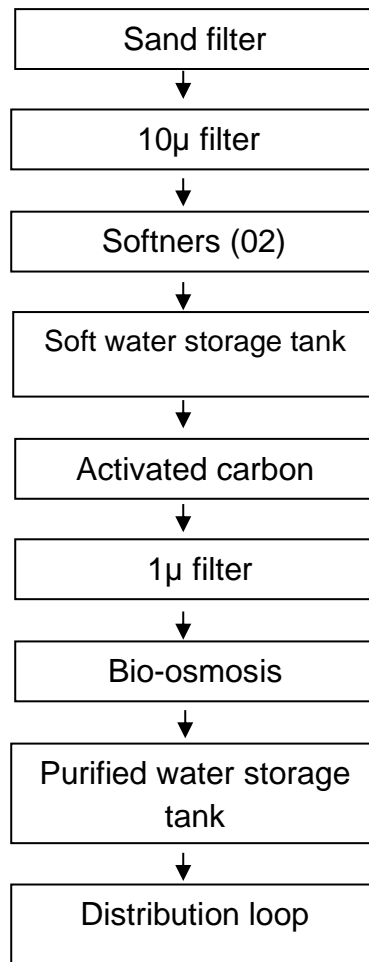


Figure II.4: Diagram of water treatment's chain

## II.6. Equipments and materials :

The equipment used for qualification must be qualified and are as follows :

Table II.1: Equipements and brands

Equipement	Brand
pH Meter	Mettler Toledo
Conductivity meter	Mettler Toledo
Autoclave	Systec
Incubators	Blinder
Hood	Telstar

## II.7.Sampling -points :

The following table groups together the different water quality check-points .

**Table II.2: sampling points localisation**

Sampling points	Localisation
<b>Pretreatment phase (1)</b>	
Raw water	Pe 001
Raw water	Pe 058
Outflow filter 10 $\mu$ F113	Pe 115
Inflow filter 10 $\mu$ F114	Pe 116
Outflow softner 01	Pe 146
Outflow softner 02	Pe 147
Outflow GAC	Pe 139
outflow Microfiltration 1 $\mu$ F137/F138	Pe 140
<b>Purified water phase (2)</b>	
RO inflow	Pe 269
1st floor RO inflow	Pe 224
RO outflow (Purified water)	Pe 254
<b>Distribution loop ( Purified water phase 3)</b>	
Upstream water UV sterilizer (loop return)	Pe 309
Water Downstream UV Sterilizer	Pe 325
<b>Points de puisage eau purifiée</b>	
Laundromat	Pe401
Solution preparation room	Pe402
Granulation 2 (technical part )	Pe403
	Pe404
Granulation 1 (technical part)	Pe405
	Pe406
Coating 2	Pe407
coating1	Pe408
Physicochemical laundromat	Pe409
Microbiology	Pe410

## II.8 . Pre-treatment : (phase 1)

we mention below the defferent physico-chemical and microbiological tests for the pretreatment water (phase1 ) .

### II.8.1. .Physico-chemical Tests :

#### II.8.1.1.pH :

For chlorine disinfection to be effective, the pH should preferably be below 8.0

- Standard: in our case it should be [6.5 - 9]

#### II.8.1.2. Conductivity:

Conductivity depends on the concentration of dissolved conductive salts, so measuring it gives an idea of the salinity of the water.

Fill a 100ml test tube with the water sample to be analyzed by immersing the electrode and removing air bubbles.

The conductivity value in  $\mu\text{S} / \text{cm}$  is read directly on the conductivity meter.

- Standard: in our case it should be  $\leq 2800 \mu\text{S} / \text{cm}$

NB:

- Standard of point Pe 224 (Water permeate 1st osmosis stage): should be  $\leq 50 \mu\text{S} / \text{cm}$  [39] [44]

### **II.8.1.3. Evaporation's residue:**

Evaporate 100ml of water to be analyzed on a hot plate to dryness and dry in an oven at 100-105 ° C for 1 hour. Place the sample in a desiccator for 10 min.

The mass of the residue obtained must be between 100 and 2000 mg / l.

- Standard: in our case it should be [100 - 2000] mg / l. [40] [44]

Important: The handling is done with hot tweezers and avoid any contact with the fingers.

### **II.8.2. Microbiological control:**

#### **II.8.2.1. Enumeration of total viable aerobic organisms (DGAT):**

Under a laminar flow hood, assemble the membrane filtration system.

- Place the 0.45  $\mu\text{m}$  filter membrane
- Shake then filter 100 ml of the sample.
- Place the membrane on a petri dish previously poured with PCA or TSA agar for the control of drinking and softened water.
- Make sure that the membrane adheres fully to the agar and that there are no air bubbles trapped underneath.
- Incubate at 30-35 ° C for 5 days, and examine the dishes after 48 hours and 3 days of incubation. [42]

**Note:**

- Carry out a dilution to 1 / 10th only if the count exceeds 200 cfu.
- Count the colonies which have developed on the surface of the filter and express the result in number of cfu / ml. (If a dilution is carried out, report the number of CFUs to the initial dilution)
- Standards: in our case it should be The water passes the test if the number of ufc is < 500 cfu / ml [42]

#### **II.8.2.2. -Detection of total coliforms:**

- Fit the membrane filtration system.
- Place the 0.45  $\mu\text{m}$  filter membrane
- Shake the sample to be analyzed then filter a volume of 100ml.

- Place the filter on 1 petri dish previously poured with Mc Conkey agar.
- Make sure that the filter adheres fully to the agar and that there are no air bubbles trapped underneath.
- Incubate the dishes at 35-35 ° C for 72 hours.
- Standards: in our case it should be the absence of red non-mucoid colonies
- Rapid confirmation tests for coliforms:
  - Gram stain: non-sporulated gram negative bacilli.
  - Oxidase test: negative
  - VBL bell broth: middle turn from green to yellow with gas production.
  - Schubert medium: fermentation of mannitol with production of gas and a red coloration after addition of the kovacs reagent (the same reaction can be obtained with the indole urea medium)
- Perform a full identification using the API20 E gallery. [42]

### **II.8.2.3.Detection of Pseudomonas aeruginosa:**

- Fit the membrane filtration system.
- Place the sterile 0.22µm cellulose nitrate filter membrane
- Shake the sample to be analyzed then filter a volume of 100ml.
- Place the filter on a Petri dish previously poured with Cetrimide agar.
- Make sure that the filter adheres fully to the agar and that there are no air bubbles trapped underneath.
- Incubate at 30-35 ° C for 72 h.
- Standards: in our case it should be if the water passes the test if there is no growth of fluorescent green colonies.
- Rapid confirmation tests for Pseudomonas aeruginosa:
  - Gram stain: non-sporulated gram negative bacilli.
  - Oxidase test: positive
  - Presence of fluorescence under UV
  - Growth at 44 ° C on cetrimide agar: positive
  - Growth on King A: positive with demonstration of pyocyanin by addition of 2 ml of chloroform and production of a blue tint.
  - Growth on King B: positive
  - Nitrate broth: positive with development of a brick red precipitate after addition of nitrate reagents 1 and 2.
- Perform an identification[42]

### **II.9. Purified water :**

we mention below the different physico-chemical and microbiological tests for the purified water ( phase 2 )

## II.9.1. Standards :

**Table II.3 : tests standards**

Tests	Specifications
Aspects	Clear and colorless liquide
Conductivity	4.3 $\mu$ S.cm at 20°c 5.1 $\mu$ S.cm at 25°c
Heavy metals	Max 0.1 ppm
Nitrate	Max 0.2 ppm
Oxidizable substance	$\leq$ 0.5 mg/l
Microbiological contamination: -DGAVT -PSEUDO AEROGENA -COLIFORM FECAUX	<100ufc/ml Absence Absence

## II.9.2. Physico-chemical tests :

### II.9.2.1..Nitrates determination:

- Preparation of solutions:
- Diphenylamine solution:
- Weigh 100 mg of diphenylamine in 100ml of sulfuric acid.
- 2 ppm nitrate solution (NO<sub>3</sub>):
- Dissolve a quantity of potassium nitrate corresponding to 0.815 g of KNO<sub>3</sub> in purified water and make up to 500.0 ml with the same solvent. Dilute 1 ml of this solution in 100 ml of purified water.
- Dilute 2ml of the resulting solution in 10ml of purified water. [45]

#### -Operating mode:

Reference solution:

In a test tube placed in ice-cold water, place a mixture of 4.5 ml of water free of nitrates and 0.5 ml of a 2 ppm solution of nitrates (NO<sub>3</sub>), add 0.4 ml of a 100 g / l potassium chloride solution, 0.1 ml of diphenylamine solution then, drop by drop and stirring, 5 ml of nitrogen-free sulfuric acid Place the test tube in a water bath at 50 ° C. [41]

Sample solution:

In a test tube placed in ice-cold water, place 5 ml of purified water in bulk and add 0.4 ml of a 100 g / l potassium chloride solution, 0.1 ml of diphenylamine solution then, dropwise and with stirring, 5 ml of nitrogen-free sulfuric acid Place the test tube in a water bath at 50 ° C.

- Standard: in our case it should be below [ $\leq$  0.2 ppm] [45]

If, after 15 min, a blue color appears, it is not more intense than that of a control prepared simultaneously and under the same conditions.

### **II.9.2.2.Oxidisable substances determination:**

#### **Preparation of solutions:**

- Diluted sulfuric acid:

To 60 ml of purified water add 5.5 ml of sulfuric acid. Leave to cool and make up to 100 ml with the same solvent.

- Potassium permanganate 0.2 M:

Dissolve 3.2 g of potassium permanganate in purified water and make up to 1000.0 ml with the same solvent. Heat the solution in a water bath for 1 h, allow to cool and filter through a sintered glass filter. [45]

- **Operating mode:**

Heat a mixture of 100 ml of purified water, 10 ml of dilute sulfuric acid and 0.1 ml of 0.02 M potassium permanganate to a boil for 5 minutes.

Standard: The solution remains slightly pink colored.

### **II.9.2.3.Heavy metals determination:**

#### **Preparation of solutions:**

- 0.1M nitric acid:

Dissolve 96.6 g of nitric acid in purified water and make up to 1000ml with the same solvent.

- Preparation of the pH 3.5 buffer solution:

Dissolve 25.0 g of ammonium acetate in 25 ml of purified water and add 38.0 ml of hydrochloric acid R1. Adjust the pH, if necessary, with dilute hydrochloric acid or dilute ammonia R1 and make up to 100.0 ml with purified water.

- Preparation of the thioacetamide reagent:

To 0.2 ml of thioacetamide solution, add 1 ml of a mixture of 5 ml of purified water, 15 ml of 1 M sodium hydroxide and 20 ml of 85% glycerol.

Heat in a water bath for 20 seconds. Prepare extemporaneously.

- Preparation of the thioacetamide solution:

Dissolve 40g of thioacetamide in 1000ml of purified water.

- Preparation of the 0.1% lead solution:

Dissolve in purified water an amount of lead nitrate corresponding to 0.400 g of  $Pb(NO_3)_2$  and make up to 250.0 ml with the same solvent.

- Preparation of the 100 ppm lead solution:



Dilute 10ml of the 0.1% lead solution in 100ml of purified water immediately before use

- Preparation of the 10 ppm lead solution:

Dilute 10 ml of the 100 ppm lead (Pb) solution in 100 ml of purified water immediately before use.

- Preparation of the 2 ppm lead solution:

Dilute 10 ml of the 10 ppm lead solution in 50 ml of purified water immediately before use.

- Solution at 1 ppm of lead (Pb):

Dilute 10 ml of the 100 ppm lead solution in 100 ml of purified water immediately before use.

Procedure: according to Method A for heavy metals

- Aqueous solution of the substance to be examined:

To 200 ml of purified water, add 0.15 ml of 0.1 M nitric acid and heat in a water bath in a glass capsule, until the volume has reduced to 20 ml.

- Sample solution

12 mL of the aqueous solution of the test substance.

- Control solution

Mix 10 mL of a 1 ppm lead (Pb) or 2 ppm lead (Pb) solution, as prescribed, and 2 mL of the aqueous solution of the test substance.

- Blank solution

Mix 10 mL of purified water and 2 mL of the aqueous solution of the test substance.

To each solution (Sample, Control and blank solution), add 2 mL of pH 3.5 buffer solution. Mix. Add 1.2 mL of thioacetamide reagent then mix immediately. Examine the solutions after 2 min.

- System compliance:

Compared to the blank solution, the control solution shows a slight brown color. [45]

**Note:**

Any brown coloration of the sample solution is not more intense than that of the control solution.

If the test result is difficult to assess, filter the solutions through a suitable membrane filter (nominal pore size 0.45  $\mu\text{m}$ ). Carry out the filtration slowly and regularly by moderate and

constant pressure on the piston then compare the spots obtained on the filters with the different solutions.

- Standard: in our case it should be  $\leq 0.1$  ppm. [43.40]

#### **II.9.2.4. Conductivity:**

##### **Operating mode:**

Rinse the conductivity measuring cell several times with purified water. Immerse the measuring cell in the sample of purified water. Once the conductivity meter has stabilized, read the temperature and conductivity value of the purified water sample (without temperature compensation)

##### **II.9.2.5. Calculating conductivity standard :**

$$C = C_i + \frac{(C_s - C_i)(T_E - T_i)}{T_s - T_i}$$

including :

**C<sub>i</sub>** : conductivity which its close temperature is inferior than the sample's temperature

**C<sub>s</sub>**: conductivity which its close temperature is superior than the sample's temperature

**T<sub>E</sub>**: sample's temperature while measuring the conductivity

**T<sub>i</sub>** : inferior close temperature

**T<sub>s</sub>**: superior close temperature

- Standard : in our case it should At 20 ° C the maximum conductivity value is 4.3  $\mu\text{s}\cdot\text{cm}^{-1}$

At the temperature of the purified water sample, the maximum permissible conductivity value is that calculated by the above formula.

#### **II.10. Sampling methodology:**

##### **II.10.1. Sampling for microbiological control:**

At first we have to affix a sample label. And Clean the tap with 70 ° alcohol and dry well using absorbent paper after that we will have to Open the tap and let the equivalent of 10 liters of water flow freely without closing the tap, quickly open the bottle, then fill it (do not fill it to the rim in order to facilitate mixing) the quantity to be withdrawn per point is 500 ml, then close immediately, If the sample checks are not carried out within two hours of taking the samples, keep them in a cool place at 2-8 ° C for a maximum of 24 hours. [41.44]

### **II.10.2. Sampling for physicochemical control:**

At first we have to affix a sample label. And Clean the tap with 70 ° alcohol and dry well using absorbent paper after that we will have to Open the tap and let the equivalent of 5 liters of water flow freely without closing the tap, rinse the bottle 3 times then fill it.

The quantity to be taken for the physic chemistry per point is 1000 ml and then immediately close the bottle, If the samples are not processed the same day, store them in a cool place at 2-8 ° C for a maximum of 24 hours.

NB: The vials must be labelled [41.44]

# **CHAPTER III:**

## Results and discussing

In this chapter, we present the results and the discussion of the analyzes carried out on the purified water as well as the drinking water that feed Tabuk Pharmaceuticals to monitor and evaluate their quality. The results will be presented in the form of illustrated tables graphically.

### III.1. Raw water result :

#### III.1-1 The physicochemical results obtained from raw water:

for a start we had to characterise our inflow raw water ,indeed the tests that we have applied on our two raw water's samples allowed us to have the following results :

**Table III.1: The physicochemical results obtained from raw water**

Points	Pe 001	Pe 058	Spécification
Aspect	Conform	Conform	Clear, colorless and tasteless liquid
pH	7.10	7.08	[6.5-9]
Conductivity	601	602	≤2800 µs/cm
Temperature	21	21	[15-30]°C
Evaporation residue	560	540	[100-2000]mg/l

- For chlorine disinfection to be effective, the pH should preferably be below 8.0 , the conductivity was somehow high which mean that's our water has a significant mineralization.
- comparing to our different standards in addition to The mass of the residue obtained which is between 100 and 2000 mg / l , we remark that the raw water In our station complies the intervals mentioned in the table above , which mean that it is ready to get to the next step of our water treatment process .

#### III.1.2. The microbiological results obtained from raw water:

Our microbiological tests allowed us to have the following results

**Table III.2: The microbiological results obtained from raw water**

Points	Pe 001	Pe 058	Spécification
DGAT	5	5	<500UFC/100ml
Total coliforms	ABS	ABS	Absence /100ml
Pseudomonas aeruginosa	ABS	ABS	Absence /100ml

**ABS : Absence**

- DGAT quantity is less than 500 UFC and almost absent which is preferable , the absence of both of total coliforms and pseudomonas aeruginosa give a green card for our water to get to the next process

### III.2.Soft water results:

#### III.2. 1. The physicochemical results obtained from the softened water:

**Table III.3: Soft water physico-chemical results**

Points	Pe 115	Pe 116	Pe 146	Pe 147	Pe 139	Pe 140	Pe 269	Pe 224	Spécification
aspect	C	C	C	C	C	C	C	C	Clear, colorless and tasteless liquid
PH	7.20	7.12	7.15	7.08	7.00	7.13	7.13	7.13	[6.5-9]
Conductivity	601	601	590	590	590	587	587	22	≤2800  µs /cm
Temperature	20	19	19	18	18	18	17	17	[15-20]°C

#### C : conform

- The electrical conductivity values of all our pre-treatment water samples are Between 587 µS / Cm and 601 µS / Cm. These values give water a significant mineralization. We can therefore conclude that the soften water withdrawn has electrical properties that comply with standards.
- For sample Pe 224 which represent the RO first floor outflow water the conductivity had been reduced to 22 µS / Cm which approved our reverse osmosis membrane process efficiency and permeability .
- pH varies between (7-7.20) in temperature (17-20c°) which is effective for a perfect chlorine disinfection.

#### III.2.2.The microbiological results obtained from the softened water:

**Table III.4: The microbiological results obtained from the softened water**

Points	Pe 115	Pe 116	Pe 146	Pe 147	Pe 139	Pe 140	Pe 269	Pe 224	Specification
DGAT	5	5	5	5	4	0	0	0	<500UFC/100ml
Total coliforms	ABS	ABS	ABS	ABS	ABS	ABS	ABS	ABS	Absence /100ml
Pseudomonas aeruginosa	ABS	ABS	ABS	ABS	ABS	ABS	ABS	ABS	Absence /100ml

**ABS : Absence**

- The absence of each of total coliforms and pseudomonas aeruginosa in our soft water complies our standards .
- DGAT varies between 0 UFC/500ml and 5UFC/500ml wich mean that our water is not contaminated

### III.3.Purified water

#### III.3.1. The physicochemical results obtained from the purified water:

**Table III.5: The physicochemical results obtained from the purified water**

Points	Pe 254	Pe 309	Pe 325	Pe 401	Pe 402	Pe 407	Pe 409	Pe 410	Spécification
Aspect	C	C	C	C	C	C	C	C	Clear, colorless and tasteless liquid
Nitrate	C	C	C	C	C	C	C	C	≤ 0.2ppm
oxidizable substances	C	C	C	C	C	C	C	C	The solution remains slightly colored in pink <500ppb
Heavy metals	C	C	C	C	C	C	C	C	≤ 0.1ppm
Conductivity	2.01	2.1	2.02	2.4	2.4	2.3	2.3	2.4	≤4.3μs / cm at T = 20° C and the conductivity obtained ≤ to the conductivity calculated at T ≠ 20 ° C

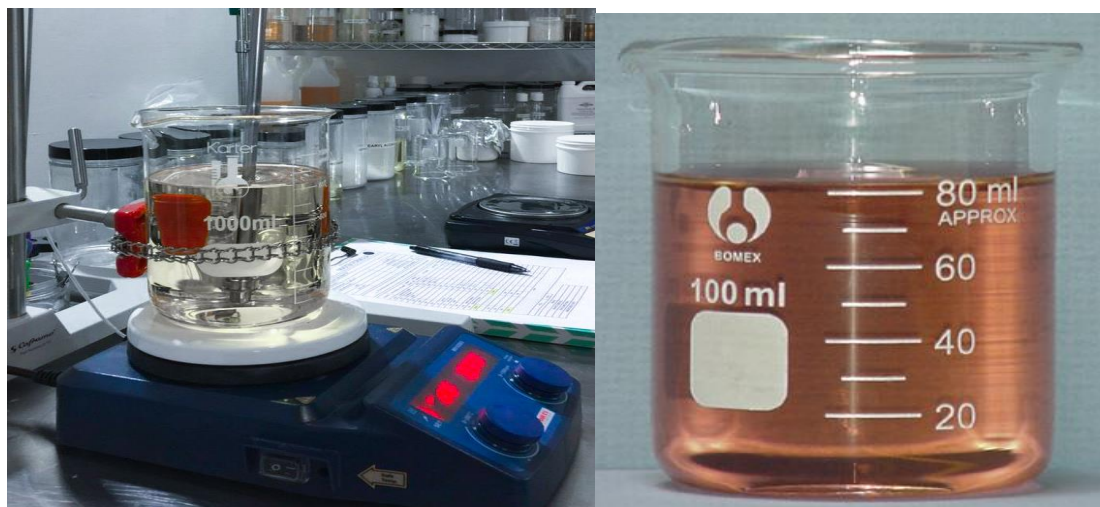
**C: conform**

#### III.3.1.1.conductivity :

- Electrical conductivity is an indicator of the degree of global water mineralization. It depends on the concentration of the ions and the temperature.
- The results obtained during our study show that almost all conductivity values presented by the different sampling points are lower than the maximum recommended value (≤4.3μs / cm at 20 ° C) by the pharmacopoeia European, which indicates the purity of our water.
- Comparing to our first inflow raw water ,and potable water aswell , remarkably the conductivity has reduced from an average of **601 μs/cm to 22 μs/cm** going out of the RO first floor so it can come out of the hole RO process **2.2 μs/cm** wich complies to our references .

### III.3.1.2 Heavy metals (Lead):

A brown coloration is observed for the blank solution which is more intense by compared to the other two solutions, this indicates the conformity of the water.



**Figure III.1:** Heavy metals brown color compared to our sample

### III.3.1.3. Oxidizable substances:

The light pink color observable in the following figure, means that the test of oxidizable substances conforms to the European Pharmacopoeia. The color is obtained for the entire number of sampling, this signifies the good work of the purification station which ensures the almost total absence of oxidizable substances.



**Figure III.2:** Oxidizable substances pink color compared to our sample

In principle,  $\text{MnO}_4^-$ , oxidizes oxidizable substances and gives colorless  $\text{Mn}^{2+}$  at the end of the reaction, but the presence of the pink color explains the non-transformation from  $\text{MnO}_4^-$  to  $\text{Mn}^{2+}$  consequently, the absence of oxidizable substances.

### III.3.1.4. Nitrates:



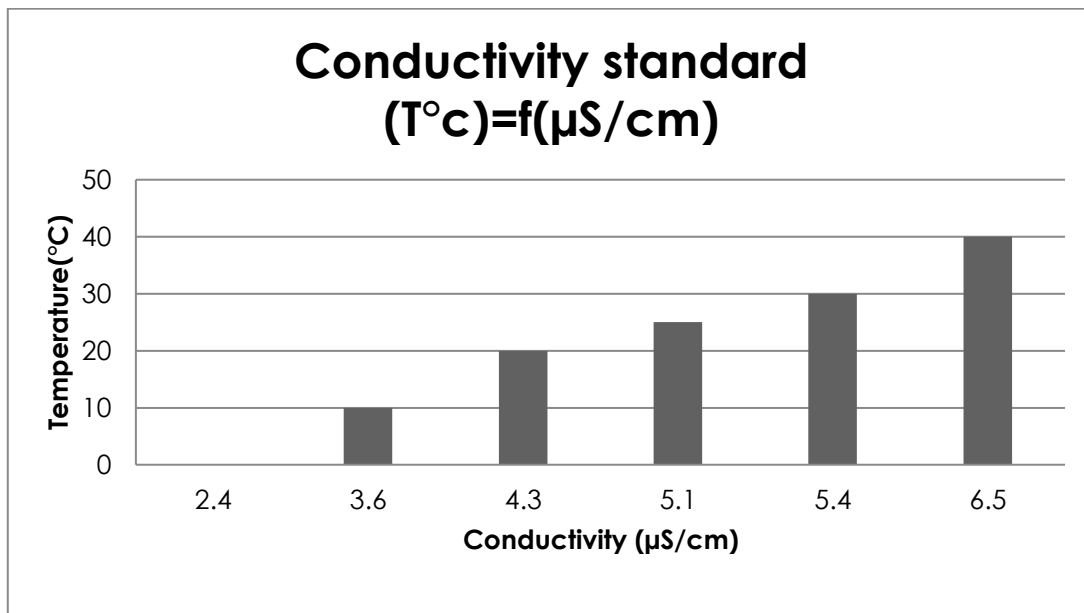
The blue coloration observed in the control solution is more intense compared to the two other solutions (sampling of the two valves), then the test is compliant.



**Figure III.3:** blue nitrate color compared to our sample

#### III.3.1.4. Conductivity standard :

- Electrical conductivity is an indicator of the degree of global water mineralization. It depends on the concentration of the ions and the temperature.
- if a conductivity measurement is made at 25° C, it can simply be reported as the specific conductance. If a measurement is made at a different temperature and corrected to 25° C, then the temperature coefficient must be considered. The specific conductance temperature coefficient can range depending on the measured temperature and ionic composition of the water.



**Figure III.4:** conductivity standard depending on temperature

### III.3.2. the microbiological results obtained from the purified water :

**Table III.6:** the microbiological results obtained from the purified water

Points	Pe 254	Pe 309	Pe 325	Pe 401	Pe 402	Pe 408	Pe 409	Pe 410	Specification
DGAT	0	0	0	0	0	0	0	0	<100UFC/100 ml
Total coliforms	ABS	ABS	Presence	ABS	ABS	Presence	ABS	ABS	Absence /100ml
Pseudomonas aeruginosa	ABS	ABS	Presence	ABS	ABS	Presence	ABS	ABS	Absence /100ml

#### ABS: Absence

- In purified water, germs can come from distribution pipes or from non-rigorous monitoring (maintenance and regeneration) of the various machines for producing purified water, which leads to the formation of biofilms (adhesion of microorganisms).
- Presence of each of total coliforms and pseudomonas aeruginosa in both of Pe325 (Upstream water UV sterilizer (loop return)) and Pe408 (coating room 1) recommend a post treatment using a chemical disinfection .

### III.4. Desinfection by ozone :

- After having a microbiological contamination in two distribution points including Pe325 and Pe 408 we immediately passed to a posttreatment using ozone , we first stop the UV and the open the ozone generator for few hours to let the O<sub>3</sub> destruct .
- Due to its high oxidation potential, ozone oxidizes the cell components of the bacterial cell wall. It is a consequence of the penetration of the cell walls. Once ozone enters cells, it oxidizes all essential components.

#### III.4.1.The physicochemical results obtained after ozone disinfection of purified water:

- The next step is then to pass the different purified water sample's check points by our previous tests, that gave us the following results :

**Table III.7: The physicochemical results obtained after ozone disinfection of purified water**

Points	Pe 254	Pe 309	Pe 325	Pe 401	Pe 402	Pe 407	Pe 409	Pe 410	Spécification
aspect	C	C	C	C	C	C	C	C	Clear, colorless and tasteless liquid
Nitrate	C	C	C	C	C	C	C	C	≤ 0.2ppm
oxidizable substances	C	C	C	C	C	C	C	C	The solution remains slightly colored in pink <500ppb
Heavy metals	C	C	C	C	C	C	C	C	≤ 0.1ppm
Conductivity	2.06	2.06	2.02	2.3	2.2	2.4	2.3	2.3	≤4.3µs / cm at T = 20° C and the conductivity obtained ≤ to the conductivity calculated at T ≠ 20 ° C

C: Conform

- Passing by the same previous tests for our purified and distribution water samples we obtain compliable results when it comes to each of conductivity that varies between (2.06 µs / cm and 2.4 µs / cm) , and nearly the absence of both of heavy metals and nitrate in addition of a low oxidizable substances below <500ppb .

#### III.4.2..microbiological results obtained after ozone disinfection of purified water:

**Table III.8: microbiological results obtained after ozone disinfection of purified water**

Points	Pe 254	Pe 309	Pe 325	Pe 401	Pe 402	Pe 407	Pe 408	Pe 409	Pe 410	Specification
DGAT	0	0	0	0	0	0	0	0	0	<100UFC/100ml
Total coliforms	Abs	Abs	Abs	Abs	Abs	Abs	Abs	Abs	Abs	Absence /100ml
Pseudomonas aeruginosa	Abs	Abs	Abs	Abs	Abs	Abs	Abs	Abs	Abs	Absence /100ml

**ABS: Absence**

- After going by an efficient ozone disinfection , the absence of total coliforms and pseudomonas aeruginosa has obviously appeared according to results mentioned in the table above in additions of a comply full DGAT quantity .
- As a conclusion our purified water has been safely produced and distributed to all the industry water point .

## **Conclusion :**

-The main objective of our study was therefore to follow the evolution of the physicochemical and microbiological quality of the drinking water which supplies the pharmaceutical Tabuk and of the water purified that goes into production..

-The results of the analyzes on samples taken weekly showed the compliance of drinking and purified water from industry:

- An average of 600  $\mu\text{S} / \text{cm}$  for the electrical conductivity of softened water and less than 2.4  $\mu\text{S} / \text{cm}$  for purified water in a temperatures tha varies between (20-30c° ) which does not exceed the standards.

- Almost a 100% absence of oxidizable substances, nitrates and lead in purified water.

- In total, the results of the analyzes complied with the standards of the European Pharmacopoeia for purified water. Our study therefore revealed that the purified water was in good physicochemical quality and microbiological after mandatory disinfection by ozone, inter-oxidation; a variable fraction of the DOC is then transformed into CODB, which then promotes the biological degradation of this organic matter, and thus reduces the problems of bacterial reviviscence .

-To this end, it is recommended to:

- Replace the pre-chlorination step with pre-ozonation

- Adopt a good mastery of the analysis of incoming water in order to properly determine an adequate treatment.

- Exploitation of existing water treatment products .

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