Chapter

# Treatment of Wastewater by Nanofiltration

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## Abstract

In recent years, some countries have implemented regulations governing aqueous discharges. With a view to sustainable development, manufacturers are looking for wastewater treatment technologies to control their discharges. Nanofiltration seems particularly suitable for the separation characteristics that it allows with regard to the size of the target molecules. Pollution by rare earths and heavy metals affects groundwater and surface water. This changed the quality of the water and made it unsafe to use. Water pollution is a big problem, given the diversity of sources and characteristics of polluting species, the main ones being industrial, urban and agricultural discharges, generated by human activity. The great difficulty being that heavy metals are not biodegradable and tend to accumulate in living organisms (fish, mollusks, vegetables, etc.) consumed by humans. For these concerns, environmental laws have become more severe. For this, the treatment of aqueous effluents has become important. It can be concluded that separation and purification chemistry is an area of topical research. The discharges coming from the industry contain heavy metals (chromium, copper, zinc, nickel, iron, cobalt, cadmium, lead, ...) which are harmful for the human health, the fauna and flora. It is necessary to be well controlled. This chapter presents a study of nanofiltration for industrial wastewater treatment.

**Keywords:** nanofiltration, industrial wastewater treatment, pollution, environment

## 1. Introduction

Increased demands by limited water resources have triggered in worldwide for innovative water practices. Many processes are used to purify water contaminated with rare earths and heavy metals, such as solvent extraction, precipitation, ion exchange, absorption and liquid–liquid extraction. The underground water resources can be polluted by wastewaters [1].

There is a lot of proceed of separation which can be applied to the treatment of discharges, among these techniques we mention: chemical precipitation, coagulation–flocculation, flotation, ion exchange, and the membrane processes as supported membrane liquid [2–4], nanofiltration (NF) [5], ultrafiltration (UF) [6, 7], reverse osmosis (RO) [8] and microfiltration recently show by [9].

Nanofiltration has a lot of applications in industry [10]. Among membrane technologies, nanofiltration is the best opportunity to solve environmental problems, such as: desalination recently shown by [11], wastewater and ground water treatment [12, 13], and heavy metals elimination [14].

These methods are valid but have many drawbacks which require the use of organic solvents which are harmful to the environment and time consuming.

With technological development in industries and the differentiation in quantity and type of waste, the development of new, more efficient techniques has become necessary. Among these techniques, membrane processes.

Currently, membrane extraction is the most widely used method for treating industrial waste and purifying wastewater. It is a very active field in separation sciences and many companies are currently producing and developing new membrane extraction techniques.

Nanofiltration is ecological technique; this advantage is particularly linked to this operation without the need for organic solvents and also because the extraction requires little time to perform.

Currently, membrane techniques play a very important role in water purification and open up new possibilities for beneficial use for water sources. They were difficult to use before for technical and economic reasons.

Membrane methods are commonly used in the purification of water and the purification of wastewater [15, 16].

Using these techniques, every five years the creation of water purification plants is multiplied by ten [17].

The "naturalists" are the first who approached the selective transport of substances through membranes [18].

In order to explain the transfer of the solvent, Abbé Nollet supposes the presence of forces between the parts of the membrane, through the membranes the passage of substances is influenced by their molecular mass [19].

The use of membrane processes in industry began in the 1960s. There is a wide variety of membranes due to the existence of several fields of application for membrane processes.

The membranes are used in the separation and concentration of ionic species or molecules in the solution and/or to separate microorganisms (bacteria, viruses, etc.) or suspended matter.

Perm selective membranes are used in membrane processes [19, 20].

The selective displacement of certain components through a membrane, under the application of a force, is the concept of membrane separation [20–22].

All membrane processes use tangential filtration to limit the accumulation of material [20, 23].

The solution of the particles which do not pass through the membrane is called "retentat", and "permeat", the solution of the components which pass through the membrane [24].

Various advantages, among which:

- Reliability and quality of the final product [20].
- Low energy consumption [20].
- The ability to treat water containing several metals.
- Good selectivity [25].
- It works without adding chemicals [26], and the ease and use of industrial integration [20].
- It works without the need for secondary chemicals [27–29].

For these reasons, the water treatment sectors use these technologies [30].

## 2. Membrane processes by nanofiltration

The nanofiltration technique is located between ultrafiltration (UF) and reverse osmosis (OI), it is used to separate components of size close to the nanometer order.

This type of membrane does not retain non-ionized organic compounds with a molar mass of less than 200 g/mol and monovalent salts. On the other hand, non-ionized organic compounds with a molar mass greater than 250 g/mol and multivalent ionized salts (calcium, magnesium, aluminum, sulfates, etc.) are retained [31].

Nanofiltration has lower electrical energy consumption, because it requires lower pressures than reverse osmosis [32].

The nanofiltration technique (**Figure 1**) is based on two separation mechanisms, separation under the effect of electrical repulsion for charged species, and separation under the effect of size for uncharged solutes [33].

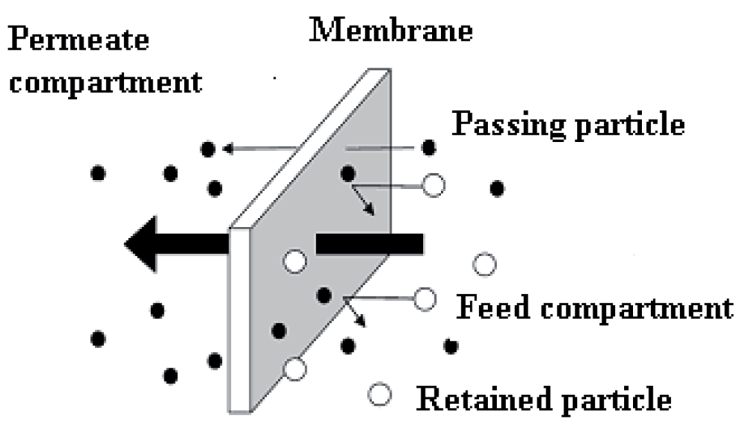


**Figure 1.**
*Selective membrane [15].*

## 3. Classification of membranes

The comparison between membranes of the same type requires the evaluation of the performance of these membranes under real conditions.

Using a few criteria such as: selectivity, permeability and lifetime, one can consider that one membrane is more adequate than another to be separated [27]. The information provided by the membrane manufacturers is insufficient to compare the membranes. To understand these membranes, the acquisition of this information is essential in order to master the transfer mechanisms and improve their performance.

Two categories of parameters are often sought:

- performance-related parameters:
    - Permeability.
    - Retention.

The concentration factor.

- Parameters related to morphology:
    - The membrane thickness.

- ○ Pore sizes.
- ○ Distribution of pores.
- ○ The charge density.
- ○ Hydrophobicity.
- ○ The adsorption and absorption capacities.

## 4. Principle of membrane separation

### 4.1 Tangential filtration

Under the action of a pump, the fluid circulates parallel to the membrane from a reservoir (**Figure 2**) [29].

### 4.2 Front filtration

The flow of the feed solution is perpendicular to the membrane (**Figure 3**) [17, 28].
**Classification according to the separation mechanism**
The processes responsible for membrane filtration are:

- Sieving.
- The friction on the walls of the pores.
- The diffusion in the membrane material and in the pores of the membranes.
- Repulsive or attractive surface forces.
- Electrostatic repulsion.

According to their separation mechanisms, the membranes are classified:

- Non-porous membranes: these membranes can be considered as dense media, the species are diffused in the volumes located between the molecular chains of the material.
- Porous membranes: the effects of friction, sieving and surface forces are dominant in these membranes.

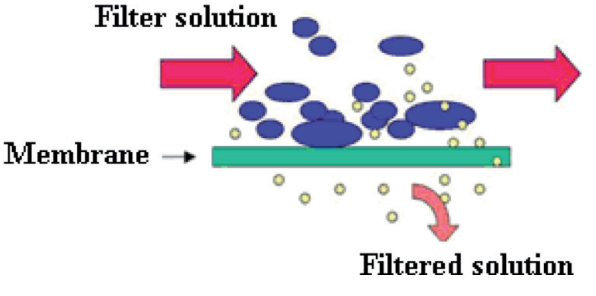


**Figure 2.**
*Tangential filtration [34].*

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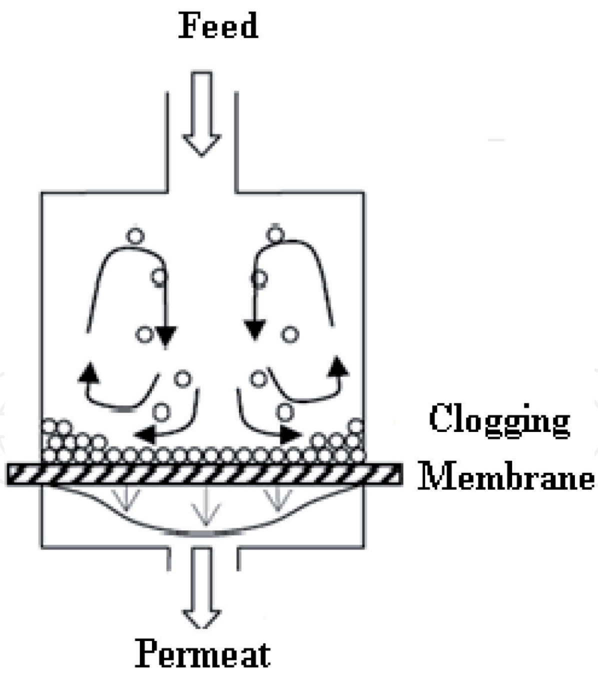


**Figure 3.**
*Front filtration [35].*

- Ion-exchange membranes: consist of dense gels, it is a special type of non-porous membrane, they can be anion-exchange (with a positive charge) or cation-exchange (with a negative charge).

**Classification according to the geometry of the membranes**

According to the geometry under which the membranes are manufactured, they are classified as:

- Flates modules (**Figure 4**):
- Spiral membranes (**Figure 5**):
- Tubular modules (**Figure 6**): are membranes with an internal diameter greater than 3 mm.
- Hollow fiber modules (**Figure 7**):

**Classification according to chemical nature**

The membranes are synthesized from inorganic materials and organic polymers. There are also mixed membranes made from inorganic materials and polymers [38].

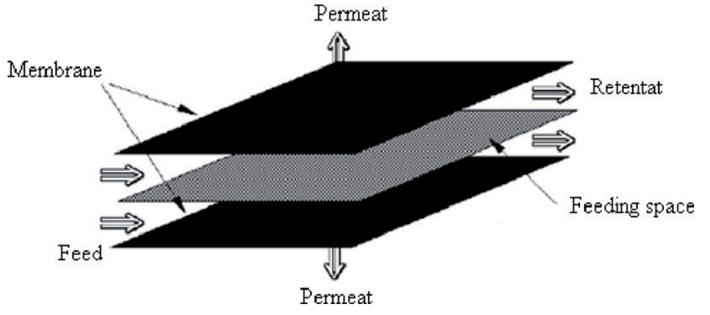


**Figure 4.**
*Flat module [36].*

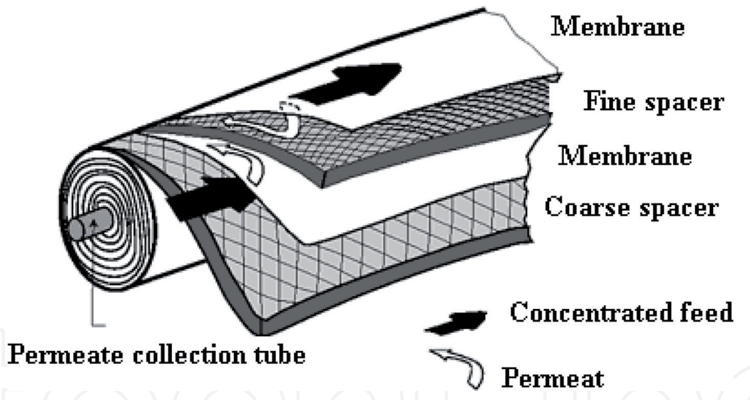


**Figure 5.**
*Spiral module [37].*

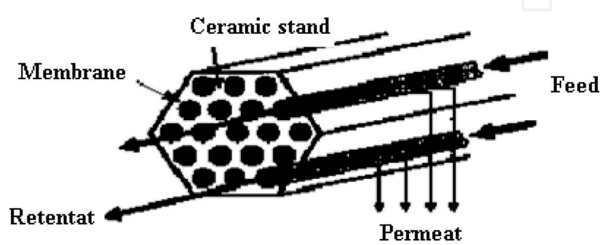


**Figure 6.**
*Tubular module [36].*

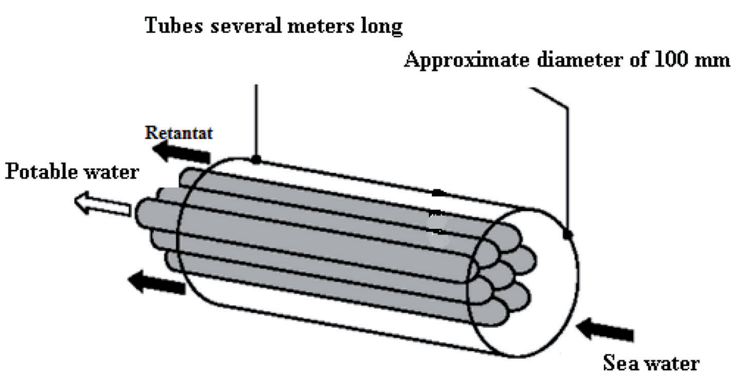


**Figure 7.**
*Hollow fiber module [36].*

The majority of usable membranes are manufactured either from inorganic materials or from polymers.

The main polymers used for the manufacture of membranes are:

- Cellulose derivatives: are inexpensive, considered more hydrophilic, this type of polymer has a low tendency to adsorb.
- Polyamides: these polymers are very sensitive to chlorine, polyamides having chemical and thermal properties superior to those of cellulose derivatives.
- Polysulfone and polyethersulfone, these polymers have good mechanical, and thermal.

Inorganic membranes are made of ceramic (zirconium, titanium, aluminum oxides). These membranes are expensive, have a mechanical, chemical and thermal stability superior to polymeric membranes, they are brittle [38].

**Classification according to morphology**

According to their structure, the membranes can be classified:

- Membranes with symmetrical structure (isotropic membranes): have the same structure over their entire thickness.

- Membranes with asymmetric structure (anisotropic membranes): The structure of the membrane is different from one layer to another.

There are two subtypes of asymmetric membranes:

- Membranes made from the same material.

- Composite membranes: are mainly composed of two layers:

Skin: a layer with a very low thickness, this layer is responsible for the selectivity of the membrane.

Support layer: it is a layer with a much greater permeability than that of the skin layer, which is thicker and which retains the mechanical resistance.

In order to increase the permeability of the membrane, the majority of commercial membranes are manufactured with an asymmetrical structure [39].

### 4.3 Size characteristics of membranes

**Physical characteristics**

**Permeate retention and flow**

The performance of filtration is characterized by the observed retention and permeates flow. Retention is expressed as follows [40]:

$$Y(\%) = \left(1 - \frac{C_p}{C_0}\right).100 \tag{1}$$

$C_p$: Concentration in permeat and $C_0$: Concentration in feed.

This parameter characterizes the effectiveness of the treatment. It quantifies the retention of a target compound.

- **Permeability of a membrane (Lp)**
    - It represents the volume or mass flow crossing the membrane per unit of membrane area.
    - Permeability is a function of temperature and pressure.
    - According to Darcy's law, the flow of solvent (Jv) is proportional to the transmembrane pressure.
    - The hydraulic permeability (Lp) is given by the following equation, this equation is valid for all membranes.

The permeate flow (Jv) characterizes the productivity and is expressed as follows:

$$J_v = L_p\left(\Delta P - \sigma \Delta \pi\right) \tag{2}$$

The permeability of the membrane is deduced from the slope to the right of the permeate flow Jv as a function of the transmembrane pressure ΔP.

- **Hydraulic resistance (Rm)**

This is the resistance of the membrane to the flow of the fluid to be filtered; the resistance is related to the permeability by Eq. (3).

$$R_m = \frac{1}{\mu L_p} \quad (3)$$

μ is the viscosity of the permeate (water).

The resistance can be calculated from the permeate flow through the membrane and the transmembrane pressure.

- **Cut-off threshold**

The SC of a membrane is the molecular weight above which the membrane retains at least 90% of the molecules. It is expressed in g/mol or in Dalton [40].

The cutoff is used in the characterization of membranes, but from a scientific point of view it is not rigorous, as it depends on the operating conditions and the characteristics of the solute.

- **Lifespan**

This is a characteristic of the membrane, because beyond which the membrane will no longer be effective [41].

- **Conversion rate**

The conversion rate is the flow rate fraction of the liquid passing through the membrane [41]:

μ is the viscosity of the permeate.

The resistance can be calculated from the permeate flow through the membrane and the transmembrane pressure.

$$Y = \frac{Q_p}{Q_f} \quad (4)$$

**Chemical characteristics**

Depending on the chemical nature of the membrane, there are interactions between the membrane and the solutes to be filtered, in particular at the level of clogging [20].

- Hydrophobicity and hydrophilicity:

Hydrophilicity depends on the ionized or polar groups of the polymers used, by nature organic membranes are hydrophobic [20].

Due to their hydrophilic properties, regenerated cellulosic membranes are widely used in ultrafiltration [42, 43].

- Surface electrical charge:

Organic membranes have two functional groups one is amine (basic) and the other is carboxylic (acid) having positive or negative charges respectively.

Due to the partial hydrolysis of the amide functions, NF membranes are negatively charged, inorganic membranes have amphoteric surfaces.

### 4.4 Phenomenon limiting the transfer of matter

**Clogging**

The clogging phenomenon (**Figure 8**) occurs when dissolved or suspended matter is deposited on the outer surface or inside the pores [43]. Due to clogging, the performance of the membrane decreases [45].

Upon contact between the membrane and the particles of the solution, a reversible or irreversible modification of the membrane was caused [40]. After this modification, the membrane needs to be replaced or cleaned [45].

The clogging mechanisms are linked to:

- The adsorption of particles through the pores (partial blockage).
- Complete blockage of the membrane pores.
- Internal blockage of the membrane pores.
- Formation of a deposit of particles on the membrane surface (forming a cake) [40, 45].

In view of the nature of the sealant, we can distinguish the type of plugging:

- Clogging by precipitation of soluble salts (scale)
- Bio-clogging (biofilm formation)
- Clogging by colloidal substances [43].

The clogging reduces the flow of water at constant pressure, so a cleaning of the clogged membrane must be applied in order to recover its initial characteristics.

Clogging is a function of:

- Type of membrane,
- The nature and concentration of solutes and solvents,

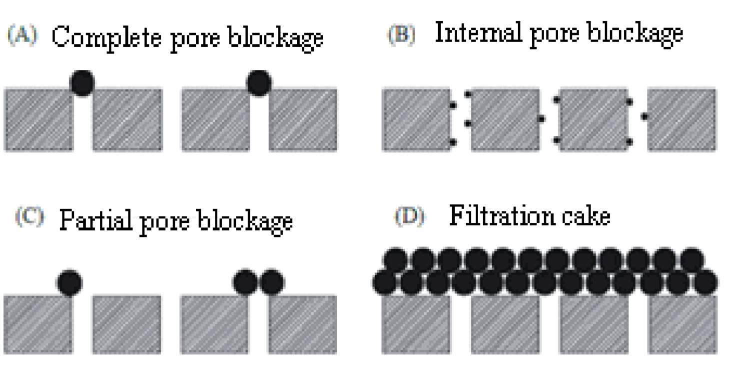


**Figure 8.**
*Clogging mechanisms [44].*

- The characteristics of the membrane surface,
- Membrane materials. The hydrodynamics of the module.
- Distribution of pore size.

**Polarization of concentration**

The accumulation of solutes retained on the surface of the membrane leads to the phenomenon of concentration polarization [43], which causes an increase in the concentration of solute at this surface [41].

When the driving force is interrupted and the permeate flow quenched, the concentration polarization is invisible [45].

Assuming that the true concentration is unidirectional along an axis perpendicular to the membrane, the total permeation flux is the sum of the retro-diffusive flux and the convective flux [44].

**Cleaning**

To remove the clogging elements, cleaning with chemicals (acids and bases) and/or physical (or mechanical) cleaning and/or by using a specific cleaning solution containing appropriate detergents [46].

**Physical (mechanical) cleaning**

Physical cleaning is used to remove and loosen the material accumulated on the membrane.

Backwashing is the most common procedure: part of the permeate passes through the membrane in the opposite direction of flow.

This requires a membrane that must physically support the inverted pressure gradient, other practices use pulsed flows.

Air/gas scrubbing: used to inject air or gas into the membrane [47].

If the membrane is not fully restored, chemical cleaning is necessary.

**Chemical cleaning**

Chemical cleaning contains two parts of acid and basic cleaning as well as rinsing.

Acid washing is used to dissolve the scale layers of metal oxides and thus prevent the formation of insoluble hydroxides that are difficult to remove [47].

The purpose of alkaline washing is to hydrolyze silica, inorganic colloids and organic and biological matter.

Surfactants are also used as cleaning solutions, they are used to:

1. Move the clogging elements in the surface.
2. Emulsified oils.
3. The solubilization of hydrophobic elements [47].

For example for polyether sulfonated membranes Tween 20 is used for cleaning [7].

## 5. Material

**Pilot equipment**

Nanofiltration experiments were carried out with the separation unit illustrated in **Figure 9**. On an industrial scale, this unit is multiplied according to needs.

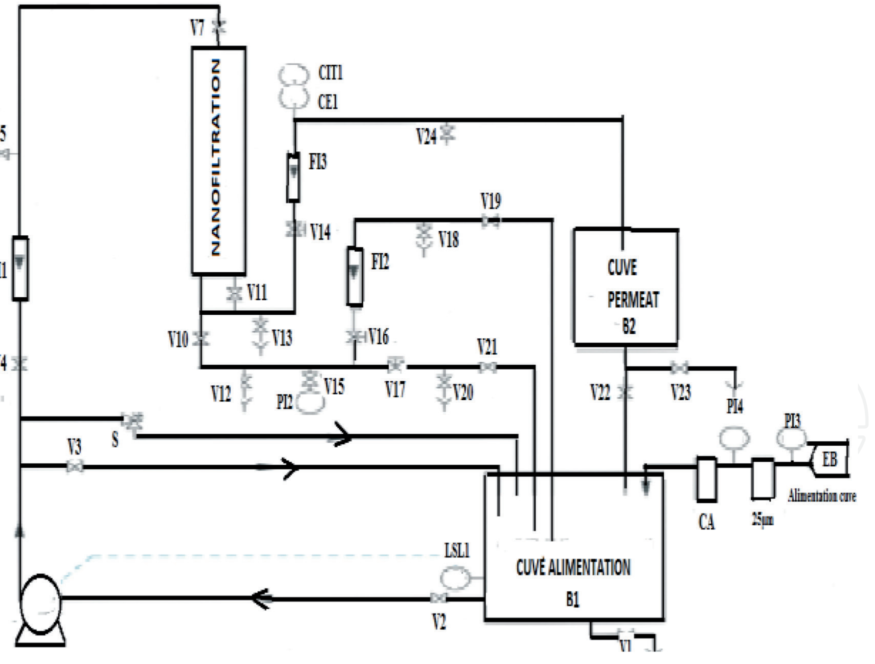


**Figure 9.**
*Experimental setup [48].*

CA is the cartridge filter with activated carbon and 25 μm of wound cartridge filter. S is the safety valve (14 bars). B1 is the feed tank (100 L). B2 is the permeate tank (20 L). C2 is the nanofiltration membrane. FI1 is the upstream flow meter (100–1000 l/h). FI2 is the downstream flow meter of retentive. FI3 is the downstream flow meter of permeate. PI1 & PI2 are the manometers at upstream and downstream of module (0–16 bars). PI3 & PI4 are the monitoring manometers of filters state (0–2.5 bars). LSL1 is the low level sensor (pump safety). CE1 is the sensor of permeate conductivity measuring. Y is the emptying, CIT1 to the electrical display cabinet. V1–5, 7, 10, 11, 14–16, 19 & 22 are the pressure regulation valves for nanofiltration process. P is the multistage centrifugal pump (high pressure).

### 5.1 Materials

Analyzes of water are carried out on turbidimeter, pH meter, conductimeter, atomic absorption spectrophotometer, UV–visible, apparatus for measuring DBO5 and DCO, and ICP-MS.

## 6. Applications

- With membrane techniques, gas separation is more economical.
- Membrane processes are better for the environment because they work without chemicals.
- Are easy and effective compared to other techniques.
- A low investment cost for their systems.
- The membranes do not require frequent regeneration.
- The determination of various compounds [49].

During nanofiltration, the electromigration, diffusion and convection are the major transport mechanisms. The atoms ions can pass through the channels between the molecular groups in the molecular structure of the membrane. The selectivity of nanofiltration membrane was defined by nature and size of these passages, ionic size, shape, pore density, pore diameter, and membrane surface charge [50, 51].

The suitability of a thin film polyamide nanofiltration composite membrane (SNTE NF270–2540) to extract heavy metal ions (Zn (II), Cr (III), Cu (II), Fe (III), Cd (II), Pb (II), Co (II) & Ni (II)) from industrial wastewater was examined [52]. The operating conditions including feed pH value, feed metal concentration and pressure were optimized. The retention of iron at pH = 5.3 and nickel at pH = 7.2 for pressure from 6.0 to 13.5 bars was quantitative (100%). For lead at pH = 4.4 for pressure from 6.0 to 10 bars, the retention was quantitative (100%).

The influence of divalent cations on the rejection of trivalent cations was examined [52].

The membrane was characterized by using the ions transport model and the consumption energy was calculated. The effect of ionic concentration and the diffusion fluxes were studied [52].

## 7. Conclusion

The water purification by nanofiltration membrane techniques is presented, as well as the different classes of membranes. The influence of physico-chemical parameters is discussed.

The effect of pH on the adsorption capacity depends on the surface charge or zeta potential of the sorbent at different pH and presence of different electrolyte.

Purification by membrane filtration is highly dependent on the species present in water and properties of the membrane.

Membrane nanofiltration in combination with others techniques showed a high rejection.

NF membrane can be used in any types of water and it can treat organic or inorganic effluents.

For a given module, we observe that there is interest in working at a high conversion rate to limit heating of the solution (energy consumption). However, the solution to be treated is concentrated very quickly. Scaling of the cartridge will occur very quickly. We must find a compromise for the three parameters $C_0$, Y and $Q_p$.

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