



Title

Toxicological risk of urban effluents discharged into the North-Western Coast in Lake Tanganyika (Democratic Republic of Congo) and their treatment by pressure-driven membrane filtration

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DEDICATION

To my wife;

To my children;

To my mother;

To my brothers and sisters;

To my late father;

To all those who are dear to me;

I dedicate this thesis.

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List of variables

A	Membrane surface (m ²)
C	Concentration of ion (molL ⁻¹)
CE	Electrical conductivity (μS/cm)
C _p	Permeate concentration (ppm, ppb)
C _r	Retentate concentration (ppm, ppb)
F	Polluting flux (kg/d)
J _w	Permeate flux (L. m ⁻² .h ⁻¹)
L _p	Permeability (L m ⁻² h ⁻¹ bar ⁻¹)
MP	Driving pressure (Pa)
P _{max}	set-point of maximum pressure
Q _c	Concentrate flow rate
Q _p	Permeate flow rate
Q _i	Flow rate (L/s)
T	Temperature (°C, K)
Δπ	Osmotic pressure differential (bar)
π	Osmotic pressure (bar)

List of acronyms

BA	Bioaccumulation
BC	Bioconcentration
BM	Biomagnification
BOD ₅	Biological oxygen demand
BTC	Belgian Technical Cooperation
CA	Cellulose Acetate
CNTs	Carbon nanotubes
CO	Organic compounds
COD	Chemical oxygen demand
CP	Concentration polarization
DO	Dissolved oxygen
DRC	Democratic Republic of Congo
EC	Electrical conductivity
EDX	Energy-dispersive X-ray
EPA	Environmental Protection Agency
FT-IR	Fourier transform infrared spectroscopy
GC	Gas chromatograph
GO	Graphene oxide
GRH	General Reference Hospital
HMR	Heavy metal removal (%)
HMMs	Heavy metals and metalloids
HMs	Heavy metals
HRC	Hydrobiology research center

HW	Households
PCB	Polychlorinated biphenyls,
PBB	Polybrominated biphenyls
PCDD	Polychlorinated dibenzodioxins
PCDF	Polychlorinated dibenzofurans
ICP-MS	Inductively Coupled Plasma Mass Spectrometer
ICP-OES	Inductively Coupled Plasma Optical Emission Spectroscopy
MF	Microfiltration
MMMs	Mixed matrix materials
MOFs	Metal-organic frameworks
MPD	m-phenylenediamine
Na ₂ EDTA	Disodium salt ethylene diamine tetraacetic acid
NF	Nanofiltration
TFN	Thin-film nanocomposite
NPs	Nanoparticles
PA	Polyamide
PAN	Polyacrylonitrile
PAN-d	Dry support
PAN-w	Wet support
PAs	Aromatic polyamides
PC	Penitentiary center
PP	Piston pump
PSF	Polysulfone
R	Rejection
R%	Rejection pourcentage
RO	Reverse osmosis
RW	Real water
SDS	Sodiumdodecyl sulfate
SEC	Specific energy consumption
SEM	Scanning electron microscopic
SRB	Sulfate-reducing bacteria
SWRO	Seawater reverse osmosis
TDS	Total dissolved solids
TFC	Thin film composite
R%	Rejection pourcentage
TMC	Trimesoylchloride
UF	Ultrafiltration
VOCs	Volatile organic compounds
WCA	Water contact angle
WHO	World health organization
WW	Wastewater
XRD	X-Ray Diffraction

Index	
Abstract	10
Chapter 1. General introduction	12
1.1. Generalities over water pollution.....	13
1.2. Pollution of the northwestern slope of Lake Tanganyika in the Democratic Republic of Congo.....	16
1.3. Treatment method of Wastewater	20
1.4. Membrane separation methods.....	22
1.4.1. Physical-chemical mechanisms of solute retention.....	25
1.4.1.1. Solute retention in NF	25
1.4.1.2. Solute retention in RO	25
1.5. Reverse osmosis as technology for removal of heavy metals	26
1.5.2. Performance of RO membranes.....	30
1.5.3. Energy consumption in RO	37
1.5.4. Main disadvantages of PA-TFC RO membranes	37
1.5.4.1. Clogging	37
1.5.4.2. Concentration polarization.....	38
1.5.5. Conclusions	40
1.6. Thesis motivation.....	41
1.7. Thesis objective.....	43
1.8. Thesis outline	44
1.9. Originality of the research.....	44
Chapter 2. Evaluation of the pollution of the northwestern slope of Lake Tanganyika in the Democratic Republic of Congo	45
2.2. Methodology.....	46
2.2.1. Presentation of the study site.....	46
2.2.2. Sampling and analytical methods.....	46
2.3. Results and Discussion	50
2.3.1. Physical-chemical characteristics of wastewater and tributaries.....	50
2.3.2. Flow rate.....	52
2.3.3. Emitters of pollutants	52
2.3.4. Characteristics of the organic load of liquid effluents.....	53
2.3.5. Determination of the hydrogram and pollutogram	55
2.3.5.1 Determination of the hydrogram	57
2.3.5.2 Pollutogram.....	58
2.3.6. Biotic parameters.....	58
2.4. Conclusions	60

Chapter 3. Determination of the Toxicological Risk of Urban Waste from the City of Uvira Dumped into the North-Western Coast in Lake Tanganyika (Democratic Republic of Congo) .	61
3.1. Introduction	62
3.2. Materials and Methods	62
3.2.1. Presentation of the Study Environment	62
3.2.2. Fish sampling	63
3.2.3. Chemical and toxicological analysis	64
3.3. Results and Discussion	66
3.3.1. Inorganic Micropollutants	66
3.3.2. Organic Micropollutants.....	72
3.4. Conclusions	74
Chapter 4. Evaluation of commercial reverse osmosis and nanofiltration membranes for the removal of heavy metals from surface water in the Democratic Republic of Congo	75
4.1. Introduction	76
4.2. Methodology.....	78
4.2.1. Sampling site of real samples	78
4.2.2. Synthetic samples	80
4.2.3. Membrane cross-flow system setup	80
4.2.4 Commercial membranes.....	81
4.2.5 Experimental procedure.....	81
4.3. Results and Discussion	83
4.3.1. Effect of the Metals Feed Concentration on the Membrane Performance	83
4.3.2. Ion removal in real waters	85
4.3.3. Comparison of real and synthetic water retention	86
4.3.4. Transmembrane flux.....	89
4.4. Conclusions	91
Chapter 5. Removal of toxic metal ions from surface water using biopolymer chitosan/ZIF-8 mixed-matrix membranes.....	92
5.1. Introduction	93
5.2. Methodology.....	95
5.2.1. Materials	95
5.2.1.1. <i>Characteristic of PAN supports.....</i>	<i>95</i>
5.2.1.2. <i>Chemicals</i>	<i>95</i>
5.2.1.2. <i>Synthesis of ZIF-8 particles.....</i>	<i>96</i>
5.2.1.3. <i>Membrane preparation.....</i>	<i>96</i>
5.2.1.4. <i>Membrane performance evaluation.....</i>	<i>98</i>
5.3. Results and discussion.....	99

5.3.1. ZIF-8 characterization	99
5.3.2. Membranes characterization.....	100
5.3.4. Membrane performance.....	108
5.3.4.1. <i>Effect of ZIF-8 on ion retention</i>	109
5.3.4.2. <i>Effect of ZIF-8 on permeate flux</i>	111
5.4. Conclusions	114
Chapter 6. General conclusions and future perspectives.....	115
6.1. General conclusions.....	115
6.2. Futures perspectives.....	117
6.2.1. Techno-economic assessment and determination of real viability in.....	117
Uvira/DRC	117
6.2.1.1. <i>Geo-security situation of the city of Uvira</i>	117
6.2.1.2. <i>Socio-technical aspects</i>	117
6.2.1.3. <i>Description of the process</i>	117
6.2.1.4. <i>Managing reverse osmosis retentate</i>	120
6.2.1.5. <i>Chemical cleaning of membranes</i>	120
6.2.1.6. <i>Management of end-of-life of reverse osmosis membranes</i>	121
6.2.1.7. <i>Economic evaluation</i>	121
6.2.1.8. <i>Financing</i>	122
6.2.1.9. <i>Surface modifications for other applications</i>	122
6.2.1.10. <i>Access to sanitation in Africa and DRC</i>	123
APPENDIX 1. CHAPTER 2.....	124
APPENDIX 2. CHAPTER 3.....	128
APPENDIX 3. CHAPTER 4.....	131
APPENDIX 4. CHAPTER 5.....	138
List of publications	180
Research papers under preparation	180
Communication in international conferences	181

Abstract

Water scarcity is one of the problems caused by global industrialization. In developing countries, population increase, rural exodus and environmental degradation are major threats to humanity. Inadequate wastewater treatment is a major concern, as it contributes greatly to the destruction of water resources when discharged into the environment. In the Democratic Republic of Congo, sanitation is not sufficiently developed. The remarkable lack of qualified human resources for the analysis and monitoring of environmental quality, technical and/or financial means does not allow for the implementation of coherent programs adapted to the realities and challenges facing the country. Much of the work carried out by researchers in the Democratic Republic of Congo is limited to the evaluation and quantification of pollution, concluding that population should not consume fish or water from polluted sites. However, there is an expression commonly used in low-income countries, which says "a hungry stomach has no ears". With this in mind, this study focuses on the application of emerging technology based on reverse osmosis and nanofiltration to treat urban effluent discharged into the north-western coast of Lake Tanganyika (Democratic Republic of Congo).

Two main global objectives were considered in this study, namely: i) the determination of pollution and toxicological risk of pollutants on the northwestern slope of Lake Tanganyika, and ii) the development of a treatment method of domestic and industrial wastewater by commercial membranes, including the preparation of a low-cost membrane to remove pollutants.

The determination of pollution was based on chemical analysis of organic and inorganic micropollutants, and the toxicity test by bio-testing was experimented in aquariums at the laboratory of the Hydrobiology Research Center in Uvira. The results show that the wastewater is polluted by highly toxic inorganic and organic compounds. The sources of pollution came from the reference hospital of Uvira, the penitential center of Uvira, the households, the artisanal food-processing industries, the degradation of non-recycled household waste and the physical alteration of the sedimentary rocks of the Precambrian period that form the substrates. The real waters sampled in Uvira were filtered on 200 μm filter paper to retain large particles that can affect membrane fouling and clogging. These real waters had not undergone any other chemical pre-treatment. After the physical pretreatment, they were filtered on commercial reverse osmosis membranes of polyamide type and NF90, NF270 to remove inorganic micropollutants. The synthetic waters were prepared in the laboratory with the specific ions under study at known concentrations. Those waters were filtered through commercial reverse

osmosis membranes, as well as nanofiltration membranes (NF90 and NF270) to remove metal ions. The experimentation of commercial membranes allowed to evaluate its performance. Next, a membrane containing chitosan and the metallic organic framework ZIF-8 (i.e., CS/ZIF-8 mixed matrix membrane) was prepared at the laboratory and characterized by XRD, SEM, FTIR, angle contact, and tested in the reverse osmotic unit using the synthetic waters at different concentrations.

The results obtained show that the retention of ions by the polyamide reverse osmosis membranes is excellent, although the permeate flux is low. The retention with nanofiltration membranes depends on the feed concentration and ionic interactions at the membrane surface. Regarding the self-made CS/ZIF-8 mixed matrix membrane, it shows better retention performance for Cr^{3+} , Pb^{2+} , Cd^{2+} and Ni^{2+} ions in a concentrated feed solution and high-water permeability at low operating pressure. In addition, industrial scaling-up of the CS/ZIF-8 membrane is more attractive, as the manufacturing cost is low and the permeate flux high. The economic feasibility analysis shows that the investment cost is high, but energy consumption is low.

Keywords: Reverse osmosis, Nanofiltration, thin film composite, thin film nanocomposite, MOF, inorganic and organic micropollutants, Lethal Dose, Lake Tanganyika,

Chapter 1. General introduction

Reverse osmosis (RO) is considered as a highly efficient process for heavy metal removal from wastewater, with high degree of purification and simple scale-up (Van der Bruggen et al., 2003; Shannon et al., 2008; Greenlee et al., 2009; Elimelech et al., 2011; Chuang et al., 2018; Luis, 2018; Friess et al., 2021). Polymeric membranes such as those made of cellulose acetate (CA), and Thin Film Composite (TFC) membranes is typically designed on a porous PSF support on which an ultra-layer, a thin barrier layer deposited on the top surface and dominate the industry (Lee et al, 2012; Chenhao et al., 2021, Zhang et al., 2022), but the TFC membrane exhibits improved solute rejection capability and reduced permeation capability to water. Conventional metal oxide ceramic membranes, membranes prepared from graphene oxide (GO), carbon nanotubes (CNTs) (Farahbakhsh et al., 2019), and mixed matrix materials (MMMs) (Ding et al., 2017; Han et al., 2017) have attracted considerable attention due to their properties, such as tunable pore structure, excellent chemical, mechanical and thermal tolerance, good salt rejection and/or high-water permeability (Cohen et al., (2015).

1.1. Generalities over water pollution

Water is the most important raw material of our planet, for humans, animals, plants and micro-organisms. Practically all the vital phenomena of the biosphere are linked to the availability of water. Water is not only the space of life, the vector of energy or the means of transport, but also the essential element of any type of production. Freshwater is one of the ecosystems most seriously threatened by the impact of anthropogenic effects over the past century (Sala et al., 2000, Dudgeon et al., 2006).

The demand for water for human activities will increase the stress on this resource. In addition, natural factors such as drought or geological constraints, population growth, the development of industrial and agricultural activities, as well as the diversification of production by-products or post-consumer waste that affect the supply and distribution of drinking water, a degradation of ecosystems and subsequently the health of the population. Quantifying and analyzing the quantity and quality of water supplies and finding a way to manage this resource to make it sustainable is paramount (Wuana et al., 2011; Salvo et al., 2014; Gupta et al., 2015; Somasundaran, P., 2004; Zheng et al., 2015; Hayat et al., 2017; Pintor et al., 2016; Chen et al., 2019; Mo et al., 2018; Gupta et al., 2013; Ipek et al., 2005; Azimi et al., 2017; Lasat, M. 1999; Herrera et al., 2009; Kumar et al., 2015; Mohod et al., 2013; Zhao et al., 2018).

The hydrosphere is among the ecosystems most threatened by the impact of anthropogenic effects over the past century (WHO, 2017; Osvaldo et al., 2000; Dudgeon et al., 2006). In most developing countries, most urban waterways are used for urban and industrial waste disposal. This results in degradation of water quality, alteration of settlements, and, most often, a decrease in biodiversity (Even et al., 2008).

Living things can be exposed to a large number of xenobiotics, synthetic or natural (toxins), from microorganisms, plants or animals, which can disrupt the normal metabolic processes of a person or animal and thus are harmful to the body. These can be toxic products, such as environmental pollutants (metals, pesticides and plant protection products, polychlorinated hydrocarbons and non-medicinal narcotics (Coumoul et al., 2017). More recently, micropollutants are linked to diffuse emissions of particles and domestic practices (Briand et al., 2018). Among the most commonly encountered organic micropollutants are 1,4-dichlorobenzene, 1,1-dichloroethane, toluene, xylenes, ethylenediaminetetraacetic acid EDTA and acetone (Beal et al., 2006). In addition, there are also pharmaceuticals and stimulants such as caffeine, salicylic acid and diclofenac, which are difficult to treat in intensive purification processes (Fu et al., 2011; Maxime et al., 2020). In Europe, the presence of pharmaceuticals in

aquatic environments is explained by their limited removal in the WWTPs (Fekadu et al., 2018; Naccari et al., 2015; Di et al., 2015; Salvo et al., 2014, Lo et al., 2013; Gupta et al., 2012a; Iannazzo et al., 2017).

Heavy metals pollution has become a cause for concern due to their non-biodegradable nature, persistence, toxicity, biomagnification and bioaccumulation in the food chain (Feng et al. 2021; Peng et al. 2021; Khan et al., 2008; Noori et al., 2022; Ihsanullah et al., 2016; Fu et al., 2011; Zhao et al., 2016). Studies by Hua et al (2012) report that industrial wastewater containing copper, cadmium, zinc, lead, mercury and manganese, etc. is often deliberately or accidentally discharged into local water resources.

Certain metals such as Zn, Cu, Mn, Ni and Co are micronutrients necessary for plant growth, while Cd, Pb and Hg have an unknown biological function and are toxic (Gaur et al., 2004). Other metals, such as Cu and Mo, have a vital function but are harmful to organisms at high concentrations (Herrera-Estrella et al., 2009).

Wang et al., (2009) categorized heavy metals of concern into three categories: toxic metals (such as Hg, Cr, Pb, Zn, Cu, Ni, Cd, As, Co, Sn, etc.), precious metals (such as Pd, Pt, Ag, Au, Ru etc.) and radionuclides (such as U, Th, Ra, Am, etc.). The most popular heavy metals are Pb, Zn, Hg, Ni, Cd, Cu, Cr, and As. Although these heavy metals can be detected in traces; however, they are still hazardous. Table 1.1 summarizes some Heavy metals, focusing on their major sources, health effects, and the permitted quantity in drinking water. The aforementioned metals and others such as Ag, Fe, Mn, Mo, B, Ca, Sb, Co, etc. are commonly available in wastewater and need to be removed (Qasem et al., 2021).

Table 1.1. Typical Heavy metals present in wastewater (OMS) (Demiral et al., 2021)

Common Heavy metals	Main sources (Demiral et al., 2021 ; Kumar et al., 2015 ; WHO, 2014).	Main organ and system affected (Duan et al., 2020 ; Marciniak et al., 2019 ; Owolude et al., 2016 ; Ngah et al., 2008 ; Upadhyay et al., 2021)	Permitted amounts (μg) (Demiral et al., 2021).
Pb	Batteries à base de plomb, soudure, alliages, pigments de gaine de câble, inhibiteurs de rouille, munitions, émaux et stabilisateurs plastiques.	Bones, liver, kidneys, brain, lungs, spleen, immunological system, hematological system, cardiovascular system, and reproductive system.	10
As	Electronics and glass production.	Skin, lungs, brain, kidneys, metabolic system, cardiovascular system, immunological system, and endocrine.	10
Cu	Corroded plumbing systems, electronic and cables industry.	Liver, brain, kidneys, cornea, gastrointestinal system, lungs, immunological system, and hematological system.	2000
Zn	Brass coating, rubber products, some cosmetics, and aerosol deodorants.	Stomach cramps, skin irritations, vomiting, nausea, and anemia, and convulsions.	3000
Cr	Steel and pulp mills and tanneries	Skin, lungs, kidneys, liver, brain, pancreas, testes, gastrointestinal system, and reproductive system	50
Cd	Batteries, paints, steel industry, plastic industries, metal refineries, and corroded galvanized pipes.	Bones, liver, kidneys, lungs, testes, brain, immunological system, and cardiovascular system.	3
Hg	Electrolytic production of chlorine and caustic soda, runoff from landfills and agriculture, electrical appliances, Industrial and control instruments, laboratory apparatus, and refineries.	Brain, lungs, kidneys, liver, immunological system, cardiovascular system, endocrine, and reproductive system.	6
Sb	Antimony is a rare element, but it does occur naturally. Though it is present more commonly as a contaminant in food than in water, its use in associated with lead plumbing is a concern. Lead, being a naturally soft native metal, has sometimes been hardened and strengthened using antimony. Where antimony occurs with lead, there may be concerns of either being present at elevated concentrations in the water	It is considered toxic even at low concentrations. According to the NCBI (National Center for Biotechnology Information), antimony poisoning can cause gastrointestinal distress, nausea and even ulcers. Antimony is also implicated in a wide range of respiratory and cardiovascular complications, and certain compounds containing antimony may even cause reproductive harm or be considered carcinogenic.	6
Ni	Stainless steel and nickel alloy production.	Lung, kidney, gastrointestinal distress, pulmonary fibrosis, and skin.	70

1.2. Pollution of the northwestern slope of Lake Tanganyika in the Democratic Republic of Congo

In Africa, population growth, rural exodus and the degradation of the environment constitute great threats which weigh on humanity. Indeed, the growing population is increasingly concentrated in the cities. All of the foregoing can dangerously affect aquatic ecosystems and pose a long-term danger to human existence. Lake Tanganyika is of international importance and is a vital resource shared by Burundi, the Democratic Republic of Congo, Tanzania and Zambia. However, the governments of the four riparian countries and the international community are increasingly recognizing that threats to the lake's biological richness and the sustainable use of its resources are rapidly increasing (Cohen, 1991; West, 2001). This obvious and substantial increase in human activities around the lake, coupled with increasing industrialization and urbanization, poses environmental problems as the city of Uvira and its factories (the port and its oil depot, the Kiliba sugar refinery) and its general reference hospital (GRH) lack wastewater treatment facilities, all of which end up in Lake Tanganyika directly or indirectly via its tributaries (Bailey et al., 2001).

This situation affects freshwater bodies, such as the case of Lake Tanganyika, given its lower altitude compared to that of the agglomerations erected on its banks. Unfortunately, it constitutes the main receiving environment for the pollution generated in Bujumbura (on the Burundian side), Uvira, Kalemie, and Moba (on the DRC side), Kigoma and Kipili (on the Tanzanian side), and Mpulungu (on the Zambian side). These agglomerations are also home to a diversity of chemical and agric-food industries and activities (markets for example) which generate enormous amounts of wastes in both liquid and solid forms (Savyimana, 2015) which, if not treated will constitute a potential danger to the lake.

Since the beginning of the 21st century, the environment (atmospheric, terrestrial, and aquatic) has been under increasing pressure from industrial and human activities, the effects of which have been rapidly felt. Any substance of anthropogenic or natural origin released into the environment and having adverse biological effects is a pollutant (Moriarty, 1990) or a xenobiotic. The latter term refers to any substance that does not occur naturally and is characterized by high toxicity at low concentrations (Ramade, 2000). Contaminants released into the environment are found rapidly in aquatic environments, particularly estuarine and coastal areas, where they can have short- and long-term effects (Burton, 1992).

The liquid effluent drainage system was built by the Belgian Technical Cooperation (BTC), and takes its source just at the foot of Mount Mitumba, which is threatened by anarchic construction that is very advanced because of the high population density at the foot of the mountain, which

has harmful effects on the environment. The demographic growth of the area endangers the lives of the inhabitants because the passage of the collector exposes the area to multiple natural disasters. This collector receives a large part of the solid and liquid household waste of the inhabitants of the Kakungwe district (Shishi Avenue), of the GRH of Uvira and of the wastewater of the Penitentiary Center (PC) which contribute to the pollution of the environment and to the imbalance of the ecosystems of the lake, following the lack of appropriate hygiene and sanitation practices.

The large collector built by BTC is connected to four outlets, namely: rainwater from the roof gutters coming from the main entrance of Shishi Avenue which flows into the collector from upstream, the Mitumba chain covering the Belle Institute, the GRH outlet and finally the wastewater outlet of the central prison which is sufficiently loaded. The wastewater and the water from the collectors are channeled into Lake Tanganyika, which constitutes pollution that can contribute to the imbalance of the ecosystems. It should be noted that the city of Uvira does not have a sewage system, nor a treatment plant for liquid and solid waste. As a result, all waste produced by Heavy metals, whether solid or liquid, is dumped into the drains of several avenues in the city to be transported to the lake (Lumami et al., 2016).



Photo 1.1: Channel obstruction by solid and liquid waste (Kapepula, 2019)



Photo 1.2: Outlet for collecting solid and hospital WW to lake

Lake Tanganyika fisheries play an important role in food security in Central and Eastern Africa (Kyezer et al., 2020). Lake Tanganyika is home to several endemic species, most of which are not found anywhere else in the world (Fermon, 2007). Fishermen in Lake Tanganyika operate close to shore in small pirogues using "lusenga" (large conical dip nets), standing gill nets, beach seines, hand lines and hooks between 0 and 5 m depth (West, 2001; Mushagalusa et al., 2014; Petit and Shipton, 2012; Keyzer et al., 2020). It is possible that this artisanal fishery is catching fish contaminated with pollutants and intended for sale to the public. This poses a hazard in the food chain through bioaccumulation (BA) and this phenomenon could regenerate

poorly in humans through the phenomenon of biomagnification (BM).



Photo 1.3: a) Handline fishing between 0 and 5 m of depth; b) Fish caught on the lake coast.

The lake has become a gigantic dumping ground for effluent laden with organic and inorganic compounds from various chemical and food processing industries, as well as household waste. Most of the towns on the shores of Lake Tanganyika have no wastewater or solid waste treatment facilities. There is no common sanitation policy in the countries concerned. For this reason, it is of utmost importance to develop a process for treating contaminated water before it is discharged into the lake. Specifically, the presence of heavy metals has been of great concern in the lake. Consequently, all of the household waste, both solid and liquid, is dumped in the gutters of several avenues of the municipalities from where they are carried to Lake Tanganyika (Lumami et al., 2016).

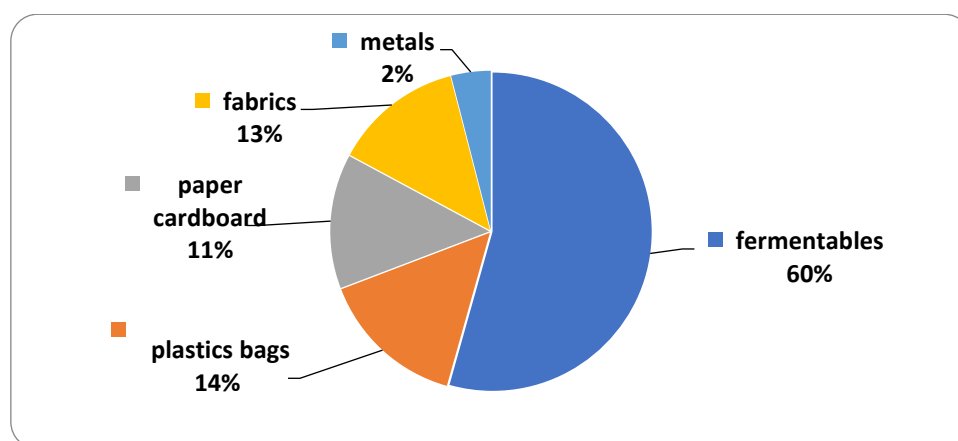


Figure 1.4. Composition of household solid waste in the City of Uvira (Lumami et al. 2016)

This is the case of the large collector built by the Belgian Technical Cooperation, which carries hospital effluents from the Uvira GRH and effluents from the PC of Uvira to Lake Tanganyika. Meanwhile, the population obtains water for domestic use from the shore of Lake Tanganyika, fishing is done near the coast between 0 and 5 meters deep (West, 2001).

Even today, some 2.6 million men, women and especially children die each year from diseases related to water and an unhealthy environment (Barbe et al., 2018). A dramatic observation due to a reality that is too little known: in 2018, one third of humanity still drinks water that is dangerous to health. 2.6 billion people still lack adequate sanitation (Barbe et al., 2018). Generally, wastewater and hospital effluent contain several organic micropollutants, such as hydrocarbons, polychlorinated biphenyls (PCB), polybrominated biphenyls (PBB), polychlorinated dibenzodioxins (PCDD), polychlorinated dibenzofurans (PCDF), detergents (or surfactants), phthalate esters and various drug substances (Lumami et al., 2021; Jin et al., 2019). Recently, numerous VOCs have been identified in wastewater, such as phthalate esters and organochlorine pesticides (Jin et al., 2019; Al-Gharabli et al., 2023), having carcinogenic and genetic toxicity to human beings (Li et al., 2017; Ahmet et al., 2005; Ye et al., 2020). It is therefore possible that the population is infected with water-borne diseases due to the contamination of coastal waters.

In addition, the ingestion of infectious agents in contaminated drinking water influences the transmission of waterborne diseases, while the quantity of water available domestically influences “water-washed” disease transmission, where insufficient water is available to allow adequate hygiene practices (Cairncross et al., 2019; Jeandrom et al., 2019)

The various annual reports of the health zone of Uvira in the DRC have established cases of water-borne diseases in the city from 2014-2019 as shown in Table 1.2. below.

Table 1.2. Annual statistics of water-borne diseases and disseminated by water-related vectors (HGR Uvira)

	2014		2015		2016		2017		2018		2019	
Disease	Case	Death	Case	Death	Case	Death	Case	Death	Case	Death	Case	Death
Cholera	1914	9	1364	7	1434	9	1266	6	1078	9	959	11
Typhoid	15128	46	18994	36	22415	2	18582	8	17896	N.D	N.D	N.D
Diarrhea	1474	14	11500	10	13318	3	14847	7	11783	N.D	N.D	N.D
Malaria	46184	175	59475	64	69216	16	72915	58	50005	N.D	N.D	N.D
Hepatitis	8	0	41	3	15	2	50	2	86	N.D	44	N.D
Hitosomiasis	6	0	6	0	49	1	29	2	114	N.D	N.D	N.D

Annual reports from the Uvira / South Kivu health zone in the DRC

The study area is shown in Figure 1.5 and is crossed by three regular collectors, namely Kibondo, Kakungwe and BTC, which drain rainwater, liquid and solid household effluents, and hospital effluents into Lake Tanganyika, the receiving environment.

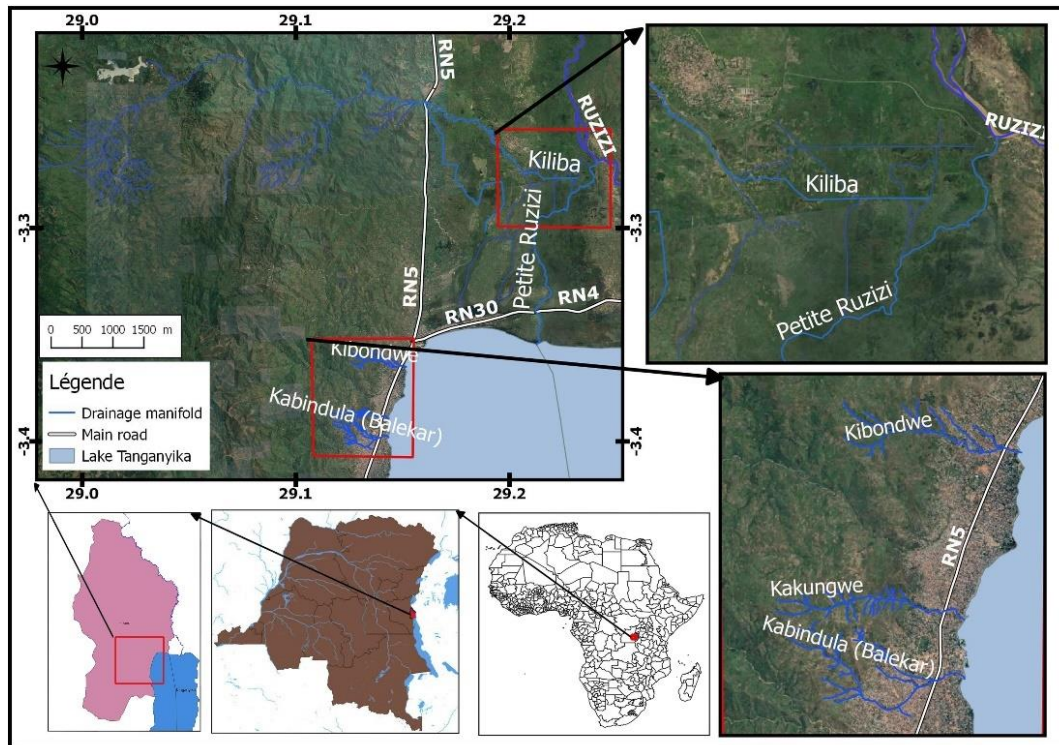


Figure 1.5. Location of the study area

1.3. Treatment method of Wastewater

Several methods can be used to remove heavy metals from wastewater, including biological and physical-chemical methods. Some researchers instead think of other technologies either employing inorganic adsorbents (such as natural minerals, ores, clays and wastes from various industries such as metallurgical solid wastes, such as bauxite red mud, slag, ash, water treatment sludge (alum) and seawater neutralized red mud), or involving organic adsorbents (such as waste organic matter derived from plants or animals) (Rai et al., 1998; Khan et al., 2008; Zvinowanda et al., 2009; Zhou and Haynes, 2010, 2011).

Among the various technologies available for biological Heavy metals removal, the most studied microbial know-how uses of sulfate-reducing bacteria (SRB) that remove Heavy metals production of metal sulfide precipitates on a large scale (Perales- Vela et al., 2006). However, its main drawbacks include: residence time (weeks), then need for continuous feeding substrates and larger bioreactors (White et al., 1997; Perales-Vela et al., 2006). Monteiro et al (2012) justifies in particular the microalgae (living and non-living biomass) do not help to remove contaminant levels. Table 1.3 below shows the advantages and disadvantages of the biological and physical-chemical methods.

Table 1.3. Advantages and disadvantages of biological and membrane processes (Wang et al., 2006 ; Chen et al., 2008 ; Perpetuo et al., 2011 ; Gadd, 1990 ; White et al., 1997; Perales-Vela et al., 2006)

Type of process	Advantages	Disadvantages
Biology	Bioremediation (microorganisms, sulfate-reducing bacteria (SRB))	- Ineffective for highly concentrated ions in solution - long adsorption residence time, - Does not purify the effluent Space consuming, - Accumulation depends on factors such as: i) The characteristics of the metal ion (atomic weight and valence), ii) Environmental conditions (pH, temperature, ionic strength, contact time, biomass concentration), and iii) The nature of the biosorbent, which can determine differences in selectivity and affinity for metal ions; here, the type and species of organisms, growth conditions, physiological state, and cellular age can all affect the mechanism of binding to the Heavy metals, - lack of awareness regarding metal–microbe interactions remain unexploited and at times in indicipherable.
	Phytoremediation (plants)	
Membrane filtration	- Reverse osmosis	- Less cumbersome, - High removal rate and purifies the effluent, - Simple scale-up
		- High capital cost, - Energy consumption

1.4. Membrane separation methods

It has become imperative to seek new technologies for remediation / removal of heavy metals to meet these requirements. It is essential for a remediation technology to be effective, economical/affordable, and consistent; furthermore, it should effectively reduce heavy metals concentrations to environmentally acceptable levels and be applicable to field conditions such as effluents and aquatic organisms (Sheng et al., 2004).

Membrane separation technology is the most effective and widely applied technology for water purification. Generally, the membrane acts as a selective barrier that allows for the passage of certain constituents while inhibiting the passage of other constituents based on different mechanisms depending on the type of membrane (e.g., size exclusion, diffusion). Filtration-based membrane processes have been classified into microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), reverse osmosis (RO), and forward osmosis (FO), in which the first two often use a microporous membrane while the last three use a thin composite membrane with a selective dense layer (Hamid et al., 2019). A description of the membranes used under pressure, with the corresponding pore diameters and the species retained, is shown in Figure 1.6.

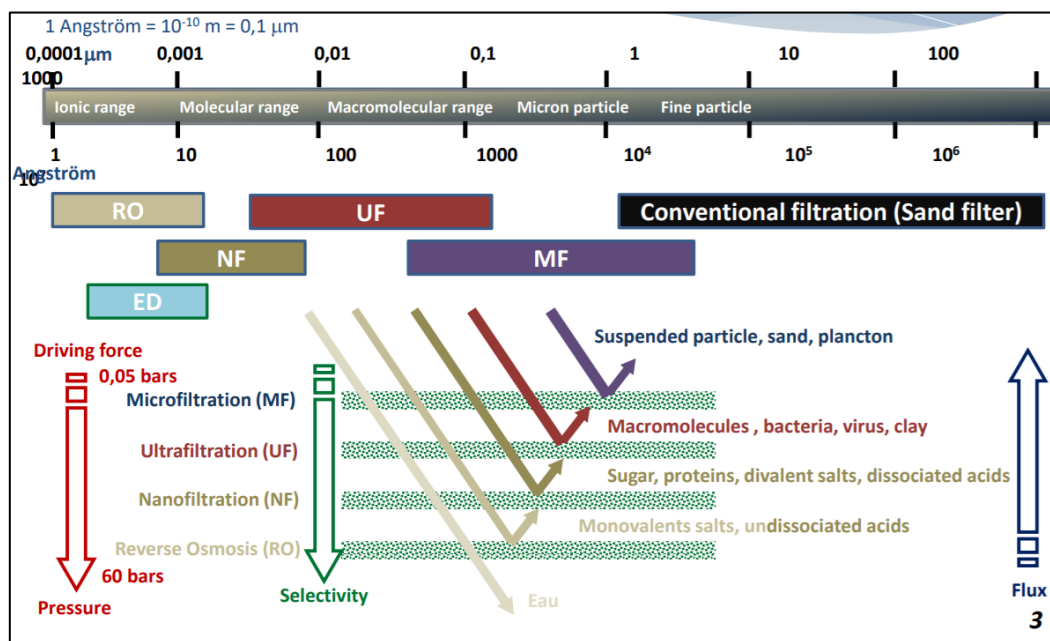


Figure 1.6. Classification of membranes for water purification in terms of pore size and species retained.

MF and UF are often used as pre-treatment before RO or NF (Song et al., 2011; Tomaszewska et al., 2013; Fan et al., 2020; Tian et al., 2021). RO and NF are known as pressure separation technologies for removing dissolved solids. RO and NF are used for brackish waters treatment, wastewater purification, and municipal waters treatment (Figoli et al., 2017; Yang et al., 2019;

Tian et al., 2021; Ganier et al., 2022). Both membranes require pre-treatment to remove the large particles that can clog them (Petrinic et al., 2015). RO and NF are more commonly used in place of conventional treatment technologies (co-agulation, flocculation, sedimentation, clarification, ion exchange and filtration). Several points diverging between RO and NF have been put forward by researchers, some of which are repeated in this section. RO is the process used to remove the smallest particles and water desalination. In RO, water and solute molecules diffuse independently through the dense layer (Paul et al., 2004), while steric hindrance, the Donnan effect and dielectric repulsion are the three factors that influence separation in NF (Labban et al., 2017).

The low cost of operation, maintenance, energy, initial installation and high flux are advantages of NF, but it is not effective in removing salinity from seawater (Hassan et al., 2015; Adda et al., 2021; Armstrong et al., 2022). On the other hand, reverse osmosis requires high pressure, energy consumption and regular cleaning of the membranes (Jeong et al., 2021). Table 1.4 below shows the main characteristics of the different processes.

Tableau 1.4. Main characteristics of the different processes (Morin-Crini et al., 2017; Soria, 2022; Yang et al., 2019; Luis, 2018).

Type	Membrane characteristics	Mass transfer mode	Pore size (nm)	Permeation	Retention	Materials	Permeation rate	Application
MF	Porous	Convection	100-1000	Solvent and dissolved matter	Particles, colloids.	Ceramics and polymeric membranes (PES, PVDF, PE, PAN, PTFE)	150 à 1500 L/ m ² /h	-Bacterial water purification. -Fractionation of milk fat globules. -Protein fractionation. -Treatment of oil/water emulsions.
UF	Porous	Convection	2-100	Solvents, salts and small molecules	Macromolecules, colloids.		50 à 300 L/ m ² /h	-protein concentration. -Clarification and stabilisation of foodstuffs, juices and wines. manufacture of liquid pre-cream. -Effluent treatment (brine).
NF	Porous	Solution/diffusion + convection	1 - 2	Solvents, monovalent ions and small molecules	Small molecules, divalent ions.		50 à 100 L/m ² /h	-Separation and concentration of antibiotics. -Fractionation of amino acids. -Softening of drinking water. -Whey concentration and demineralization.
RO	Dense	Solution/diffusion	<1	Solvent	Monovalent ions.		10 à 60 L/m ² /h	-Concentration of whey, blood and egg white. -De-alcoholization of wines and beers. -Water desalination.

1.4.1. Physical-chemical mechanisms of solute retention

1.4.1.1. Solute retention in NF

NF is the separation process for multivalent salts and non-ionized organic compounds. The removal of uncharged components is mainly the result of differences in diffusion velocities in a non-porous structure, which also depend on molecular size. This solute elimination mechanism is influenced by steric hindrance, which retains the solute with the largest size relative to the membrane pores. If the solute has the same valency as the fixed charge on the membrane surface, the stronger the Donnan effect, and finally dielectric repulsion occurs when the ions are in a medium with a high dielectric constant (Du et al., 2023).

Morin-Crini et al. (2017) reported that when filtering a concentrated solution on a negatively charged membrane, the concentrated ions overwhelm the charge of the membrane material and decrease the electrostatic repulsions between the ions and the membrane surface. The pH of the solution can modify the variations in negative or positive surface charge, which influences the exclusion of ions at the membrane-solution interface.

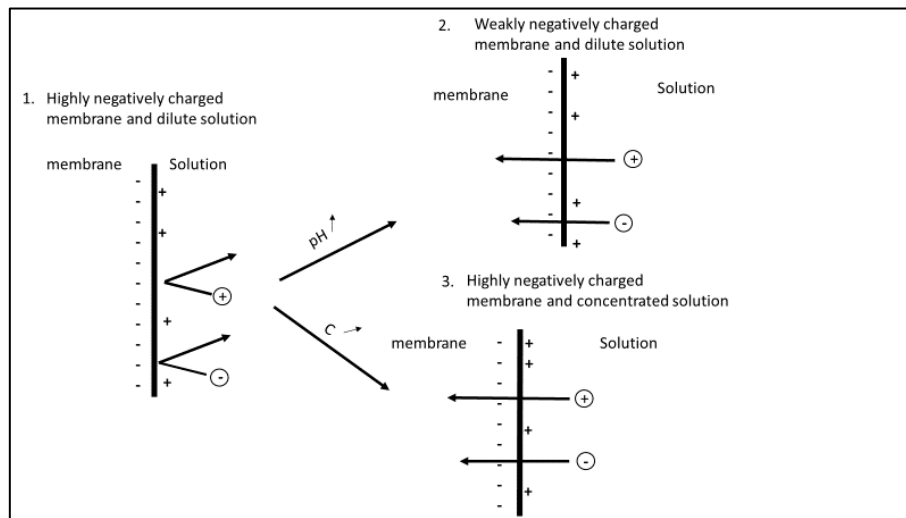


Figure 1.7. Ion retention mechanism under the influence of pH (1 and 2) and concentration (1 and 3) (Morin-Crini et al, 2017)

1.4.1.2. Solute retention in RO

Solute transport in RO has been described by models based on irreversible thermodynamics, Maxwell-Stefan based models, hydrodynamic or pore models and the solution diffusion theory (Luis, 2018). Rejection is determined by the solute permeability coefficient (Luis, 2018):

$$B = \frac{D_i K_i}{\Delta x} \quad (1)$$

Here, K_i is the solute–membrane partition coefficient, D_i is the solute diffusion coefficient in the membrane, and Δx is the membrane thickness. Operation conditions can affect significantly the separation performance of RO. Increasing feed pressure increases permeate flux and salt rejection (Shingidi et al., 2021). Increasing the feed temperature directly affects the viscosity of the water, the salt transmission rate increases which affects solute rejection (Zhao et al., 2023). In addition, it is extremely important to pretreat the feed solution to avoid membrane clogging, which will reduce solute rejection.

Membrane technology was chosen as the subject of this study because of its effectiveness in removing highly charged solutes from solutions, its high capacity to purify effluent, and its cost and low energy consumption compared with conventional processes. However, in the course of this research, RO membranes were exploited more than NF membranes for reasons based on the rejection of monovalent and multivalent ions and all other uncharged organic compounds.

1.5. Reverse osmosis as technology for removal of heavy metals

RO is considered the most efficient process for heavy metal removal from wastewater, with high degree of purification and simple scale-up. Reverse osmosis (RO) membrane process, which can provide high quality water through removing all the impurities in aqueous phase, such as dissolved salts, organic compounds, pharmaceuticals (Foureaux et al., 2019) and inorganic compounds, has become a key technology to alleviate the water crisis (Shannon et al., 2008; Greenlee et al., 2009; Elimelech et al., 2011; Chuang et al., 2018).

Polymeric membranes such as Cellulose Acetate (CA) based membranes and Thin Film Composite (TFC) membranes dominate the industry, but the TFC membrane exhibits improved solute rejection capability and reduced permeation capability to water (Chenhao et al., 2021). However, the cellulose acetate membrane has its own limitations, such as high pH sensitivity and lower tolerance to high temperatures. Conventional metal oxide ceramic membranes, membranes prepared from graphene oxide (GO), carbon nanotubes (CNTs), and mixed matrix materials (MMMs) have attracted considerable attention due to their properties, such as tunable pore structure, excellent chemical, mechanical and thermal tolerance, good salt rejection and/or high-water permeability.

The first works on RO date back to the seventies (Kremen et al., 1977). For example, Kremen and coworkers had demonstrated the possibility to purify wastewater from various metal ions with an integrated process containing RO and precipitation units (Kremen et al., 1977; Algieri et al., 2021). Ujang and Anderson showed the possibility of removing Zn^{2+} and Cu^{2+} using a

low-pressure RO process in the presence of a chelating agent (EDTA) (Ujang et al., 1996). They found that operating pressure, EDTA concentration, and temperature significantly influenced the permeate flux. Some years later, the NF and RO performance for copper (Cu^{2+}), and cadmium (Cd^{2+}) removal has been considered (Qdais et al., 2004). The experiments were performed with polyamide membranes characterized by a spiral wound configuration (Algari et al., 2021). Harharah et al. (2022) showed that permeate flux and Cu (II) removal were directly proportional to operating pressure and feed temperature. The pressure increases from 10 to 40 bar and the removal % increases from 89.98 to 94.21% on the Dow Polyamide TFC BW30XFR membrane. Pretreatment of real industrial wastewater by electrocoagulation, followed by reverse osmosis, revealed 99.89% removal of Cr^{3+} ions (Rasha et al., 2020). Coagulation/flocculation pretreatment of industrial and mining wastewater followed by reverse osmosis on Polyamide membrane (DOW™ FILMTEC™ BW30-440i) removed turbidity, total dissolved solids (TDS) concentrations, antimony, arsenic, nickel, zinc and iron were reduced by 85%, 96%, 95%, 66%, 82%, 48% and 10% respectively in the permeate (Samaei et al., 2020). Figure 1.8 shows an illustration of pressure membrane filtration.

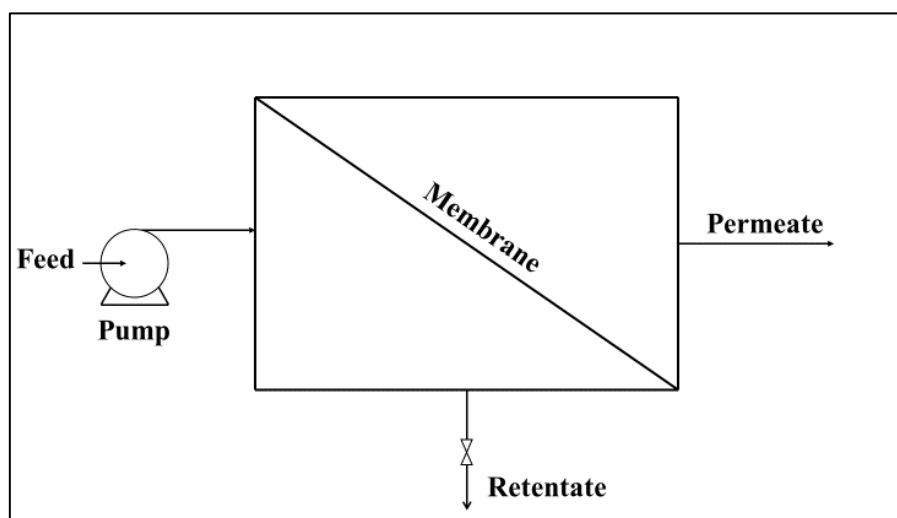


Figure 1.8. Illustration of a pressurized membrane filtration unit.

1.5.1. Types of membranes used RO

RO water treatment contains a semi-permeable membrane. There are two common types of membranes: Cellulose Acetate and Thin film composite. One advantage of the CA membrane is that it is more resistant to chlorine than the TFC membrane. However, TFC membranes are much more efficient and superior in performance overall compared to CA membranes (Li et al., 2010)

Chlorine is the most common industrial oxidizing biocide in water treatment that is used for disinfection of domestic water and for the removal of tastes and odors from water. It is also typically used in RO treatment processes in order to control microorganisms that biofoul and clog the membrane (Ahmadiannamini, 2016). Chlorine alters the physicochemical properties of PA membranes (Jaydevsinh et al., 2017).

The main membrane used in the RO process is the type of TFC, a very thin PA active layer which is formed on a porous substrate is the main characteristic of TFC (Ismail et al., 2015; Xu et al., 2013). The thickness of the thin film is $< 0.2 \mu\text{m}$ with a size of the interstitial voids $\leq 0.5 \text{ nm}$ between the polymer chains (Maruf et al. 2012; Ismail et al., 2015). Typical TFC-RO membranes consist of three layers: a polyester backing fabric, a porous polysulfone (PSF) interlayer, and an ultrathin PA selective layer (Porter, 1989; Song et al., 2019; Song et al., 2020; Cadotte et al., 1981; Gan et al., 2020; Freger, 2005; Zhang et al., 2022). The PA selective layer, formed by the interfacial polymerization reaction of aqueous amine solution and acyl chloride and characterized by a ridge-and-valley structure in the nano-and microscale, is the determinant of the TFC-RO membrane's selectivity and water flux (Porter, 1989; Chuang et al., 2018). Most of the active polymer layers are derived from two basic types of polymers: cellulose CA and aromatic polyamides (PAs).

However, the presence of various inorganic and organic compounds in the water affects fouling, selectivity and flux of the membrane and, a possible way of reducing it is to perform the pretreatment of the feed utilizing MF and/or UF processes. The potentiality of UF-RO process for industrial wastewater treatment has been investigated by Petrinic et al. (2015). The UF process permitted to remove of almost 90% of suspended solids. The RO process, subsequently performed, removed the metal ions and organic/inorganic compounds with efficiency range of 91.3–99.8% (Algieri et al., 2021).

Therefore, TFC membranes can preserve the desired selectivity only at low water permeance ($1\text{--}20 \text{ L m}^{-2} \text{ h}^{-1} \text{ bar}^{-1}$) (Agboola et al., 2021). Nanocomposite membranes, also known as mixed matrix membranes or hybrid membranes, combine the benefits of both organic membranes and inorganic materials and so permit to successfully increase the water permeability and reduced fouling (Jeon et al., 2018; Zheng et al., 2017; Bi et al., 2018).

Currently, these membranes loaded with different inorganic particles are also applied in metal ion removal (Castro-Muñoz et al., 2021), membranes mixed/coated with various emerging (nano)materials, e.g. carbon nanotubes (Farahbakhsh et al., 2019), graphene oxides (Chu et al., 2017; Chu et al., 2017), M-Xenes (Ding et al., 2017; Han et al., 2017), metal-organic frameworks (MOFs) (Basu et al., 2017; Denny et al., 2016), have been developed, which have

attracted the interest of the water industry due to the exceptional characteristics including their selectivity, high tunable porosities and large accessible surfaces (Lu et al., 2014), their improved hydrophilicity and resistance to fouling, high capacity to easily combine particular species/features without changing the topology of the framework (Evans et al. 2014) and their various potential applications (Furukawa et al., 2013).

Recently, the use of MOFs, especially ZIF-8, has attracted the attention of most scientists due to its unique characteristics, such as large surface area, large adsorption capacity, thermochemical stability, hydrophobicity and durable synthesis at room temperature (Abbasi et al., 2020; Li et al., 2021).

Chitosan (CS) has a hydrophobic character is an important natural renewable biomass, one of the cheapest polysaccharides naturally with abundant groups (Agnihotri et al., 2004; LogithKumar et al., 2016; Yang et al., 2016; Liu, 2019), is used as a selective layer on the membrane surface due to its unique properties such as antibacterial activity, biocompatibility, non-toxicity and good film-forming ability (Xiaowei et al., 2018; Kumar et al., 2016; Mohamed et al., 2019).

CS, due to the presence of hydroxyl and amine groups, is widely used to remove Heavy metals ions from aqueous solutions (Rodrigo et al., 2006; Sajjad et al., 2009). However, the poor mechanical properties of CS limit its potential (Xuejie et al., 2020).

This chapter presents the various research works carried out in RO to remove Heavy metals from wastewater. This information is based on the different materials and specific methods of synthesis of RO membranes and the different operating conditions on the treatment of wastewater.

The information available in this chapter will allow researchers to become familiar with available work, then to consider the synthesis of new innovative membranes in RO. It provides researchers with a complete and up-to-date table on the discharge of metals, metalloids and describes the performance of different membranes in terms of permeate flux and methods of removing Heavy metals from wastewater resources.

1.5.2. Performance of RO membranes

Several studies have been conducted on the performance of the RO membrane under different operating conditions in terms of pressure, flow rate (Dimitriou et al., 2017), concentration and temperature (Ruiz-García et al., 2020).

Permeate flux is one of the key factors for evaluating the performance of membranes, which reflects the amount of permeate and product rate and is a factor demonstrating the membranes efficiency (Luis, 2018). The permeate flux is directly proportional to feed pressure and negatively dependent on feed concentration (Ansari et al., 2021).

Many previous studies demonstrate that the enhancement of roughness and leaf-like structures on the PA's top surface is positively related to the water flux (Song et al., 2019; Song et al., 2019; Ma et al., 2018; Yan et al., 2019; Wu et al., 2020; Kamada et al., 2014; Kong et al., 2010; Tan et al., 2018; Al-Jeshi et al., 2006; Ma et al., 2019). Jiang et al. (2018) prepared some ultra-thin films to improve the permeating flux of the film, which was accomplished by spreading out the PA layer from the crumpled to the smooth on the free organic/aqueous interface. The study proves that water permeance increases with the thickness decrease; however, the PA layer's thickness measured by AFM was referred to as its entire height (apparent thickness) (Jiang et al., 2018).

Zhang et al. (2022) studied the influence of the intrinsic thickness (single wall thickness) of the PA layer on the water permeability by adjusting the concentration of reactive monomers. It confirms that the true permeation resistance of water molecules originates from the intrinsic thickness portion of the membrane.

Although TFC membranes have excellent salt removal performance, there is a trade-off between permeability and selectivity. The main advantage of MMMs is to combine the low manufacturing cost, exceptional selectivity and high packing density of polymeric materials with the long-term stabilities, high mechanical strength and regenerative capacity of ceramic materials (Rozaini et al., 2019; Zhu et al., 2019; Forman et al., 2019; Rezakazemi et al., 2019; Ayaz et al., 2019; Yang et al., 2019).

Table 1.5. List of key studies on the removal of Heavy metals from wastewater by using reverse osmosis

Membrane	Element	Initial (ppm)	Conc	Removal efficiency (%)	Condition	Permeates flux	Remarks	Réf
PAs (ES 20)	Cu ²⁺ , Ni ²⁺ , Cr ⁶⁺	10 -500		Synthetic feed: > 99.5, 99.9 and > 99.5,	1 – 5 bars, pH : 7-9	24.64 L/m ² .h	- Synthetic and industrial wastewaters were used. - The membrane effective area was 60 cm². - Feed solution was kept at a temperature of 20 °C.	Ozaki et al., 2002
				Industrial feed: 99.03, 99.37 and 98.75		18.63 L/m ² .h	- pH value was varied between 3 and 9. - Permeate flux decreased with the increase in feed concentration. - Increasing the pH of the feed solution raised the heavy metal ions removal rate.	
Polymer Membranes	Co ²⁺	25		98	2 bars, pH : 4		- Synthetic wastewater was used. - UF UPM-20 membrane and RO OPAM-KN membrane were modified by synthetic cation-exchange resin KU-2-8n and natural clay mineral montmorillonite. - Membrane separation and sorption combination could be used for concentrating or purifying heavy metal ions.	Leon et al., 2005
Thin-film membrane (TFM)	Ni ²⁺	44-169		99.3	pH: 6.5-7.5		- Synthetic wastewater was used. - Addition of 240 ppm concentration of EDTA enhanced zinc and nickel ions removal efficiencies from 98.9% to 99.6% and from 99.3% to 99.7%, respectively. - The operating pH was 4 to 8. - Feed pH and conductivity did not greatly influence removal rates. - Increasing the added EDTA increased the feed conductivity and removal efficiency of metal ions.	Ipek et al., 2005
	Zn ²⁺	64-170		98.9				
TFC	Cu ²⁺ Ni ²⁺	500		99.5	5.03 bar, pH: 7.8		- Synthetic wastewater was used. - Removal rate of copper ion was higher than that of nickel due to the bigger ionic size of copper.	Mohsen-Nia et al., 2007

						- Osmotic and applied pressures affected metal ion removal. - Addition of Na₂EDTA enhance metal ion removal efficiency.	
polyamide	Ca ²⁺ Mg ²⁺ Na ⁺	451 1102 82	97	6-22 bar	29.3 L/m ² . h.	- Industrial wastewater was used.	Lui et al., 2008
TFC PA	As ³⁺ As ⁵⁺	<0.50	20-55 91-99	4 – 6 bars, pH: 4, 5, 6, 7		- Synthetic wastewater was used. - Operating pH ranged from 3 to 9. - The initial As⁵⁺ concentration and pH value ranging from 6 to 9 showed a slight influence on the retention efficiency of the membrane. - RO removed > 90% of As⁵⁺ residual. - Due to the presence of arsenite as a neutral molecule, the RO membrane did not effectively remove As³⁺	Chan et al., 2008
Polyamide	Cu ²⁺	10 - 100	70 - 95	1 – 6 bars, pH: 7-8		- Synthetic wastewater was used. - Addition of surfactant sodiumdodecyl sulfate (SDS) improved the Cu²⁺ removal efficiency to 90% - 99%. - The use of 3D electrode cell electro-reduction technology resulted in a Cu²⁺ mean removal efficiency of 59% - 75%. - Electro-reduction process was slightly affected by the addition of SDS and EDTA. - Low-pressure RO recorded average removal efficiency of copper ion of 85%. - The initial pH of a solution was 6.0	Zhang et al., 2009
	Cu ²⁺ Co ²⁺ Zn ²⁺ Pb ²⁺ As ⁵⁺ Cd ²⁺ Cr ⁶⁺	0.012 0.140 0.162 0.165 0.972 6.360 0.149	100 100 90.74 100 100 99.86 87.92			- Wastewater has been subjected to a series of biological treatment processes to remove suspended solids and reduce COD and BOD₅	Saad et al., 2010
AD-SWRO, AG-BWRO,	B ³⁺	10-11 10.5-10.9	B>91.9 Mg ²⁺ , Ca ²⁺ 99.5	15 – 30 bars, pH: 8.6	94.7 L/m ² . h	- Geothermal water was used. - Four types of RO membranes were used.	Oner et al., 2011

BW-30-BWRO, AK-BWRO	Na ⁺ Ca ²⁺ Mg ²⁺ K ⁺	366 26 3.7 26	Na ⁺ , K ⁺ >99.3		- All the tested membranes showed > 96.5% silica and salt removal efficiency. - Increase in operating pressure showed favorable variations in both quantity and quality of permeate flux. - Operating pressure and type of membrane highly influenced both permeate flux and boron rejection efficiency. Silica rejection was over 96.5%	
TFC PA (TW-30 et XLE, Filmtec™)	B ³⁺	70	>99	5 - 20 bar, pH: 7-11	- Synthetic wastewater was used. - RO removed boron in the presence of chelating polyols containing a 1,2-diol group. - Permeate flux boron removal was affected by feed pH, initial feed concentration, and operating pressure. - Increased in boron removal was recorded with an increase in applied pressure. - Boron concentration in the permeated was found to be below 1 mg/L even at high recovery. - Rapid membrane degradation or membrane fouling was not detected.	Dydo et al., 2012
	Cr ⁶⁺	50 100 500 1000	>91 %	15-35 bar, pH : 1-6	- Synthetic wastewater was used - At the beginning of each experiment, pH of the feed water (1 L) was adjusted to the desired pH level by addition of 0.1 M NaOH or 0.1 M HCl and it was placed in the feed water tank. - The system was operated in the permeate recycle mode	Çimen et al., 2014
TFC PA	Ni ²⁺ Cu ²⁺		98	0.5 MPa	- Addition of EDTA to form complexes with ions - Studies employing solutions of single ions and also mixture of ions in different concentration ranges reached removals of 99%	Pieres et al., 2016

	Ni ²⁺	50	98.5	1 – 4 bars, 10-40°C,	10 to 56 L/m ² .h	- Synthetic wastewater was used	Algureiri et al., 2016
	Cu ²⁺	100	97.5	pH: 2-5.5			
	Pb ²⁺	150-200	96				
Graphene	Cu ²⁺	0.5 – 3 M	100.0 (for OHgraphene), > 98.0 (for B-graphene), and 95 (for NH graphene)	50 – 300 MPa		- Synthetic wastewater was used. - Three types of functionalized (hydroxyl (OH), nitrogen (NH), and boron (B) groups) nanoporous graphene membranes were used as RO membranes. - Increasing membrane pore density increased the removal efficiency while increasing the ion concentration in the feed decreases the permeate flux. - OH, B and NH graphene's reached 100%, > 98% and 95% Cu ²⁺ removal efficiency, respectively	Li et al., 2017
TFC PA	Co ²⁺ , Ni ²⁺ Mg ²⁺	39.4 214.9 2429	98.6 98.1 98.6	41 bars, pH: 1.46	Permeate flux was 2.9 L/m ² .h.bar	- Industrial wastewater was used. - Both RO and NF were used to treat POX effluent generated from the gold ore mining process. - The operating range of pH for RO are 4-11. - Under the acidic conditions, the stability of the RO membrane was satisfactory.	Ricci et al., 2017
TFC PA	B ³⁺	5.688	34 – 48	21-76 bar, pH: 3-11	7.1-32.5 L/m ² . h.	- Industrial wastewater was used. - Triple-stage RO system was adopted. - Greater than 91% water recovery was achieved by a two stage RO system. - TDS rejection factor was in arrange of 91.1% - 97.7%, while that for and COD was over 95%. Also > 94.0% was achieved for Selenide removal. - Rejection factor for ammonia was in a range of 57.4% - 77.3% - Water recovery efficiency was > 91% for a two-stage RO system	Cingolani et al., 2018
PA TFC IP / SA	NaCl	500	94.2	5 bars, 25°C, pH : 3-6,5	42.0 L/m ² .h	- Three identical test cells with an effective filtration area of 23.60 cm ² were parallelly installed in the permeation setup	Yu et al., 2018

SW30	Cr	0.2-1.3 3.2-5.3	97	10-20 bar, 566 – 830 L/m ² h	pH : 3, 25±5°C	- wastewater produced by the aluminum industry - RO membrane is made of polyamide with non-porous active skin layer	Atès et al., 2018
	Ni		99				
	Al		99				
	Pb ²⁺	0.034	< 1 ppb	1.76 MPa,	pH : 6.12	- Pre-treatment of wastewater with biological waste: cereals, rice pods, coffee waste, tea waste, sugar beet pulp, mushrooms,	Thaçi et al., 2019
	Zn ²⁺	0.153	< 0.002 ppb				
	Cd ²⁺	0.025	< 0.1 ppb				
	Co ²⁺	0.018	< 0.2 ppb				
	Mn ²⁺	1.146	0.006 ppb				
	Ni ²⁺	0.004	< 0.5 ppb				
Dow PA TFC BW30XFR	Cu ²⁺	25	89.98 to 94.21 %	10, 20, 30 et 50 bar, 25, 35 et 45°C; pH : 2 - 11	Effect of Operating Pressure	- A copper stock solution of 2000 (mg/L) was prepared from copper sulfate pentahydrate salt - The temperature correction factor (TCF) was studied - Every 10 min, the permeate water was collected and weighed to obtain the mass of permeate that would then be used to calculate the permeate flux and the metal rejection. - The product water samples were analyzed using a total dissolved solids (TDS) meter (VSTAR20, Thermo Scientific, Waltham, MA, USA) - The results show that when the pressure increases from 10 to 40 (bar) while keeping the other parameters constant (T = 25 °C, QF = 2 L/min, CF = 50 ppm), the permeate flux increased 180% and removal % increases from 89.98 to 94.21% - The results show that when the temperature increased from 25 to 45 °C with other constant parameters (P = 10 bar, QF = 3.2 L/min, CF = 50 ppm), the permeate flux increased 45% from 46.12 to 67.0 (kg/m ² ·h) (permeate flux is increasing 2% every 1 °C);	Harharah et al., 2022
		50			44.28 to 124.98 kg/m ² ·h;		
		100			Effect of Feed Temperature		
		150			46.12 to 67.0 (kg/m ² ·h);		
					Effect of Feed Concentration		
					87.41 to 83.86 (kg/m ² ·h).		
					Effect of Feed Flow Rate		
					82.2 to 84.3 (kg/m ² ·h)		
BW30XFR	Cr ⁶⁺	5 30	99.8 94.3	10; 30; 45 bars and 25,	30 to 158 kg/m ² ·h (25°C)	- Samples were subjected to incrementally increasing operating pressure; 10, 30, and	Shigidi et al., 2022

		100	77.2	35, 45 et 55 °C.	45 bar and flow rates; 2.2, 3.4, and 4.5 L/min under various temperatures; 25, 35, 45, and 55 °C	
				70 to 226 kg/m ² .h (55°C)	- Results obtained presented a higher rejection percentage at lower feed concentrations with a value up to 99.8% for 5 ppm in comparison to 94.3% for 30 ppm and 77.2% for 100 ppm - The permeate flux at 25 °C is recorded for all tested samples in the range of 30 to 158 kg/m²h, this range has risen at 55 °C under the same conditions to the range of 70 to 226 kg/m²h - Indicating alteration within the membrane pore size due to temperature increase and high applied pressure concluding high sensitivity of polymeric membranes towards changing permeate flow rate with increasing temperatures.	
RO X-20TM	Cr ³⁺ Ni ²⁺ Cd ²⁺ Pb ²⁺ Sb ³⁺ As ³⁺	15, 26, 56 ppm	99 98–99 99 99 98–99 98–99	50 bars	- Synthetic water and real water - These samples were first filtered on Whatman MN 615. Φ 125 mm REF 431 012 filter paper model to retain large particles in solution, before being filtered through RO and NF membranes in the laboratory.	Lumami et al., 2022
NF90	Cr ³⁺ Ni ²⁺ Cd ²⁺ Pb ²⁺ Sb ³⁺ As ³⁺	15, 26, 56 ppm	95–98 97–99 89–92 73–76 61–64 51–54	38 bars	- Rejection of three ions in solution by adjusting the pH of the feed solution and a chemical pretreatment before filtration. - The methodology of this study differs from previous studies by filtering more ions in solution without prior chemical pre-treatment.	
NF270	Cr ³⁺ Ni ²⁺ Cd ²⁺ Pb ²⁺ Sb ³⁺ As ³⁺	15, 26, 56 ppm	98–99 98 92–95 77–82 61–64 48–53	38 bars		

1.5.3. Energy consumption in RO

The energy consumption of RO technology, which is largely determined by the core component of RO membrane, is still relatively high and the extensive application of reverse osmosis process is limited to a certain extent (Lee et al., 2011).

Recently, RO membranes operating at ultra-low pressures have been developed (Van der Bruggen et al., 2003, Matsuura, 2001; Matsuura et al., 2001; Semiat, 2000), which allow the desalination of brackish water at pressures comparable to those applied in NF.

The energy consumption in RO has undergone huge improvements over the past five decades, the specific energy consumption of seawater reverse osmosis (SWRO), has undergone huge improvements over the past five decades, specific power consumption of SWRO has been reduced by 20 kWhm⁻³ in 1970 to only 2.5 kWhm⁻³ in 2010. It has been estimated that 0.69 - 0.79 kWhm⁻³ could be saved by intelligent process integration with intrinsic heat recovery (Kiho et al., 2020). The low energy consumption in RO wastewater has been confirmed by (Van der Bruggen, 2021) the latter explains that recycling wastewater is more favorable than seawater desalination and would operate at a pressure much lower.

1.5.4. Main disadvantages of PA-TFC RO membranes

1.5.4.1. Clogging

Fouling is a major limiting factor in RO applications (Malaeb et al., 2011). It is caused by clogging of pores or adsorption of solutes on the surface of the membrane (Van der Bruggen, et al., 2003). Pretreatment of feed water is necessary to prolong the lifetime of the membrane. membrane and prevent fouling Pretreatment usually consist of chemical coagulation, fine filtration, ultrafiltration (UF) or microfiltration (MF), scaling control such as softening, and acidification for pH regulation (Chian et al., 2007). Membrane fouling can be due to (1) microbial growth (Tran et al., 2007; Speth et al., 2000), (2) scaling, (3) dissolved organics, or (4) particulate matter can form compact cakes (Gabelich et al., 2002) Several studies have shown that the smooth surface and hydrophilicity of membranes reduce their vulnerability to fouling (Mondal et al. 2008; Vrijenhoek et al., 2001; Van der Bruggen et al., 2003).

1.5.4.2. Concentration polarization

Concentration polarization is a phenomenon that negatively effects the overall process efficiency of membrane-based processes (Koseoglu et al., 2018). It occurs when the concentration of a specific component increases or decreases at the boundary layer close to the membrane surface due to the selective transport through the membrane (Luis, 2018). In pressure separation processes, the solute is typically retained by the membrane, leading to a concentration profile similar to Figure 1.9.B below is obtained since the component will permeate faster through the membrane, being in the boundary layer where the transport is limited by diffusion (Luis, 2018). It has is similar in function to capacitive deionization by removing salt ions from the feed. However, ICP electrokinetic phenomenon, which occurs when a voltage is applied across a nanojunction or an ion permselective membrane. The basis of this method is ion depletion and ion enrichment, which are dynamic changes in ion concentration near the membrane to maintain electroneutrality (Rabiee et al., 2019).

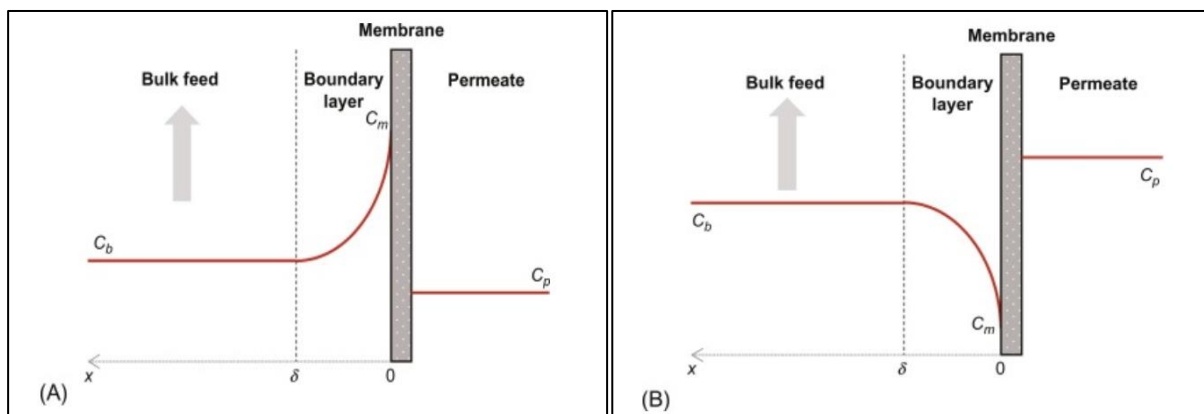


Fig. 1.9. Concentration profile with concentration polarization: (A) mass transfer limited by the membrane and (B) mass transfer limited by the boundary layer (Luis, 2018).

Concentration polarization produces a decline in the transmembrane flux, low retention of low molecular weight solutes and can lead to higher retention for mixtures of macromolecular solutes.

Table 1.6. Advantages and disadvantages of RO membranes (Baker et al., 2012 ; Chou et al., 2015; Oui et al., 2005; Gohil et al., 2017; Zhao et al., 2019 ; Yao et al., 2019 ; Zhu et al., 2019 ; Forman et al., 2019 ; Rozaini et al., 2019 ; Rezakazemi et al., 2019, Ayaz et al., 2019 ; Yang et al., 2019)

Membrane type	Advantage	Disadvantage
PA	- Exceptional water purification performance, - Improved chlorine resistance by secondary interfacial polymerization synthesis method	- The manufacturing cost is higher, - Compromise between permeability and selectivity, - Low permeate flux, low - chlorine resistance, - High energy
CA	- Better separation performance, - A low percentage of mineral fillers such as aluminum oxide has remarkably improved the compaction resistance of the membranes, - exceptional overall chlorine resistance, - Low manufacturing cost compared to PA	- The filtration performance depends on the degree of acetylation, - Higher acetylation will result in higher selectivity but lower water permeability, - Stable in the pH range 4-6 - High pH sensitivity - Lower tolerance to high T°
MMM	- Excellent chemical, mechanical and thermal tolerance - Good salt rejection and/or high-water permeability, - Low energy consumption - The main advantage of MMMs is to combine the low manufacturing cost, outstanding selectivity and high packing density of polymeric materials with long-term stabilities, high mechanical strength and regeneration capability of ceramic materials	- Selectivity depends on the number of ions in solution

1.5.5. Conclusions

The RO membrane process is very promising because of its high efficiency for the removal of organic and inorganic compounds from wastewater and simple scale-up. Removal of inorganic micropollutants varies between 95-100%, depending on the type of polymer, materials and operating conditions applied.

Permeate flux and percent removal are directly proportional to operating pressure on PA membranes. Recently, RO membranes operating at ultra-low pressures, similar to nanofiltration, have been applied to purify wastewater. It has been observed that polymeric membranes are very vulnerable to changes in operating temperature.

Improved performance at RO in terms of heavy metal discharge from wastewater and permeate flow can be achieved by pre-treating effluent to remove suspended and other particulates and, combining with nanoparticles in particular polymer matrix characteristics.

1.6. Thesis motivation

Health is a legitimate right claimed by every human being. The Democratic Republic of Congo (DRC), is confronted with problems of solid and liquid waste management due to the urban sprawl. wastewater treatment is one of the special issues considered by many researchers around the world in the last decade.

The DRC is the fourth country in Africa where the population density is becoming increasingly galloping at the national level estimated at 105 044 646 inhabitants. In the DRC, sanitation is not sufficiently developed and a remarkable lack of qualified human resources for the analysis and monitoring of environmental quality is evident.

In the country's urban areas, sanitation in general and liquid sanitation in particular are characterized by underdevelopment of both physical infrastructure and regional administrative structures. The widespread practice is to discharge effluents (domestic and industrial) into the environment without prior treatment, often into the rivers that run through the cities. As domestic effluent production is strongly correlated with water consumption, public structures also produce large volumes of wastewater.

Septic tank emptying is managed by the private sector and the national sanitation program. The latter program is a state structure, which is only operational in Kinshasa, the country's capital. The fact is that this sanitation practice is limited simply to emptying and the sludge is dumped at the emptying station in the city center, in the open air, but the rest of the process relating to evacuation and dumping/treatment is not a priority for public decision-makers as well as for civil society and the population. Moreover, industries are also important sources of pollution production. Sewage systems seem to be absent; and where they do exist, sewers are often dilapidated and are simply pipe systems used to drain raw sewage and rainwater flows directly to natural receptors (rivers, lakes, ocean).

Therefore, the concept of treatment is not high on the political agenda, and the population knows little about it. There are almost no wastewater treatment practices and all the treatment plants inherited when the country gained independence are out of service or have disappeared. Individual sanitation, left to private initiative, which largely dominates this sector, uses techniques that are carried out without control and, most of the time, are very basic due to the users lack of means.

The negative impacts of this situation affect the entire city and lead to a very degraded environment leading to a high prevalence of communicable diseases, morbidity, poverty, etc. The city of Uvira is not spared the threat of pollution due to the current high population density

estimated at 326 196 inhabitants according to the 2020 annual report of the Uvira health zone, creating enormous challenges in the supply of drinking water, drainage due to anarchic constructions, management and treatment of solid and liquid waste from households (HW), hospital institutions, craft and agri-food industries that are directly and/or indirectly discharged into the environment of Lake Tanganyika without any prior treatment.

This study aims at estimating the pollutant load and proposing the necessary corrective actions through pressure membrane filtration.

1.7. Thesis objective

The overall objective of this study was to evaluate the pollution load and consequent risk exposure to fish and humans in contact with the water of Lake Tanganyika and to carry out a comparative treatment exercise with commercial and modified membranes.

The study will provide decision makers with appropriate cost and performance methods to clean up polluted water before it reaches the receiving environment. The results of this research will be used for the protection, treatment, water quality improvement and sanitation of the Democratic Republic of Congo and the Central African region as a whole.

Specific research objectives:

- To evaluate the level of pollution in effluents both in the collector and in Lake Tanganyika.
- To evaluate the toxicological risk incurred by both fishes and the Riverine population.
- To evaluate the performance of both commercial and modified membranes for the removal of heavy metals from polluted water.
- To characterize the modified membranes by using various spectroscopic techniques.

The main research question to be studied is:

What is the exposure risk of fish and humans in contact with polluted water with heavy metals and, is nanofiltration or reverse osmosis a potential treatment technology for the efficient removal of heavy metals?

To achieve the above specific objectives, the following research questions can be asked:

- What are the occurrences, bioaccumulation and toxicity of micropollutants in polluted water?
- What is the risk incurred by fish and the riverine population in contact with polluted water?
- Which membranes are more appropriate to remove heavy metals from polluted water?
- What are the possible scenarios for the application of membranes in Uvira and Democratic Republic of Congo?

1.8. Thesis outline

This thesis is structured in six chapters:

The first chapter introduces the pollution context in Lake Tanganyika and reverse osmosis and briefly describes the state of the art of this process.

The second chapter presents the state of pollution in the city of Uvira located in the northwest of Lake Tanganyika. The lack of sanitation leading to the degradation of the local environment and in all cities of Democratic Republic of Congo has been described in this chapter.

The third chapter deals with the toxicological risk assessment of pollutants in living beings and determines the lethal concentration. This chapter briefly describes the bioaccumulation and toxicity of heavy metals and these results were used to develop strategies for the removal of pollutants.

The fourth chapter presents the evaluation of commercial membranes for the removal of inorganic pollutants from surface waters, this data acts as reference for the synthesis of the new membrane.

The fifth chapter is reserved for the general conclusion and the technical-economic evaluation and determination of the real viability of the implementation of the membrane process in Uvira and in the Democratic Republic of Congo.

1.9. Originality of the research

This study presents several original features. Firstly, the feed solution considered a large range of concentration of several heavy metals. The removal rate depends on the feed concentration and the properties of the membranes during manufacture.

Another original feature is the modification of the CS/ZIF-8 mixed matrix membrane, which operates at a relatively low pressure of 29 bar. The low operating pressure applied in a highly concentrated feed solution, in this study, prevents the membrane from being vulnerable to fouling and the energy consumption is low. The low pressure, high ion selectivity of highly concentrated solutions and high permeability are the key performance indicators for this study. Finally, the study focuses on the application of membrane processes in the Democratic Republic of Congo, Central Africa and low-income countries with low energy supplies.

Chapter 2. Evaluation of the pollution of the northwestern slope of Lake Tanganyika in the Democratic Republic of Congo

This Chapter presents the qualitative and quantitative analysis of pollutant loads from effluents discharged into Lake Tanganyika, with a view to evaluate remediation strategies. The discharge of effluents into Lake Tanganyika calls for concern as it poses serious threats to the biological wealth of the lake. Physic-chemical and microbiological parameters were evaluated for pollution from the effluent source to Lake Tanganyika (discharged point) using standard procedures. Results revealed significant pollution of the lake from prisons, the general hospital of Uvira and household channels. The prison of Uvira was the highest emitter of organic loads with a 42% chemical oxygen demand (COD), followed by the general hospital (30%) and finally households (28%). The absence of *faecal coliforms*, *Escherichia coli* and *Salmonella sp.* could be explained by the high concentration of chloride (varied from 1174 to 1388 mg/L) in this wastewater. The identification of latrines near the said collector reveals that 64% of septic tanks are connected directly or indirectly to the collector and 36% of indigenous latrines. The population is called upon to carry out individual sanitation, by building septic tanks that meets established standards and local authority's requirement to assist in establishing a waste station which can mitigate the problem. This chapter aims at establishing the state of the art on the pollution carried to Lake Tanganyika in quality and quantity and will allow to establish guidelines for the fight against pollution.

2.2. Methodology

2.2.1. Presentation of the study site

The study site was located by Google Earth and then Arc GIS version 9.3 software was used to design the map (Figure 2.1). The study took place in the City of Uvira, which has an estimated population of 311699 and a population density of 19481 inhabitants per km² according to the 2020 annual report of the Uvira Town Hall, and it is located at the end of the northwest basin of Lake Tanganyika in the East of the DRC in the Province of Sud-Kivu (West, 2001). It is located between 03°20' and 04°20' south latitude, 29° and 29°30' east longitude. The City of Uvira is bounded to the North by the Ruzizi plain, to the South by the Fizi Territory, to the West by the Mitumba Mountains and finally to the East by Lake Tanganyika which gives it an opening towards the Burundi, Tanzania and Zambia from the port of Kalundu. A global positioning system (GPS) was used to take the geographic coordinates of the sampling sites with coordinates 29°08'32.7" East, 03°23'31.2" South and 29°08'39.0" East, 03°24' 26.3" Figure 1.5 showed the location of the study area and Figure 2.1 shows the sampling sites.

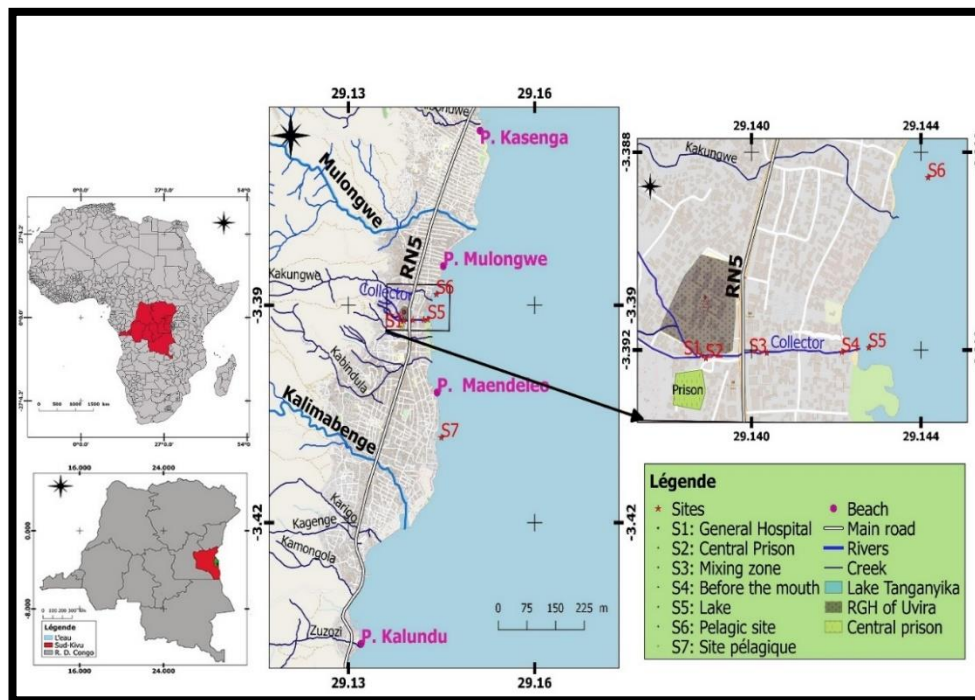


Figure 2.1. Localization of sampling sites.

2.2.2. Sampling and analytical methods

In this study, the European standard (NE), the French standard (NF), the International Organization for Standardization (ISO) and the French association for standardization

(AFNOR) methods were used because of their reliability, with analysis errors being negligible. Samples were taken from four sites along the BTC collector and the fifth site was from Lake Tanganyika in the direction of effluents, in order to determine the dilution of pollutants in the Lake (see the dotted lines on the map). Eleven samples were taken from each site from February 2015 to October 2018. Surface water samples 0 meter deep from Lake Tanganyika and the collector were taken in 1L bottles and then kept cold at 10°C. The samples were sent to the various laboratories in coolers containing cold accumulators to absorb the heat. Analyses were carried out as soon as the samples arrived at the water chemistry laboratory of the Higher Pedagogic Institute/Bukavu, the environmental analytical chemistry laboratory at the University of Burundi/Bujumbura and the microbiology laboratory at the University of Dar Es Salaam. At the chemistry laboratory of the Hydrobiology Research Center/Uvira, chemical analyses were carried out directly after sampling.

The measurement of the flow rate was carried out by a chemical or dilution method, which consists in injecting a miscible substance (sodium chloride) of known concentration in water, and measuring the concentration at a certain distance downstream (good mixture) over time. The flow rate was then calculated using the following formula (Agence de l'eau, 2006):

$$\text{Flow rate (L.s}^{-1}\text{)} = \frac{(Q_s \times 1000 \times 1.86)}{(C_d \times T_f)} \quad (2)$$

Where:

Q_s : quantity of salt injected in grams; T_f : flow time in seconds.

C_d : difference between the average conductivity (C_m) and the initial conductivity (C_i) in $\mu\text{S/cm}$, Eight flow rate measurements were the subject of this sampling during normal times (around 7 am to 9 am) and on the other hand a single sampling was made just 10 minutes after a heavy morning rain on October 9, 2015.

The physical-chemical parameters studied were: pH, electrical conductivity, suspended matter (SM), Chemical Oxygen Demand (COD) and Biochemical Oxygen Demand for 5 days (BOD_5). The pH was determined in situ by a Hanna HI 9828 pH meter fitted with a probe measuring electrical conductivity and dissolved oxygen.

The suspended matter was determined by filtration of a volume of waste water on a cellulosic filter (125 mm); (Rodier, 1996).

Unfiltered samples of total phosphorus and nitrogen were measured using a Hach Lange model DR 2800 spectrophotometer. 2 mL of the sample was added to the near-dose test tube containing the reagents, after mineralization for 1 hour, and then left for 30 minutes.

Chloride was determined by the Mohr method, according to the international standard ISO 9297.

$$NaCl = \frac{V_{AgNO3} \times N_{AgNO3} \times M_{NaCl} \times 1000}{V_{(Test\ sample)}} \quad (3)$$

Turbidity was determined by the photometric method ISO 7027 Turbidimeter.

Suspended particles (SP) were determined by filtering a volume of wastewater through a glass fiber filter membrane (AFNOR standard, NF T90-105) of 0.47µm diameter (RODIER, 2005),

$$SP = \frac{w_2 - w_1}{V} \times 100 \quad (4)$$

The COD was with the test tube quasi dosed containing all necessary reagents. After mineralization at 148°C for 2 hours, let stand and then make the reading with with the spectrophotometer DR 2800 model Hach Lange, eight measurements per site were performed. The determination of the biological oxygen demand, BOD₅ brute, was performed on unfiltered samples using OxiTop® systems, by ISO 5815 - 1: 2003 and ISO 5815-1: 2019(Fr). A quantity of the sample indicated for the estimated BOD₅ range was introduced. The whole was placed in an incubator that ensured a constant temperature of 20°C.

Coliforms are bacteria that have been artificially grouped together in a group that includes bacteria of telluric and faecal origin. This group has no taxonomic significance, and they are considered to be technical indicators of general hygiene control (faecal contamination) and cleaning systems (Boyer, 2022).

Coliforms and *E. coli* were analysed by the membrane filtration method, (CEAEQ, 2016; USEPA, 2002) and *Salmonella sp* by the presence/absence method, (CEAEQ, 2013). Macro invertebrate was determined by the keys (19.Cl; 19.C3; 19.C6; 19.C11), (Tachet, 2003). The pollutant load was calculated using the following relationship (EPA (NURP), 1983):

$$F (kg/d) = Q_i \times C \times T_i \quad (5)$$

Where:

F: is the polluting flux,

Q_i: the flow rate,

C: the concentration of a given pollutant, T_i: time interval.

The water quality was assessed according to the classification of Bliefert and Perraud (2001) (Table 2.1) and the simplified list to assess the quality of surface water (Table 2.2).

Table 2.1. Classification of stagnant and running surface water according to their quality (Bliefert and Perraud, 2001)

Quality class	Rate of saturation O ₂ (%)	Dissolved O ₂ mg/l	BOD ₅ (mg/L)	COD (mg/L)	Quantity of water
1A	≥ 90	≥ 7	≥ 3	≥ 20	Excellent
1B	70....90	5 7	3....5	20....25	Good
2	50.....70	3....5	5....10	25....40	Average
3	< 50	< 3	10....25	40.....80	poor
4	-	-	> 25	> 80	Out of range

Table 2.2. Simplified list for the evaluation of chemical qualities of surface water

Dissolved O ₂ (mg/L)	BOD ₅ (mg/L)	COD (mg/L)	NH ₄ ⁺ (mg/L)	Total P (mg/L)	Fecal coliform (CFU/100 mL)	Category
>7	<3	<20	<0.1	<0.1	<20	Excellent
7 - 5	3 - 5	20 - 25	0.1 – 0.5	0.1 – 0.3	20 - 2000	Good
5 - 3	5 - 10	25 - 40	0.5 - 2	0.3 – 0.5	2000 – 20	Average
3 - 1	10 - 25	40 - 80	2 - 8	0.5 - 3	>20000	Bad
<1	>25	>80	>8	>3	-	Very Bad

Source: Secretary of State to the Ministry for Energy, Mines, Water and the Environment, in charge of Water and the Environment « Web www.water.gov.ma. »

a: Multiparameter Hanna HI 9828 b: Spectrophotometer DR 2800

c: OxyTop bottles for BOD₅ d: Mineralizer

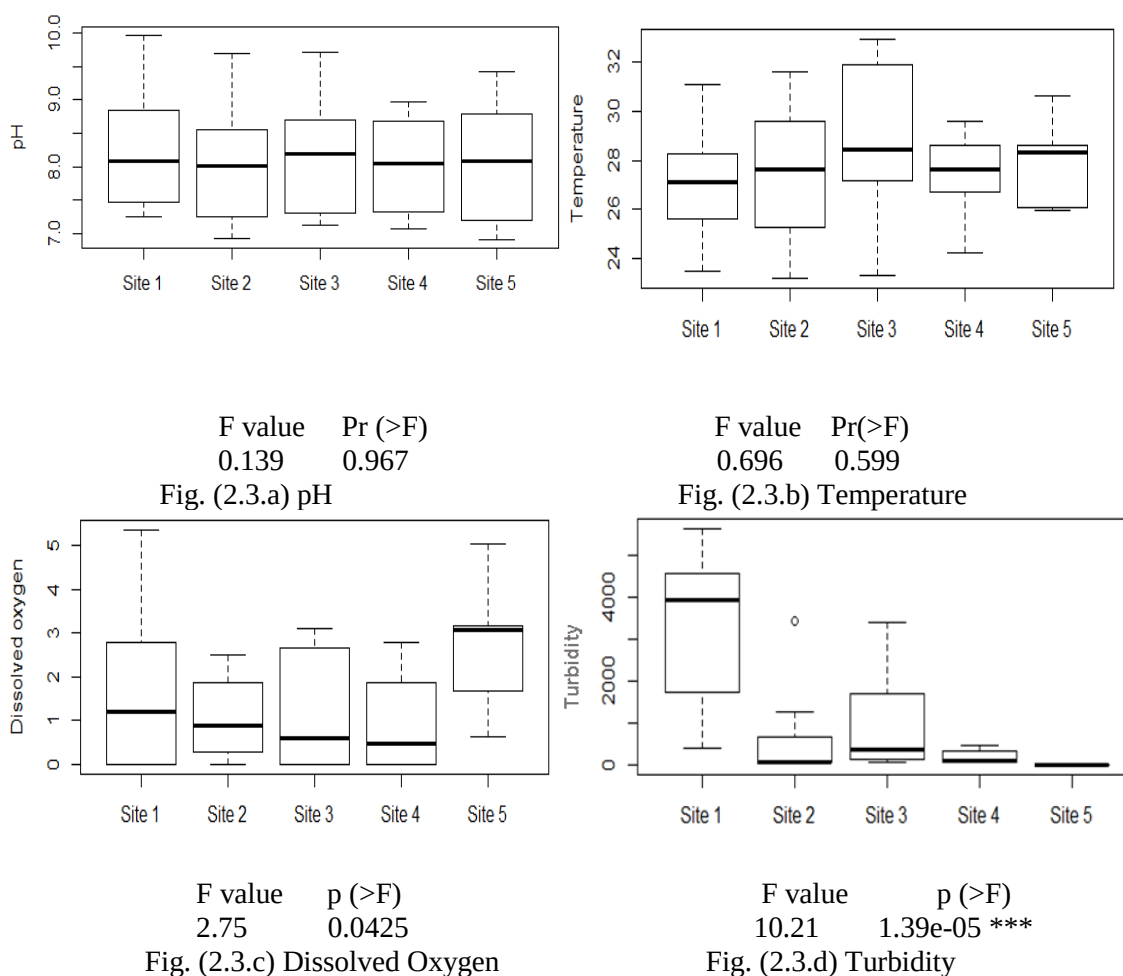
Figure 2.2. Equipment used for these parameters.

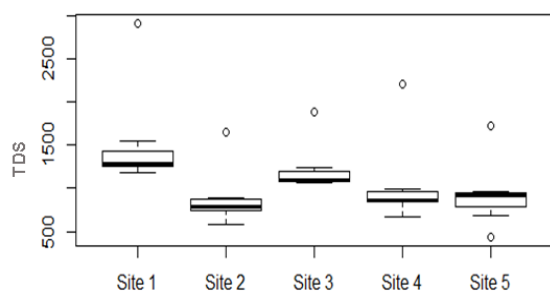
2.3. Results and Discussion

2.3.1. Physical-chemical characteristics of wastewater and tributaries

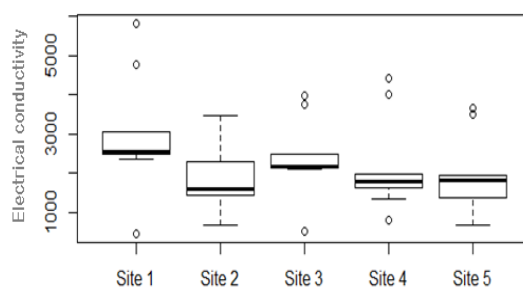
Results of global chemical and physical parameters are presented on Figure 2.3. The pH had its minimum and maximum values between 7 and 9 (Figure 2.3.a). Acidic character was observed at sites 2 and 5. Low pH (acidic waters) increases, in particular, the risk of the presence of heavy metals in a more toxic ionic form. High pH increases ammonia concentrations and statistical analysis shows that there was no significant difference ($p = 0.967 > 0.05$) between the pH values.

The variability of the pH depends on natural processes due to interactions between carbonate rocks and wastewater rich in organic matter. The discharge of wastewater containing detergents and soap products and septic tank greywater influenced the basicity of the pH in the collector. The very slightly acidic pH was observed in a few samples at sites 2 and 5, and it would be obtained by the interactions of the physical alteration of rocks with atmospheric CO_2 .





F value p (>F)
2.385 0.0734
Fig. (2.3.e) Total Dissolved Solids



F value p (>F)
1.513 0.214
Fig. (2.3.f) Electrical Conductivity

Legend

*** - significant

The temperature of the water ranged from 25 to 32°C (Fig. 2.5b). It plays an important role in the solubility of salts and gases including, among others, oxygen necessary for the balance of life. In addition, the temperature of this wastewater was very high due to the pollutant load and decreased the dissolved oxygen (DO) content of the water. Statistical analysis showed that there was no significant difference ($p = 0.599 > 0.05$) between the measured temperatures. The DO (Fig. 2.3.c) of the wastewater was too low, which leads to a sharp decrease and sometimes lack of DO which can lead to the disappearance of certain species, the decrease of self-purification, the accumulation of foul-smelling deposits (odours), accelerated growth of plant (including algae). Statistical analysis showed that there was a difference ($p = 0.042 < 0.05$) between DO values.

With the exception of the lake, the wastewater from other sites contained inadmissible turbidity (Fig. 2.3.d) and statistical analysis showed that there was a significant difference ($p = 0.00001 < 0.05$). This significant turbidity of the water lead to a reduction in its transparency which reduces the penetration of solar radiation useful for the aquatic life (photosynthesis). The increased turbidity of the water and the deposition of sediments counteract the growth of algae, which could have profound effects on other components of the food chain. In the study of ostracods through a variety of habitats that were disturbed by sediments in a light, moderate or high way, Cohen et al. (1993) found that ostracods from highly disturbed environments (both hard and soft substrates) were significantly less diverse than those in less disturbed environments, with differences in specific richness ranging between 40 and 62%. Total Dissolved Solids (TDS) (Fig. 2.3.e) values were much higher for all the sites and statistical analysis showed that there was no significant difference ($p = 0.0734 > 0.05$) between its values. All the waters analysed had electrical conductivity (EC) that were not acceptable because they

were much higher than the guideline value of 1000 $\mu\text{S}/\text{cm}$ (Fig. 2.3.f). The large conductivity could be attributed to the strong mineralization of the water and the presence of sedimentary rock in the city. Large organic pollution loads increase EC. Statistical analysis shows that there is no significant difference ($p = 0.214 > 0.05$) between the EC.

2.3.2. Flow rate

The flow rate of the collector was 0.52 L/s for normal time and 4.87 L/s under the rain (Table 2.3). These values were higher than other rivers and collectors of wastewater such as the Bwindi and Kawa rivers at Bukavu which are respectively 0.27 and 0.11 m^3/s . This flow rates makes it possible to assess the pollutant load which flows daily into Lake Tanganyika.

Table 2.3: Liquid flow rate measurement

N°	Sampling time	Flow rate L/s
01	Normal time	0.52
02	Under the rain	4.87

2.3.3. Emitters of pollutants

The three major emitters of pollutants were the Penitentiary Center (PC), the Uvira GRH and HW. The PC was designed for a capacity of 150 prisoners, but unfortunately it had 1025 persons (about seven times more at the time of this study), according to the annual statistics of the Penitentiary Center (PC) of Uvira, established in December 2019. The 2019 annual report of the GRH, describing an average of 400 patients per month and 111 HW with 284 people had been identified. Approximately 1709 people were thus connected to the collector.

In all, we found that the major emitters of pollutants from this collector, out of the 100% of COD, are the PC (42%), followed by the GRH (30%) and households (28%). Unlike COD, BOD_5 showed that HW production (33%) was higher than that of the GRH (30%). This is because hospital effluent seems not to be easily biodegradable as compared to HW waste; Figure. 5 and 6 shows the current state of BOD_5 and COD.

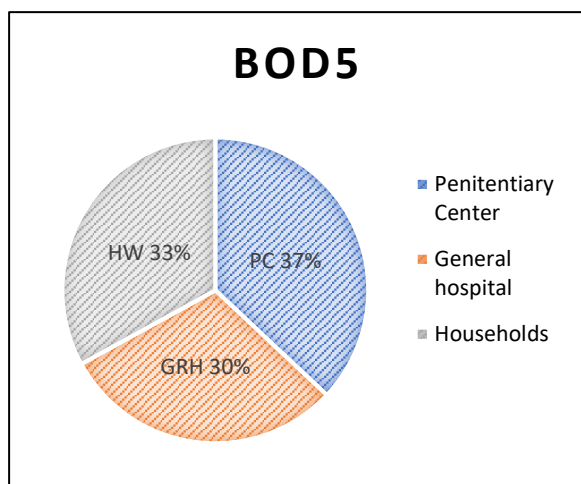


Figure 2.4: % BOD5 of various emitters

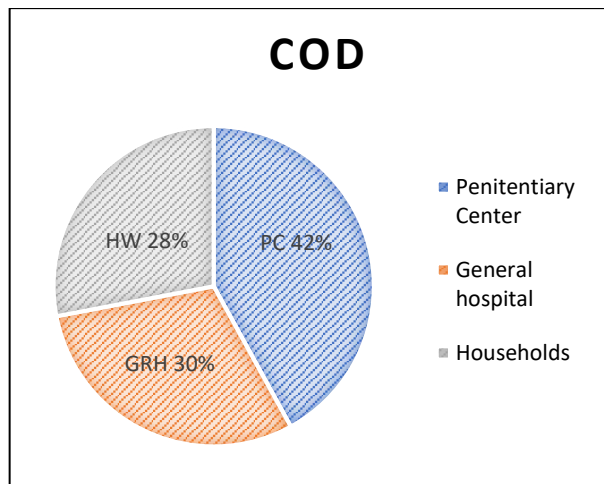


Figure 2.5: % COD of various emitters

In the set of households that were overloading the collector, 64% of these HW use septic tank latrines, whose drainage pipes were connected directly to the collector and 36% of these HW use traditional latrines wherein as soon as it is full, the HW destroys it to build another. The identification and quantification of toilets near the collector are shown on Figure 2.6.

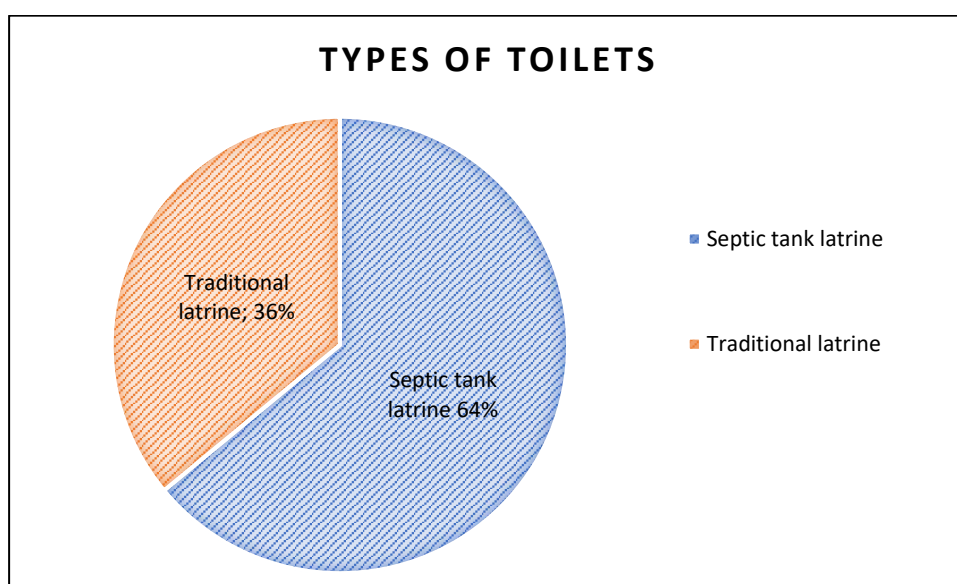


Figure 2.6: Quantification of the types of toilets surrounding the collector

2.3.4. Characteristics of the organic load of liquid effluents

The values of BOD₅ and COD shown on Figures 2.7 and 2.8 are too high in certain sampling sites. Site 5, which is the lake, presented certain values a little lower than those of the other sites because there was dilution along the line. Despite this, these values were still higher compared to the established standard. Ndrianirina et al. (2020) presented results whereby BOD₅ values

ranged from 85 to 225 mg/L while COD values ranged from 175 to 530 mg/L in domestic wastewater from the urban centre of Antananarivo, Madagascar.

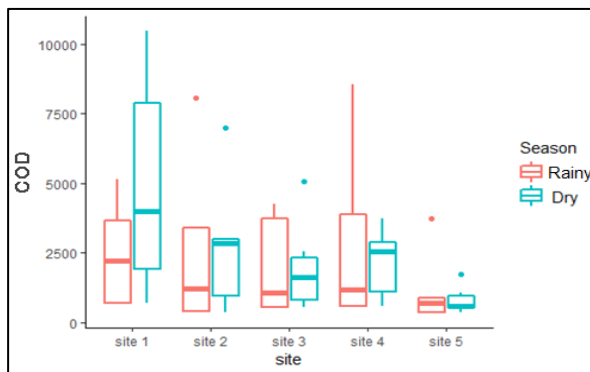


Figure 2.7: Seasonal variation of COD

F value = 2.046, $p = 0.104$

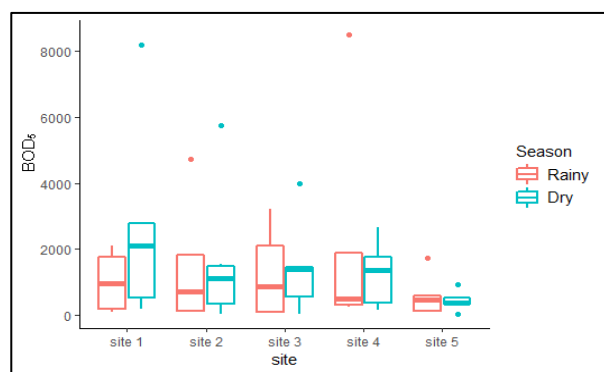


Figure 2.8: Seasonal variation of BOD₅

F value = 0.931, $p = 0.455$

The BOD₅ indicates the amount of oxygen which bacteria and other microorganisms consume in a water sample during the period of 5 days at a temperature of 20 °C to degrade the water contents aerobically. BOD₅ is thus an indirect measure of the sum of all biodegradable organic substances in the water. The COD and BOD₅ values of this collector were very much higher than the standards and varied from site to site. Their values varied seasonally (F value = 2.04 for COD and 0.931 for BOD₅). Figure 2.9 shows the degree of degradability of the effluent in the collector. By calculating the degree of biochemical degradation α ($\alpha = \text{BOD}_5/\text{COD}$), its values varied from one site to another. These values are sometimes close to 0.5 which explains tha biochemically non-biodegradable compounds are present in high concentration.

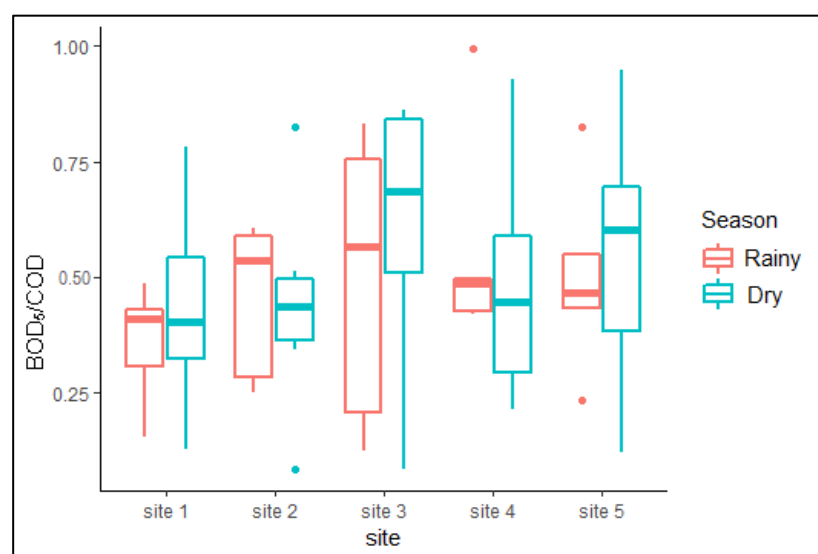


Figure 2.9. BOD₅/COD ratio of the various sites

The relationship between BOD₅/COD varies depending on the period, and the following observations were made: site 1 (exit of the PC) evacuated non-degradable liquid in February, March, May 2015, and February, March, May, September, and October 2016, while the waste evacuated in June, July and August 2016 where more biodegradable. Site 2 (exit of the GRH) released biodegradable waste to the BTC collector in February and March 2015, July, August, September and October 2016 and on the other hand in May 2015, February, March and June 2016, its wastes was not biodegradable. Site 3 is the mixing zone between the PC (site 1) and the GRH of Uvira (site 2), the choice of this site allowed us to assess the contribution of heavy metal to the collector.

2.3.5. Determination of the hydrogram and pollutogram

The average pollution parameters of this collector varied significantly in space. Peaks were most observed around 10 a.m. to 1 p.m. Statistical analysis by Kruskal wallis and ANOVA showed that the difference between the variations is not significant ($p > 0.05$). Table 2.4 summarizes the means and standard deviations of the spatial variation of the pollution parameters.

Table 2.4. The means and standard deviations of the pollution parameters.

	7 a.m. (X $\pm$ SD)	10 a.m. (X $\pm$ SD)	1 p.m. (X $\pm$ SD)	4 p.m. (X $\pm$ SD)	chi-squared	p
Flow rate (L/s)	0.79 $\pm$ 0.12	0.82 $\pm$ 0.23	0.92 $\pm$ 0.36	0.71 $\pm$ 0.08	0.74	0.8
COD (mg/L)	367.66 $\pm$ 216.8	568.66 $\pm$ 478.0	911 $\pm$ 1023.72	292.66 $\pm$ 84.47	1.05	0.7
BOD ₅ (mg/L)	88.3 $\pm$ 48.04	221.66 $\pm$ 242.6	10.93 $\pm$ 487.88	59.33 $\pm$ 14.3	1.30	0.7
NH ₄ ⁺ (mg/L)	9.48 $\pm$ 3.26	10.72 $\pm$ 6.38	10.93 $\pm$ 6.17	11.23 $\pm$ 1.76	0.55	0.9
Total N (mg/L)	23.83 $\pm$ 3.35	19.58 $\pm$ 7.4	19.7 $\pm$ 7.5	21.55 $\pm$ 3.48	1.25	0.7
Total P (mg/L)	12.67 $\pm$ 5.12	27.21 $\pm$ 29.08	30.45 $\pm$ 33.6	18.59 $\pm$ 11.40	0.28	0.9
TDS (mg/L)	512 $\pm$ 518.16	382.66 $\pm$ 356.42	2090 $\pm$ 3084.36	386.66 $\pm$ 246.84	0.81	0.8
Chloride (mg/L)	1221.33 $\pm$ 86.63	1273.33 $\pm$ 187.08	1338 $\pm$ 147.39	1253.33 $\pm$ 44.61	1.51	0.6

* $p < 0,001$: Highly significant, ** $p < 0,01$: very significant and *** $p < 0,05$: significant

From Table 2.4, it was observed that the average values of pollution parameters were very high compared to the standards established by Bliefert and Perraud (2001) and State Secretariat for Energy, Mines, Water and Environment, in Rabat in Morocco (S.E.E.E).

The temporal variation of pollutants of the said collector was also varying daily. The statistical test of Kruskal wallis and ANOVA showed a significant daily difference in COD ($p = 0.04 < 0.05$), NH₄⁺ ($p = 0.03 < 0.05$) and Chloride ($p = 0.03 < 0.005$). Table 2.5 summarizes the means and standard deviations of the temporal variation of the pollution parameters.

Table 2.5. Temporal variability of the physicochemical parameters

	23/07/2018 X $\pm$ SD	25/07/2018 X $\pm$ SD	27/07/2018 X $\pm$ SD	chi-squared	p
Flow rate (L/s)	1.002 $\pm$ 0.26	0.72 $\pm$ 0.05	0.71 $\pm$ 0.13	5	0.06
COD (mg/L)	977.7 $\pm$ 825.8	238.5 $\pm$ 65.8	388.7 $\pm$ 151	6	0.04***
BOD ₅ (mg/L)	387.5 $\pm$ 396	56 $\pm$ 8.5	85.7 $\pm$ 47	4.5	0.1
NH ₄ ⁺ (mg/L)	12.6 $\pm$ 0.82	6 $\pm$ 2.7	13 $\pm$ 3.7	6.5	0.03***
Total (mg/L)	23.7 $\pm$ 2.1	17 $\pm$ 7.3	22 $\pm$ 3.7	1.6	0.43
Total P (mg/L)	38.5 $\pm$ 30	13.5 $\pm$ 5.2	14.5 $\pm$ 10	1.4	0.49
TDS (mg/L)	1573.5 $\pm$ 2718	215 $\pm$ 23	740 $\pm$ 295	4.5	0.1
Chloride(mg/L)	1388 $\pm$ 98.1	1252.5 $\pm$ 61	1174 $\pm$ 82.6	6.6	0.03***

* p < 0,001: Highly significant, ** p < 0,01: very significant and ***p < 0,05: significant;

The daily averages of COD, BOD₅, N ammoniacal, total N, total P, suspended matter and chloride on Monday 23/07/2018 were very much higher compared to those on Wednesday 25/07/2018. On the other hand, the COD, BOD₅, N ammoniacal and suspended matter on Friday 27/07/2018 were higher than the values on 23/07/2018 and 25/07/2018. Low concentrations were observed on the second day of sampling (Wednesday 25/07/2018). This difference in daily concentrations is explained by the anthropic activities carried out in the central prison of Uvira which includes significant number of prisoners whose sanitary conditions are not appropriately respected. Prisoners usually do their maintenance work at the start and end of the week, according to information received from their officials. The chloride content was too high, although not harmful, but is an important indicator of the presence of pollutants.

Results for COD, BOD₅, suspended matter and ammoniacal N were very high compared to the results of Buhungu et al. (2017) on the Kinyankoge river in which ammoniacal N varied from 3.2 to 11.5 mg/L, COD from 59 to 85 mg/L and BOD₅ from 26 to 38.6 mg/L. They were also higher than those of Njoyim et al. (2016) in which, N ammoniacal varied from 0.56 to 2.6 mg/L and Chloride from 0.7 to 8.6 mg/L; Ndikumana et al. (2013) who studied the collectors of Bujumbura and COD varied between 209 and 676 mg/L, BOD₅ between 39 and 114 mg/L. Pollution parameters of this collector were also higher than the parameters of the wastewater of Bukavu rivers (Kapepula et al., 2015). This collector takes a significant amount of suspended matter (average 1574 mg/L) into the lake. Lake Tanganyika usually blows a strong wind towards the south and this wind in turn produces strong waves. In the agitated state of the lake, the pollutants take an orientation perpendicular to the left lateral. On the other hand, if there is no wind, no waves will be generated and the pollutants would disperse to the lake linearly. Table 2.6 shows the evolution of the dilution of pollutants in Lake Tanganyika.

Table 2.6: The dilution of pollutants in Lake Tanganyika

Linear dilution in meter	COD mg/L	BOD ₅ mg/L	Total N mg/L	Ammoniacal N mg/L	Total P mg/L
Discharge system	131	79	17.2	9.2	15.6
Linear dilution 1	121	70	13.1	7.1	12.2
Linear dilution 2	118	72	11.8	4.02	6.28
Linear dilution 5	112	76	4.3	1.1	2.1
Linear dilution 8	98	71	2.08	0,8	0.9

It was observed that the pollutant load was higher at the coast of the lake and the dilution was linear in normal time at Lake Tanganyika up to a distance of 8 m. There was a significant amount of ammonia in the lake, which is an indication of contamination by discharges from domestic sources Buhungu et al. (2018). Human or animal urine indeed contains large amounts of urea which quickly transforms into ammonia.

2.3.5.1 Determination of the hydrogram

The collector flow was not constant, and showed a maximum around 10 a.m. and 1 p.m. By analyzing Figure 2.10, it can be seen that in normal times, the flow rate of this collector did not vary appreciably. The peaks observed on July 23, 2018 from 10 a.m. to 1 p.m. are due to the intense activities of the PC of Uvira at the beginning of the week and also these hours were strategic, because school children are getting ready for school and the others arriving home get ready for housework.

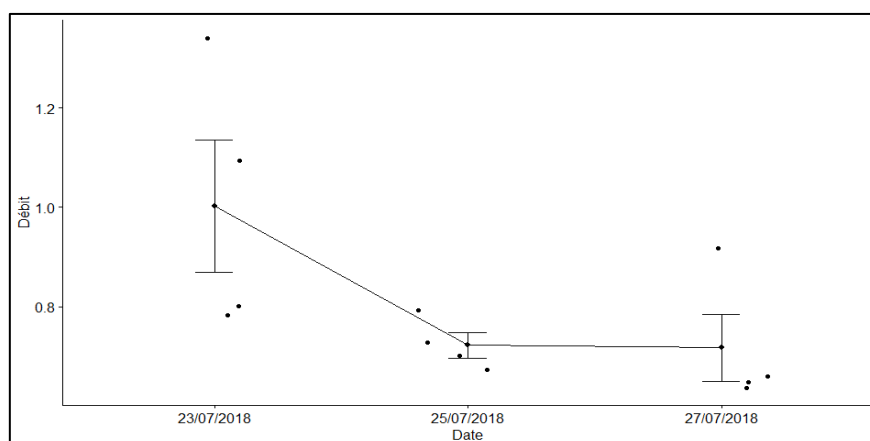


Figure 2.10. The daily hydrogram of the collector BTC

2.3.5.2 Pollutogram

The wastewater from the central prison of Uvira, the GRH of Uvira and HW was very polluted because the pollutant flow in kg/d carried into Lake Tanganyika presented much higher values. The high flow rate was observed at the beginning and at the end of the week; this is explained by the maintenance activities at the PC which are usually carried out on Monday and Friday. Table 2.7 presents the values in kg/d of pollutant flow discharged into Lake Tanganyika.

Table 2.7: Pollutant flow sent to Lake Tanganyika in kg/d

	Parameters	23/07/2018	25/07/2018	27/07/2018
Pollutant flow (kg/d)	BOD ₅	4.2	0.43	0.65
	COD	10.5	0.43	0.65
	NH ₄ ⁺	0.13	0.05	0.1
	Total N	0.25	0.13	0.17
	Total P	0.41	0.1	0.11
	TDS	17	1.67	5.67
	Chloride	15	9.73	9

kg/d: The quantity in kilograms of pollutants carried to the lake per day

2.3.6. Biotic parameters

Total coliforms in the samples increased from site 4 at the collector outlet to site 5 at Lake Tanganyika at a distance of 2 meters from the vertical inlet of the pollutant. The increase in the number of *Total Coliforms* in the lake is explained by the open environment of the lake, which receives all the solid and liquid waste from the city of Uvira (Lumami et al., 2016), and this solid waste are piled up on the shoreline of the lake, and currently the shoreline of the lake is considered a public dumping ground.

Faecal Coliforms, *E. coli* and *Salmonella sp* were completely absent in all the samples. *Coliforms* are naturally present in the environment, as well as in human and animal faecal waste. The high number of *Faecal Coliforms* is caused by the penetration of soil or organic matter into the water or by conditions favouring the growth of other types of *coliforms*. Therefore, the absence of *faecal coliforms* at other sites and the complete absence of *total coliforms*, *E. coli* and *Salmonella sp*, is explained by the high chloride content in these waters and other pollutants that inhibit their presence. Nchofua et al. (2020) upon analysing water for quality control in Bangolan, Northwest Cameroon, discovered that *faecal coliforms* were present in all the water samples with counts ranging from 8-160/100 ml. The identification of macro invertebrate made in the collector, revealed the presence of macro invertebrates of the order of *Diptera*, families of *Culicidae* and *Muscidae*, of the genus *Anophele* and *Musca*.

Mosquitoes (Diptera: Culicidae) present a major risk for human health worldwide (Van der Beek et al., 2020). Their larvae develop in wastewater, stagnant water and water that is rarely stirred, making it a perfect breeding ground (Carnevale et al., 2009). However, waterborne diseases are grouped into four categories: diseases related to an acute or chronic lack of clean water, diseases related to water contamination, diseases related to contamination of water by toxic substances and vector-borne diseases. These diseases are cholera, dengue fever, diarrhea, scabies, hepatitis, malnutrition, onchocerciasis, malaria, schistosomiasis, trachoma and typhoid (Collard et al., 2021). People are exposed to the risk of carcinogenic diseases if they regularly consume water or fish contaminated with undegradable substances such as heavy metals (Pb, Cd, Hg, etc.). On the other hand, for metals with a biological function (Cu, Fe, Zn, Mn, etc.) in cells, there is bioregulation and therefore no health risk. All these constraints are caused by environmental degradation, which requires remediation.

2.4. Conclusions

The measurement of the main pollution parameters reveals significant pollution of BTC collector by waste from the penitentiary, the general reference hospital of Uvira as well as households. This had a negative impact on Lake Tanganyika, that is the average values of COD and BOD₅: at site 1, were 3814 and 1890 mg/L; at site 2: 2713 and 1568 mg/L; at site 3: 2009 and 1361 mg/L; at site 4: 2538 and 1712 mg/L; at site 5: 994 and 472 mg/L respectively. The PC of Uvira was the first emitter of the organic load with 40% of COD and BOD₅, followed by HW (32%) and finally 28% at the general referral hospital of Uvira. During sampling, it was identified that 64% of households have septic tank latrines which are directly or indirectly connected to the collector, and as soon as they are full, households take advantage of emptying them in the rain.

The hydrogram flow rate varied between 0.7 to 1 m³/s during sampling and this influenced the average amount of pollutant load, which varied daily from 0.65 to 10.5 kg/d for COD, 0.65 to 4.2 kg/d for BOD₅, 0.05 to 0.13 kg/d for ammoniacal N, 0.17 to 0.25 kg/d for total N, 0.11 to 0.41 kg/d for total phosphorus, 9.73 to 15 kg/d for chloride and finally from 5.67 to 17 kg/d for suspended matter.

There was absence of *total coliforms*, *faecal coliforms*, *E. coli* and *salmonella sp* and could be explained by the high concentration of chloride in this wastewater, which varied from 1174 to 1388 mg/L and also others pollutants that inhibit the latter.

Regarding the results, the raw sewage from the PC of Uvira, GRH in Uvira as well as households are all connected to the collector built by the BTC and present values of the major physicochemical pollution parameters that relatively exceed the general maximum permissible values of direct and indirect discharges into the receiving environment.

Lake Tanganyika is at high risk of environmental pollution; hence the need to treat raw wastewater before discharge, as it poses a very high risk on the food chain. The company in charge of the distribution of drinking water in Uvira is unable to serve the entire population due to the large demographic distribution and a good number of persons rely on Lake Tanganyika with contaminated water which may be the cause of waterborne diseases and would also affect coastal fishing. As for the lake, the pollutant effect will cause a rupture in the trophic chain by the elimination of endemic and polysensitive species. The adoption of an individual sanitation system would be an asset for the protection of the environment and Lake Tanganyika in particular.

Chapter 3. Determination of the Toxicological Risk of Urban Waste from the City of Uvira Dumped into the North-Western Coast in Lake Tanganyika (Democratic Republic of Congo)

This Chapter focuses on determining the toxicological risks of urban waste from the city of Uvira, discharged into Lake Tanganyika, on the surrounding population. Volatile organic compounds were measured in a variety of solid waste matrices, including inorganic micropollutants in wastewater and fish. The concentrations of Hg and Pb in the lake were found to be 1.21 and 1.42 $\mu\text{g/L}$ respectively and between 0.83 to 18.36 $\mu\text{g/L}$ of Hg and 8.25 to 670 $\mu\text{g/L}$ of Pb, at the collector outlet. The presence of trace metallic elements, such as Cr, Co, Ni, Cu, Zn, As, Sb, Hg and Pb, were detected at high concentrations compared to the WHO standard. An toxicology experiment herein on wastewater samples showed lethal pollutant concentrations of the order of 0.0055 mL/mL which killed at least 50% of fish (LC50), confirming the toxicity of the wastewater. These potentially harmful effluents also contain volatile organic compounds originating in high concentration from the pharmaceutical discharges of the general Uvira hospital, in particular: toluene, ethylbenzene, m-xylene/p-xylene, o-xylene and chloroform in higher concentrations compared to the norm. Other components such as benzene, bromodichloroethane and 1,1-dichloroethane were found to be present, but at a concentration below 0.05 ppb. A variety of trace organics can be suspected to be present as well.

3.1. Introduction

The problems associated with heavy metals contamination were first highlighted in industrialized countries, due to their larger industrial developments, and especially following accidents due to cadmium pollution in Sweden, and mercury in Japan (Minamata disease from 1949 to 1965) (Fernand et al., 1975; Lévy et al., 2008). Micropollutants are transferred through the aquatic environment to fish, humans and other piscivorous animals (Chen et al., 2000). Thus, aquatic life may be exposed to high concentrations of heavy metals and trace metals depending on their bioavailability and the particular organism exposed and may become persistent in the environment, constitute a degradation challenge aggravated (Kalay et al., 2000; Gouzy et al., 2008; Musibono et al., 2000).

The level of industrial activities is lower in most African countries, with 19% in North Africa and 14% in Sub-Saharan Africa, thus allowing activities to be monitored. This contributes to increased productivity, reduction of waste and emissions, and management of energy consumption (Le, 2019). There is growing awareness of the need to rationally manage aquatic resources and in particular to control waste discharges into the environment (Rashed et al., 2001; Chale et al., 2002; Okwonko et al., 20015; Asony et al., 2007).

The main aim of this Chapter is to determine the toxicity caused by waste discharged into Lake Tanganyika, due to lack of proper sanitation, and the negative impacts of this situation of the inhabitants of Uvira. It should be pointed out that the direct or indirect manipulation of humans for research purposes in the Democratic Republic of Congo is not easily authorised by the ethics committee, and the administrative burden could not allow it. For this reason, we studied fish that are eaten by humans. In a previous study (Lumami et al., 2020), the threat of physical-chemical pollution on the lake coast was studied. The present work is geared towards the development of a master plan for the management of liquid and solid effluents in the countries bordering Lake Tanganyika as well as everywhere else, and to develop strategies for the elimination of wastewater pollutants.

3.2. Materials and Methods

3.2.1. Presentation of the Study Environment

The wastewater for the toxicological test was collected in 20 L plastic drums and for the heavy metals assay was collected in 1 L bottles, at point S4 with coordinates 029°08'39.0" longitude N and 03°24'26.3" latitude E and was taken to the aquarium room of the Hydrobiology Research Center for experiments. Figure 2.1. above shows the site in green at Lake Tanganyika, the fish

capture site, was located 20 m away from homes. This study was mainly centered around these sites. The site in yellow color was used to water samples from Lake Tanganyika in order to determine the pollutant doses therein.

Wastewater at different dilution concentrations and Lake Tanganyika wastewater for heavy metal analyses were kept cold at 10°C to avoid any modification. Samples analysed in Bujumbura, Bukavu, Dar es Salaam, Uganda and Belgium were shipped in coolers containing cold accumulators to absorb the heat.

From a geological point of view, the slope of the western edge of the graben of Lake Tanganyika is represented by a metamorphosed Precambrian ensemble and a Cenozoic ensemble which is a superficial formation (Ilunga, 2006; Weis, 1959). These are sediments from Precambrian rocks (schists, quartzophyllades, quartzites, mica schists, gneisses, migmatites, dolomitic limestones and mylonites) which undergo physical alteration and their particles form these substrates in our sampling sites.

The rocks are interspersed in veins of white quartz, amphibolite lenses and pegmatite (Weis, 1959). The two fish sampling sites in the littoral zone of Lake Tanganyika are close and present the same characteristics, for example the vegetation consists of reeds (*Phragmites australis*) associated with lawn and the presence of sediments predominantly made up of quartzite, quartz and pegmatite gravels with a diameter of more or less 7 to 11 cm with the presence of green algae growing thereon. Natural sand and silt in small quantities were also observed along the shoreline of the sampling site.

3.2.2. Fish sampling

Nylon monofilament netting with mesh sizes of 0.8 cm, 1 cm and 1.5 cm was used to catch the fish. The net was set for one or two hours, depending on the state of agitation of Lake Tanganyika. When the net reached the bottom using a mask, the fish were chased towards it to be encircled. The fish caught in the net were brought back to the shore to be carefully removed so as not to injure them. The fish were transported to the lab using the 20 L bucket in which an air pump was installed to provide oxygen during their transport over a distance of around 1 km, from Lake Tanganyika to the HRC aquariums. On arrival at the HRC aquarium room, the fish were placed in a transit aquarium for a 2 hour rest to remove any tired fish.

In the aquariums, the fish were fed with earthworms and a paste made from maize and manioc flour after cutting the paste into small pieces. Stones containing algae were added to the

aquariums to supplement the nutrition of the algivorous fish. Other specific foods were not used to avoid any further contamination caused by these foods.

The fish sampled in Lake Tanganyika and the dead fish in the aquariums after spending time in wastewater were weighed on a precision balance supplied by Ohaus. The total length of the fish was measured using callipers, then dried at 60°C in the study. These samples were sent to dry boxes in Bujumbura and to Belgium for analysis of heavy metals using ICP-MS and ICP-OES.

The taxonomy of the fish used for the toxicological test is shown in Table 3.1.

Family	Genre	TL in mm	Weight (g)	Diet regime	pH	T°C	Ref
Cichlidés	Simochromis diagramme	157	63	Herbivore	7,8 - 8,8	23 - 28	Vanhove et al., 2010;
	Simochromis babaulti	98	13	Herbivore	7,8 - 8,8	23 - 28	Steenberge et al., 2020
	Oreochromis niloticus	152	60	Omnivore	6,5 - 8	26 - 30	Habibah et al., 2021

Table 3.1. Description of the individuals used in the biotic tests; TL: Total length.

3.2.3. Chemical and toxicological analysis

The samples used for analysis of the metallic trace elements were etched with 8 ml HNO₃ 65% to ensure the perfect dissolution of the residual metal particles and 2 ml H₂O₂ 35%; measured with ICP-MS: Thermo Scientific XSERIES 2 Quadrupole Inductively Coupled Plasma Mass Spectrometer (ICP-MS).

Volatile organic compounds (VOCs) with boiling points below 200°C, were determined using a hybrid method (SAIC 1992). The method uses a Gas chromatographic/mass spectrometric procedure with sample introduction by means of purging and trapping in order to analyze several compounds. A short, narrow I.D. SPB-624 column was used to shorten the analysis time to under 11 minutes. A high desorbed flow through the trap was necessary to prevent band broadening of the gaseous VOCs. This was accomplished by increasing the split ratio, thus increasing the total flow through the trap during desorption.

The method calibration data: 8260 /Haps/Method/Analyze/Headspace/ER_HSS_Tri-Bed_PPB_Quant.mth on the peak search parameters were as follows.

Table 3.2 below describes the HAPSITE ER Instrument: Gas Chromatograph/Mass Spectrophotometer.

Peak search parameters	Units	Value
Peak resolution	NA	5
Sound level multiplier	m/z	2000
Minimum area	NA	50000
Minimum width	Meter	7
Maximum width		70
Minimum adjustment	NA	0.900
Minimum purity	NA	0.400
Analysis time	Minutes	10

NA: not available, m/z: Thomson per second

Helium, an inert gas, was bubbled through a vessel containing the water sample. The VOCs partitioned into the gas phase were transported to a sorbent trap, where they are adsorbed. After the purge cycle, the sorbent trap was heated and the volatiles were swept into the GC column, where they were separated on the basis of their boiling points. The gas chromatograph was connected directly to a mass spectrometer which had bombarded the compounds with electrons as they sequentially exited the GC column. The resulting fragments, which have unique charge and mass characteristics for each compound, were detected by the mass detector of the spectrometer. The signal from the mass detector was used to construct a compound mass spectrum which was used to identify the compound. The detector signal intensities for the selected ions unique to each target compound were used to quantify the amount of the compound in the sample.

The toxicity tests were carried out in the aquariums of the Hydrobiology Research Center (HRC-Uvira) by bio-tests during which the fish have been exposed to a given pollutant in order to assess the concentration levels causing death.

These fish were chosen for the following reasons: 1) they are consumed by the local population; 2) they are found on the rocky coast (Grégoir et al., 2014) and are easy to catch; and 3) their efficiency to resist aquariums. They were caught on two rocky sites considered to be unpolluted in Lake Tanganyika.

This population of aquatic organisms was exposed to polluted wastewater at different concentrations of raw effluent that were successively diluted by a decimal dilution (10^0 , 10^{-1} , 10^{-9}), in aquaria for 96 hours to estimate the toxicity in order to assess the concentration levels causing toxic effects. These tests were carried out under controlled light conditions in a well-ventilated non-cumbersome aquarium room at room temperature between 24°C to 25°C. Furthermore, all aquaria were supplied with oxygen using a RENA air pump and EHEIM filter,

all 220V. An aquarium served as the control solution for the water samples from the lake, and in each dilution aquarium there were 20 individual fishes. The LC50 calculation method was used according to the literature (Finney, 1971; Branko et al., 2014).

$$LC50 = LC100 - ((\Sigma ab))/n \quad (6)$$

LD50: lethal dose 50

LD100: 100 % lethal dose

a: difference between two successive doses. b: average death rate between two successive doses.

n: average number of animals per batch

The least squares method consists in minimizing the sum of the squares of the deviations, in multidimensional cases between each point of the regression cloud and its projection that is parallel to the y-axis on the regression line.

3.3. Results and Discussion

3.3.1. Inorganic Micropollutants

The water samples used for toxicological analysis are listed in Table 3.3. It is worthy noting that the water from Lake Tanganyika was used as a reference and the wastewater samples from the collector diluted by 10 to 90% were considered as indicators of the toxicological level.

The highest concentrations for the different wastewater samples are underlined.

Table 3.3. Results of chemical analysis on aquarium water at different dilutions, in µg/L.

Conc aquarium	Cr	Co	Ni	Cu	Zn	As	Sn	Sb	Hg	Pb
Eau du lac	5	0.3	1.11	1.23	0.96	2.65	< 0.05	0.21	1.21	1.42
10%	3.18	1.42	3.69	0.87	0.24	6.09	< 0.05	2.18	<u>0.83</u>	< 0.05
20%	2.08	1.25	2.34	0.82	< 0.05	6.33	< 0.05	1.05	0.61	< 0.05
30%	3.83	1.64	3.96	1.72	16	5.35	0.05	3.01	0.48	< 0.05
40%	2.95	0.74	1.67	0.36	< 0.05	3.47	0.05	0.88	0.41	< 0.05
50%	4.92	1.01	2.79	1.74	< 0.05	4.3	<u>0.09</u>	1.08	0.37	< 0.05
60%	<u>7.25</u>	1.96	3.32	1.51	13	3.93	< 0.05	0.42	0.36	<u>8.25</u>
70%	6.2	0.58	1.69	1.33	2.24	4.59	< 0.05	<u>14</u>	0.33	< 0.05
80%	6.42	1.26	2.3	0.96	< 0.05	2.55	< 0.05	1.67	0.3	0.88
90%	7.15	0.97	1.55	<u>1.95</u>	<u>5.34</u>	2.86	< 0.05	0.31	0.26	< 0.05
WW	5.55	<u>2.76</u>	<u>4.36</u>	0.9	0.47	<u>7.09</u>	< 0.05	1.07	0.22	< 0.05
Normes OMS mg.l ⁻¹	0.05		0.07	2	3	0.01	0.01	0.02	0.01	0.01

%; dilution in percentage, Standards: (ESDAC, 2007)

Presented in Table 3.4 are the guidelines for the quality of surface and drinking water, as updated in 2017, which are the benchmarks for drinking water safety As can be seen from Table 3.3.

Table 3.4. WHO Surface Water Quality Standard

Element	Concentration normally found in surface water	WHO guidelines
Sb	< 4 µg/l	0.02 mg/l
As	-	0.01 mg/l
Cd	< 1 µg/l	0.003 mg/l
Cr ³⁺ , Cr ⁶⁺	<2 µg/l	Chrome total: 0.05 mg/l
Cu ²⁺	-	2 mg/l
Pb	-	0.01 mg/l
Hg	<0.5 µg/l	0.01 mg/l
Ni	<0.02 µg/l	0.07 mg/l
Zn	-	3 mg/l

2017, quality guidelines for drinking water

The reference values are lower than those obtained from the effluents from the collector, except those for Hg.

High concentrations of Hg and Pb were measured in the lake with values of 1.21 and 1.42 µg/L respectively. With regards to sampling in order to determine the toxicity of the effluent from the collector, the highest values were found to be: Pb: 8.25; Sb: 14; Zn: 16; Cr: 7.25; Co: 2.76; Ni: 4.36; Cu: 1.95; As: 7.09; Sn: 0.09 and Hg: 0.83 µg/L. The variation of the different concentrations in the aquarium as listed in Table 3.3 does not depend on the wastewater dilution factor as these samples were taken at the same site but on sampling time, since the samples were collected on two different days. It was observed that the variation of the concentrations could be due to the different activities carried out by the users of the said collector. It was also confirmed that concentrations at the same site could be different depending on the interval and sampling time. The wastewater was found to contain inorganic micropollutants such as heavy metals and organic micropollutants, which have a negative health and environmental impact through the contamination of aquatic environments. These micropollutants originated from domestic sources such as drugs, cosmetics, detergents, pesticides, batteries, batteries and surfactants. Since hydrophobic micropollutants such as heavy micropollutants are preferentially stored in the sediments, they represent a direct risk for organisms living or feeding in the sediments as well as the higher organisms feeding on them.

These pollutants present proven or suspected harmful effects, although their concentrations in samples from Lake Tanganyika are slightly lower than those in the collector effluents. These values are higher than those reported in literature Chale, F. (2002), for which Cu < 0.006 ppm;

Pb is 0.007 and Zn < 0.00. In addition, our results are lower than the results Maurya et al. (2019) whose metal concentrations are slightly higher than the effluents of the wastewater treatment plants of Jaganpur and are respectively Pb, 1480 ppb; Zn, 1159 ppb; Cd, 119 ppb; Co, 163 ppb and Cr 123 ppb whereby it was also observed that the concentrations of Cu, Zn and Cd were below the detection limit. The values in Table 3.4 describe the interaction, during 4 days, between the fish and its polluted environment in the aquariums. Some of the fish died in some aquariums, and those that survived were killed in order to determine the concentration the heavy metals.

Table 3.5. Results of analysis of fish in aquariums with different concentrations, in $\mu\text{g} / \text{kg}$

Conc aquarium	Cr	Co	Ni	Cu	Zn	As	Sn	Sb	Hg	Pb
Lake water	0.21	0.13	0.21	0.54	<u>21.13</u>	0.18	0.06	0.01	0.11	0.49
10 %	0.15	0.05	0.28	<u>4.35</u>	<u>21.03</u>	0.08	0.01	0.01	0.08	<u>0.16</u>
20 %	0.37	0.12	0.25	<u>0.95</u>	<u>15.5</u>	0.15	0.01	0.01	0.01	<u>0.2</u>
30 %	0.27	0.17	0.17	0.28	9.49	0.22	0	0	0.01	<u>0.11</u>
40 %	0.36	0.39	0.28	<u>0.81</u>	<u>13.6</u>	0.2	0.01	0	0	<u>0.2</u>
50 %	0.42	0.16	0.25	0.57	<u>14.31</u>	0.33	0.02	0	0.02	<u>0.23</u>
60 %	0.24	0.09	0.19	0.48	<u>14.17</u>	0.26	0.02	0	0.02	0.28
70 %	0.13	0.1	0.29	0.57	<u>14.89</u>	0.21	0.02	0	0.02	<u>0.2</u>
80 %	0.24	0.11	0.62	0.64	<u>14.54</u>	0.24	0.01	0	0.03	0.2
90 %	0.1	0.06	0.09	0.21	<u>12.47</u>	0.19	0.01	0	0.03	<u>0.13</u>
WW	0.24	0.08	0.16	0.31	<u>13.55</u>	0.25	0	0	0.06	<u>0.21</u>
Normes OMS mg.l^{-1}				30		0.1			0.45	0.5

Wastewater: dilution with x% of wastewater

The underlined values in Table 3.5, are relatively high values of heavy metals concentrations in the fish after a 96 hour-stay in the various aquariums. For the aquariums in Table 3.3, these values were lower in the wastewater. The effect of bioaccumulation of these metals by fish was observed. The non-underlined values in Table 3.3, were higher in the aquariums but not bioconcentrated by the fish. This is because of a regulatory mechanism for some heavy metals in the fish as an organism. Bioaccumulation is illustrated by the variation in the concentration of the pollutant in the bioindicators used (fish) at different concentrations; bioregulation is illustrated by the non-significant variation of the pollutant in the bioindicators exposed to different concentrations. In short, when the concentration of the pollutant in the exposed organism varies proportionally with the different concentrations tested, there is

bioaccumulation and the opposite is bioregulation. Table 3.5 equally made it possible to assess the case fatality rate from wastewater in aquariums, which could be used for the determination of the LC50. It is necessary to emphasize that some heavy metals were bioaccumulated in fish while others were stable. The death of fish could be not only due to the presence of metals but also due to other organic pollutants. A wastewater toxicity test study on *Oreochromis niloticus* was conducted in the aquariums and the results are shown in Table 3.6.

Table 3.6. Description of acute toxicity test on *Oreochromis Niloticus* in aquaria expressed in mg.kg^{-1}

Species		
<i>Oreochromis Niloticus</i>		
100% concentrated wastewater		Wastewater diluted at 50 %
ETM	X $\pm$ SD	X $\pm$ SD
Al	707.25 $\pm$ 419.3	30.33 $\pm$ 15.69
Cr	1.5 $\pm$ 0.57	0.33 $\pm$ 0.57
Mn	49.5 $\pm$ 17.59	28.33 $\pm$ 19.55
Ni	2.5 $\pm$ 0.57	2 $\pm$ 1
Cu	3.7 $\pm$ 0.5	2.66 $\pm$ 0.57
Zn	53.75 $\pm$ 20.48	50 $\pm$ 19
Pd	0.34 $\pm$ 0.17	0.033 $\pm$ 0.02
Ag	0.062 $\pm$ 0.060	0.013 $\pm$ 0.0057
Cd	0.022 $\pm$ 0.026	0.02 $\pm$ 0.025
Pb	0.76 $\pm$ 0.16	0.22 $\pm$ 0.026

After a stay of 24 hours in the aquaria of the HRC containing raw concentrated and diluted (to 50%) wastewater, the *Oreochromis niloticus* species of the *Cichlidae* family, Order, *Perciformes*, captured in Nyangara Pond developed *Ectofungus* all over the skin. Investigations revealed that the fish accumulated heavy metals in their ecosystems thus an improvement to existing results. Chale et al. (2002) carried out a research on *Oreochromis* from Lake Tanganyika and obtained the following values: Cu: 5; Mn: 0.9; Zn: 28; Pb: 2.7 and Cd 0.2 $\mu\text{g/g}$ in the fish sampled at Utinta. For those sampled at Kirando, he reported Cu 3.5; Mn: 1.6; Zn: 58; Pb: 6.1 and Cd 0.2 $\mu\text{g/g}$. These results clearly demonstrate the risk of discharging wastewater directly into Lake Tanganyika, as it has harmful consequences on biodiversity.

The results in Table 3.7, which made it possible to assess the lethal levels of wastewater in the aquariums, were used to determine the LC50 on the basis of daily deaths. The LC50 curves are shown in Figure 3.1. The median lethal concentration (LC50) is a quantitative indicator of the toxicity of a substance. This indicator measures the dose of a substance causing the death of

50% of a given animal population under specific experimental conditions, usually during acute exposure.

Table 3.7. Lethal dose (LC50)

Concentration	# Deaths				# Living	% Living
	1 st day	2 nd day	3 rd day	4 th		
10 ⁰	20	-	0	0	0	0
10 ⁻¹	18	2	0	0	0	0
10 ⁻²	5	15	0	0	0	0
10 ⁻³	0	0	0	0	20	100
10 ⁻⁴	0	0	0	0	20	100
10 ⁻⁵	0	0	0	0	20	100
10 ⁻⁶	0	0	0	0	20	100
10 ⁻⁷	0	0	0	0	20	100
10 ⁻⁸	0	0	0	0	20	100
10 ⁻⁹	0	0	0	0	20	100
Witnesses	0	0	0	0	20	100

With regards to the curve presented in Figure 3.1, it was noticed that all the twenty fishes studied were dead on the first day in the raw sewage tank (100% concentrated). After 24 hours, the deaths were observed in the aquariums diluted to 10⁻¹ and 10⁻². There were no deaths in aquariums diluted from 10⁻⁹ to 10⁻³ wastewater during the 4 days. However, several observations relating to the behavior of fish have been reported, including the appearance of sores infected and fungi all over the body. These pollutants damaged the fins of the fish and the swimming movement was significantly reduced. There was also exophthalmos and the presence of trematode in the aquarium containing the concentrated wastewater. The LC50 of the acute toxicity test carried out over 4 days corresponded to 0.0055 ml/ml. The LC100 was observed in raw wastewater, after a few hours which explains why the effluent is very toxic.

The values underlined in Table 3.5, are the bioaccumulated by the fish during 4 days in the wastewater of the aquariums. Zn and Pb are bioaccumulated while with Cu, there is more control from the aquarium by 50 to 100%. The fishes had also succeeded to stabilize metal ions such as Cr, Co, Ni, As, Sn, Sb and Hg, found in the control aquarium of Lake Tanganyika at 100%. Zinc is an essential trace element for human health. It is important for the physiological functions of living tissue and regulates many biochemical processes.

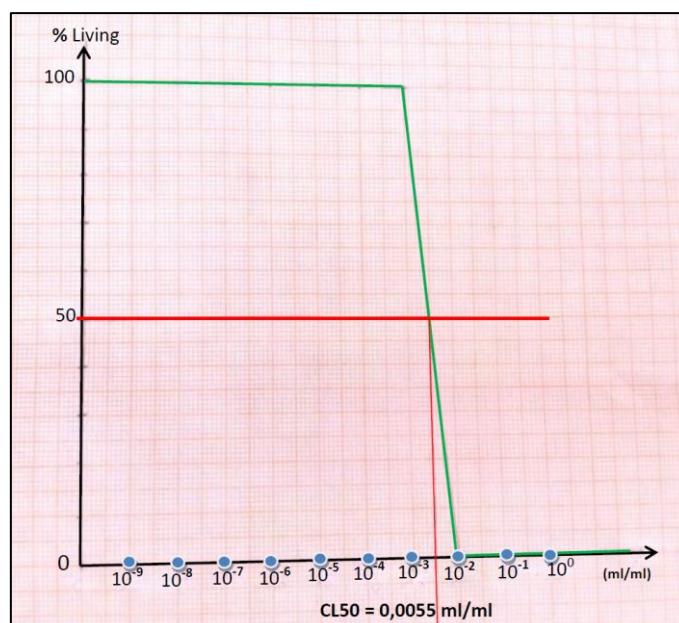


Figure 3.1. Determination of LC50

However, excess zinc in the human body can cause prominent health problems such as stomach cramps, irritation, vomiting, nausea and anemia (Oyaro et al., 2007). Copper plays an essential role in animal metabolism but its excessive ingestion leads to serious toxicological problems such as vomiting, cramps, convulsions and even death (Paulino et al., 2006). The direct effects of these metals in fish are not yet well understood. In the present study, the toxicological effects of the stable metallic trace elements Hg and Pb were investigated on samples obtained from 5 different sites and the results are presented in Table 3.5.

Table 3.8. Monitoring of hazardous metallic trace elements in the collector

	Hg ppb			Pb ppm			p-value
	$\bar{X} \pm SD$	Mim	Max	$\bar{X} \pm SD$	Min	Max	
CP	17.07 ± 0.82	16.032	18.007	0.41 ± 0.16	0.245	0.612	PC & GRH = 0.24
GRH	13.57 ± 4.90	6.726	18.367	0.51 ± 0.19	0.379	0.67	PC & GRH = 0.05
HW	15.3 ± 1.36	13.64	18.007	0.37 ± 0.3	0.009	0.6	HW & Lac = 0.38
LT	10.37 ± 4.77	6.801	17.415	0.3 ± 0.2	0.188	0.61	PC & TL = 0.08

* $p < 0.001$: Highly significant, ** $p < 0.01$: very significant and *** $p < 0.05$: significant.

According to the values in Table 3.8, the concentrations of Pb and Hg vary from one site to another. Evidently, the concentrations are very high at the 4 sampling sites of the collector. It is noted that these concentrations were also high in Lake Tanganyika. Among the sites investigated, the statistical difference was significant only at the central prison and HW sites, with $p = 0.05$. Sources of Lead in the environment include foundries, metallurgy industries, coal combustion, waste incineration, batteries, electrical devices, biocides, plastics, boxes of

cans contaminated with old lead pipes and certain containers glazed with lead or lead-tin alloy (false “tins”) (Sindy, 2006; Lamprea, 2009).

3.3.2. Organic Micropollutants

The wastewater from this sewer contains not only inorganic micropollutants, but also micropollutants and organic compounds that contain a number of toxic substances and cause fish deaths. These compounds are usually present in concentrations of the order of ppb ($=\mu\text{g/L}$). Table 3.9 presents the concentrations (ppb) of VOCs in the wastewater obtained from the GRH of Uvira, the mixing of the GRH, the PC and the HW which are connected to the collector and the coast of Lake Tanganyika at the outlet of the collector.

Table 3.9. Concentrations in ppb of organic compounds in wastewater and Lake Tanganyika.

Compound	GRH	GRH, PC, HW	LT	Standard
Benzene	<0.05	<0.05	<0.05	10^{-4}
Toluene	0.53	0.678	0.341	10^{-4}
Ethylbenzene	0.065	0.071	<0.05	10^{-4}
m-Xylene/p-Xylene	0.067	0.051	0.054	2.10^{-4}
o-Xylene	0.1	<0.05	<0.05	2.10^{-4}
Chloroform	0.498	0.983	<0.05	10^{-5}
Bromodichloromethane	<0.05	<0.05	<0.05	-
1,1-Dichloroethane	<0.05	<0.05	<0.05	6.10^{-6}

Generally, WW and hospital effluent contain several organic micropollutants such as: hydrocarbons, polychlorinated biphenyls (PCB), polybrominated biphenyls (PBB), polychlorinated dibenzodioxins (PCDD), polychlorinated dibenzofurans (PCDF), detergents (or surfactants), phthalate esters, various drug substances, but these were not detected in the present study. Perhaps high-performance laboratory equipment was required for their detection. Nevertheless, toluene, ethylbenzene, m-xylene/p-xylene, o-xylene and chloroform were detected at concentrations higher than the recommended standard. Benzene, bromodichloroethane, and 1,1-dichloroethane were present in the samples but in concentrations less than 0.05 ppb, which is the limit of detection for ICP-MS. The samples collected at the mixing point of the GRH, PC and HW effluents before leaving Lake Tanganyika were found to contain: toluene, ethylbenzene, m-xylene/p-xylene and chloroform at concentrations above the standard but with no o-xylene.

According to our results, the hospital effluent was found to contain toluene, ethylbenzene, m-xylene/p-xylene, o-xylene and chloroform in higher concentrations than the standard. Moreover, Benzene, bromodichloroethane and 1,1-dichloroethane were detected in these samples, but in concentrations less than 0.05 ppb, which is the ICP-MS detection limit. It was observed that the concentrations of toluene, ethylbenzene and chloroform increased during mixing, which explains the contribution of the PC, which has a dispensary. In Lake Tanganyika, a high concentration of toluene and m-xylene/p-xylene was observed, compared to the standard, which presents a great danger to the biodiversity and the surrounding population that depends on this water for domestic purposes.

Very few studies on this topic have been published in literature. The research works carried out on the two rivers Rhine and IJsselmeer in Europe (Hofman et al., 1997; Exttoxnet, 2002; Van der Bruggen, 2002), yielded lower values than those reported in this study, in particular: 0.2 µg/L toluene, <0.1 µg/L bromodichloroethane and <0.1 µg/L chloroform in the IJsselmeer river water and <0.1 µg/L toluene, <0.1 µg/L bromodichloroethane and 0.2 µg/L chloroform for the Rhine river water. Previous studies showed that the micropollutants found in urban water (waste and rainwater) come from domestic, artisanal, industrial activities, road traffic, rain runoff water and road cleaning, maintenance practices of urban spaces (green spaces, facades, etc.) (Petrucci et al., 2014).

Some compounds have easily identifiable sources such as parabens or pharmaceutical residues which largely come from domestic uses and are restricted to wastewater or polycyclic aromatic hydrocarbons which are mainly produced by automobile traffic and are abundantly present in rainwater. On the contrary, micropollutants such as alkylphenols, have many origins linked to the multiplicity of their uses (Briand et al., 2018). Other micropollutants come from domestic cleaning products like disinfectants and body care products (Deshayes, 2015; Zedek, 2016). The probable sources of micropollutants identified in this study in the city of Uvira are degradation of wastes from the processing of agro-food products, used car parts such as lead accumulators, HW waste dumped in the environment, effluents of pharmaceutical residues and phytosanitary products into the environment.

3.4. Conclusions

This study had as main aim to determine the ecotoxicological risk of urban waste from the city of Uvira discharged into Lake Tanganyika on the northwest coast of the Democratic Republic of Congo. The study showed the presence of inorganic micropollutants at high concentration compared to the standards such as: Cr, Co, Ni, Cu, Zn, As, Sb, Hg and Pb, while Sn was found to be too low and negligible.

The presence of Pb and Hg at significant concentrations was observed at all sampling sites including the shore of Lake Tanganyika. Some organic micropollutants were identified in high concentrations except those in the samples obtained from the General Hospital of Uvira which is attributable to pharmaceutical residues.

Differences between exposed species can be explained by the age and size of exposed individuals of the same species; by their trophic level (diet) for individuals of different species and by the individual health status.

The acute ecotoxicological experiment carried out on aquariums at the Center for Research in Hydrobiology showed that about 50% of the fish population in the investigated region is likely to die at the concentration of 0.0055 ml/ml of would guarantee the purity of Lake Tanganyika. It is recommended that the population gets urgently aware of the need to protect the environment in order to improve the quality of life. It is recommended that the relevant environmental protection department is involved to evaluate the existing legislation on the protection of surface water against pollution, to update it and to ensure its strict application. Water analyses have confirmed that the wastewater from the above-mentioned sources is toxic.

Chapter 4. Evaluation of commercial reverse osmosis and nanofiltration membranes for the removal of heavy metals from surface water in the Democratic Republic of Congo

This Chapter evaluates the performance of commercial reverse osmosis (RO) and nanofiltration (NF) membranes for the removal of metal ions from synthetic water and surface water taken from the north-west of Lake Tanganyika in the city of Uvira, east of the Democratic Republic of Congo. Metal ion analyses were performed by the standardized ICP-MS and ICP-OES methods. The RO membrane showed higher metal ions rejection in high-concentration solutions (synthetic samples) prepared in the laboratory as well as in low-concentration samples from real raw water collected near Lake Tanganyika. Rejection levels were higher than 98% for Cr^{3+} , Pb^{2+} , Cd^{2+} , As^{3+} , Ni^{2+} , and Sb^{3+} ions in the synthetic solutions, and 99, 99, 99, 99, 98 and 99%, respectively, in the real samples. The concentrations of metals in the permeate varied depending on the feed concentration and were 0.15 to 1.02 mg/L, 0.33 to 22 mg/L, and 0.11 to 22 mg/L in RO, NF90, and NF270 membranes, respectively. Regarding the NF membranes, the rejection of Cr, Ni, and Cd ions was interesting: 99, 9 and 92%, respectively. However, it was lower for Pb, As, and Sb ions: 78 and 64%, respectively. The flux of NF was 329 to 375 L/m².h, much higher than for RO membranes, which had a flux of 98 to 132 L/m².h. The studied membranes are thus a feasible solution to remove the studied metals from real water sources at low concentrations since they meet the standards of the World Health Organization on specific values assigned to chemicals from industrial sources and human habitation areas where these ions are present in drinking water.

4.1. Introduction

Heavy metals are difficult to remove from the environment because they cannot be chemically or biologically degraded and are ultimately indestructible (Fu et al., 2014). The World Health Organization (WHO) produces a series of water quality guidelines covering drinking water, the safe use of wastewater, and safe bathing water (WHO 2017). Table 4.1 presents the surface and drinking water quality guidelines, the latest version updated in 2017, which are the benchmarks for the safety of drinking water.

Table 4.1. World Health Organization's quality guidelines for drinking water (WHO 2017)

Element	Concentration normally found in surface water	WHO guidelines
Sb	< 4 µg/l	0.02 mg/l
As	-	0.01 mg/l
Cd	< 1 µg/l	0.003 mg/l
Mn	-	0.1 – 0.2 mg/l
Cr ³⁺ , Cr ⁺⁶	<2 µg/l	Chrome total: 0.05 mg/l
Cu ²⁺	-	2 mg/l
Pb	-	0.01 mg/l
Hg	<0.5 µg/l	0.01 mg/l
Ni	<0.02 µg/l	0.07 mg/l
Zn	-	3 mg/l

Several processes can be used to remove metal ions from wastewater, in particular: precipitation, adsorption on activated carbon, ion exchange, membrane filtration, and hybrid systems (Barbooti et al., 2011; Chen et al., 2018; Kazemipour et al., 2008; Dabrowski et al., 2004; Radcliffe et al., 2017; Ejraei et al., 2019). Membrane separation technology is the most cost-effective and widely applied technology for water purification. Generally, the membrane acts as a selective barrier that allows for the passage of certain constituents while inhibiting the passage of other constituents based on different mechanisms depending on the type of membrane (e.g., size-exclusion, diffusion). Filtration-based membrane processes have been classified into microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), reverse osmosis (RO), and forward osmosis (FO), in which the first two often use a microporous membrane while the last three use a thin composite membrane with a selective dense layer (Hamid et al., 2019). In order to remove ions, NF and RO membranes have demonstrated good retention capacities. Boussouga et al (2020) showed that the variations in water salinity had no impact on the high rejection of As(V) in nanofiltration while the variation of pH significantly affected the rejection of As(V) and the presence of humic acid in the water increased the overall release of As(V). In the presence of humic acids, an improvement in As(V) rejection occurred with the NF270 membrane by 82-94%. At different salinity conditions (0.58 – 20 g L⁻¹), rejection of

As(V) with both NF270 and NF90 membranes showed a consistently high removal of 82–88% and 93–98%, respectively. The elimination of Sb compounds did not depend on the pH of the solution since the oxidation state of antimony changes from Sb (III) to Sb (V) in a very short time. The elimination of Sb (III) and Sb(V) is nearly constant over a pH range of 3–10 unless Sb (III) removed with the NTR-729HF membrane greatly decreases by 60% to 45.7% with a reduction in pH from 7 to 10 (Kang et al., 2000). Experiments conducted by Qdais et al (2004), show that RO and NF membranes can remove synthetic wastewater containing 250 ppm of Cu^{2+} and Cd^{2+} ions at 98% and 99% and the permeate concentration would be reduced to 3 ppm. Using NF, more than 90% of the copper ions were removed. Mohsen-Nia et al. (2006) examined the effect of increasing ion size on membrane rejection, Cu^{2+} and Ni^{2+} ions were chelated by the disodium salt of ethylenediaminetetraacetic acid (Na_2EDTA), a commonly used chelating agent. The results obtained indicate that Na_2EDTA can increase the size of the chelated Cu^{2+} and Ni^{2+} ions and, therefore, their rejection in the RO removal process was 99.5 %.

Ipek et al. (2005) showed that treatment of the feed solution with EDTA did not influence the removal of Zn^{2+} in NF. The removal of Ni^{2+} and Zn^{2+} ions from the untreated solution was 99.3 and 98.9%, and after treatment, 99.7 and 99.6% respectively. The recovery rate, rejection, and saturation factor all increased with increasing applied pressure. Increasing the pH improved the removal of Pb^{2+} from 86 to 99%, but did not affect the removal of Ni^{2+} from 93 - 99%; this is due to the differences in interaction between the surface of the membrane and the anion present in the solution (Jekel et al., 2006). The rejection of Ni^{2+} and Cr^{6+} ions increased with increasing pH but did not change significantly by transmembrane pressure difference (10, 20 and 30 bar) for NF90 and NF270 membranes. The optimum conditions were found at 30 bars with a pH of 10 for the NF90 and NF270 membranes. Under optimal conditions for the NF90 membrane, the Ni^{2+} and Cr^{6+} rejection values were 99.2 and 96.5%, respectively. For the NF270 membrane, the Ni^{2+} and Cr^{6+} rejection values were 98.7 and 95.7%, respectively (Basaran et al., 2016). Lead ion rejection was greater than 98%, and increased slightly with increasing crossflow velocity and operating pressure and decreased slightly with increasing feed metal concentration. The rejection and permeate flux decreased slightly and increased sharply, respectively, with increasing feed pH (Basaran et al., 2015). The elimination of Ni^{2+} , Pb^{2+} , and Cu^{2+} ions from synthetic water using nanofiltration was 85%, 78%, and 66%, respectively, under specific conditions of concentration, pH and temperature (Gherasim et al., 2014). The pH of the solution and feed concentration has a significant impact on the separation of metals, and the pressure plays a different role on the rejection and permeability of metals, depending on the kind of membrane. According to solution diffusion theory, water and solute molecules diffuse through

the dense release layer (Algueiri et al., 2016). The rapid diffusion of water molecules and slow diffusion of solute molecules result in increased rejection; meanwhile, the slow diffusion of water molecules and rapid diffusion of solute molecules result in a decreased rejection. The water flux also has a common dilution effect.

Given the fact that much work has been carried out in RO and NF on the rejection of three ions at maximal in solution by adjusting the pH of the feed solution and a chemical pretreatment before filtration, the present study presents a two-fold objective: first, the evaluation of the performance of several commercial membranes in reverse osmosis and nanofiltration in terms of retention, flow rate, and permeability, applied to synthetic water containing six different ions; and second, the assessment of the retention of several ions in real waters without prior pretreatment, which is a source of pollution to Lake Tanganyika. There are several types of commercial membranes, and the specific choice of RO X-20TM, NF90 and NF270 was motivated by the promising performance of these membranes for the removal of metals from water (Chai et al., 2019).

4.2. Methodology

4.2.1. Sampling site of real samples

Real water samples were taken at the discharge collector located at the point indicated in Figure 2.1. This is the last point after the households and before the real water enters the lake, and where we sampled the maximum amount of real water emitted with the coordinates 029°08'39.0" of longitude N and 03°24'26.3" of latitude E in the city of Uvira, in the Democratic Republic of Congo, flowing northwest into Lake Tanganyika. These samples were first filtered on Whatman MN 615. Φ 125 mm REF 431 012 filter paper model to retain large particles in solution, before being filtered through RO and NF membranes in the laboratory.

A chemical analysis was made to determine the concentration of each element in the actual wastewater (shown in Table 4.2). The initial concentrations were determined by IPC-MS and OES, measured in ppb and ppm. ICP-MS was used to detect low-concentration metals in the permeate and real waters and ICP-OES was used to determine toxic metal ions in high concentrations in synthetic waters. Real samples were filtered before being introduced in the membrane crossflow device. Surprisingly, the initial concentration before filtration was higher than after filtration, which may be due to the adsorption of metals on the removed particles. In Table 4.2, the measured concentrations before and after filtration are shown.

Table 4.2. Ion concentration in the real water sample.

Elements	Concentration Before Filtration (ppm) *	Concentration After Filtration (ppb) *
Co	<0.05	28.13
Al	0.155	<1
As	<0.05	6.36
Ba	0.085	16.96
Cd	<0.05	<0.05
Cr	0.06	<0.05
Cu	<0.05	5.66
Fe	0.815	23
Mn	0.47	337.27
Ni	0.03	6.25
Pb	<0.05	<0.05
Sb	<0.05	1.57
Sn	<0.05	<0.05
Ti	<0.05	26.33
W	<0.05	5.27
Zn	<0.05	184.26
Ca	40	47,390
K	66.5	53,920
Mg	15	16,900
Na	78	930
Si	16	18,700

* ICP-OES was used to determine the concentrations before filtration (in ppm) and ICP-MS was used to measure the concentration after filtration (in ppb).

Table 4.3. Specifications of the membranes used in the experiments (information provided by the supplier).

Characteristics	RO X-20™ Membrane	NF90 Membrane	NF270 Membrane
Allowable operating pH range	4–11 Continuous	NA	NA
Maximum operating temperature, °C	NA	45	45
Maximum feed turbidity, NTU	2	NA	NA
Maximum feed SDI, 15 min	5	NA	NA
Average salt discharge (%)	99.5	97	NA
Minimum salt discharge (%)	98.5	NA	NA
Filtration pressure (bar)	NA	41	41

NA: not available.

4.2.2. Synthetic samples

In order to determine the membrane performance under different metal concentrations in water, synthetic solutions were prepared by adding specific amounts of metals, simulating high-concentration effluents (i.e., industrial effluents) and low-concentration effluents (i.e., water in natural water bodies) of heavy metals (chromium, arsenic, nickel, antimony, lead and cadmium). Synthetic water was chosen to test the membranes because of its high concentration, which is similar to raw water from the plant. The low-concentration solutions had 15 ppm, and the high-concentration solutions had 56 ppm; the solutions were freshly prepared from an analytical-grade standard solution (Sigma Aldrich, Merck KGaA 64,271 Darmstadt, Germany) containing 1000 mg L⁻¹ of As³⁺, Cr³⁺, Pb²⁺, Cd²⁺, Ni²⁺, and Sb³⁺. A standard solution (Certipur®) was used to prepare the stock solutions.

4.2.3. Membrane cross-flow system setup

The Armfield FT17 lab-scale device was used for evaluating the performance of membranes in a crossflow filtration application. This device enables rapid determination of crossflow filtration performance using a range of membrane types with relatively small sample volumes (1 L). Figure 4.1 shows the schema of the experimental set-up. Both prefiltrated real samples (Section 4.2.1) and synthetic solutions (Section 4.2.2) were used as the feed solution in the crossflow setup.

The solution to be treated is poured into the 1L capacity 316 stainless steel feed container. The vessel is fitted with a jacket to allow cooling, as repeated circulation of the retentate through the high-pressure feed pump causes a significant rise in liquid temperature of between 5 and 60°C and can damage the membrane. An optional chiller (FT17-15) can be attached to the jacket or the user can attach his own chiller supply.

The feed is recirculated through the filter by a triple piston feed pump. Filtration pressure is generated using the sprung back pressure (V2) and is shown by the pressure gauge. Permeate is collected in the beaker. In the basic system the feed pump runs at fixed speed with a simple on/off switch.

V1 is a stainless steel pressure relief valve set to relieve at a little above 40 bar. The filter assembly consists of two basic part made from 316 stainless steel. There is an inlet and exit port for the retentate in the top part. Machined into underside of the top part is a spiral fluid flow path. This directs the retentate flow across the filter surface below. Two basic types of filter may be fitted. The basic system may be used with thin polymer membranes with cut-offs

ranging from MF through to RO. T1, P1, high level, low level, permeate mass are obtained with the FT17-50 option.

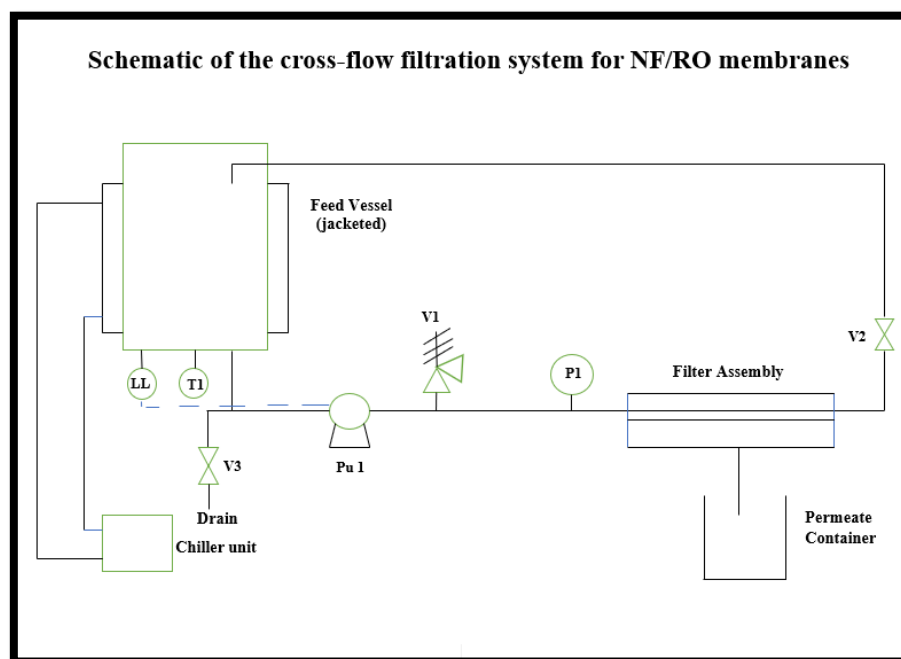


Fig 4.1. shows the schema of the experimental setup.

Legend: PI: pressure indicator (left); LL: low-level sensor (pump protection); LH: high level (vessel over-filling protection); P1: pressure sensor; T1: temperature sensor; V3, V2, V1: Valves

4.2.4 Commercial membranes

The membranes used in this work are the polyamide urea RO X-20TM, NF90, and NF270, supplied by Lenntech Water Treatment Solutions (Distributieweg 3, 2645 EG, Delfgauw, The Netherlands). The selection of these membranes for this study was based on average salt removal. The specifications of these membranes are presented in Table 4.3.

4.2.5 Experimental procedure

Before the experiment, the membranes were soaked in ultrapure water for 30 min. The membranes were then inserted into the crossflow RO setup to filter the samples at different pressures, notably 50 bar for RO and 38 bar for NF. The choice of this pressure is justified by the characteristics of the nanofiltration membrane, which can be operated up to a maximum pressure of 41 bar. Referring to the literature (Maher et al., 2014), the pressure of 10, 20, and 30 bars did not significantly change the rejection at nanofiltration.

The prefiltrated real water (see Table 4.2) and synthetic solutions were filtered in the RO unit. The feed capacity was 1/2 L at a constant maximum pressure of 50 bar in RO and 38 bar in NF. The temperature varied from 299.14 K for ions concentrated at 15–26 ppm and 303.14 K for

ions concentrated at 56 ppm in RO, and in nanofiltration was 296.14 K for ions 15–26 ppm at 297.14 K for 56 ppm concentration. After filtration, the permeate and retentate of the real waters, because of their low feed concentrations, were analyzed by the standardized method ISO 17294-1 and ISO 17294-2 with an inductively coupled plasma mass spectrometer (ICP-MS), model NexION 5000 Multi-Quadrupole ICP Mass Spectrometer, provided by PerkinElmer®, and inductively coupled plasma optical emission spectroscopy (ICP-OES) provided by Agilent Technology was used for the synthetic solutions because of their higher concentrations. This analytical technique allows for the identification of the atomic composition of a particular sample. It uses the unique photophysical signals of each element to successfully detect the type and relative amount of each element within the complexity of a compound. From values of composition in the retentate and permeate (C_r and C_p , respectively), the retention was calculated as indicated in Table 4.4. In addition, the permeation flux, J , and the permeability, L_p , were obtained using the equations also listed in Table 4.4. In this work, the osmotic pressure was 0.026 bar, leading to a driving pressure (transmembrane pressure) of 48.96 bar.

Table 4.4. Main parameters used to evaluate the performance of RO and NF membranes.

Parameter	Formula	Equation	Ref
Retention (%)	$R = \left(1 - \frac{C_p}{C_r}\right) \times 100 \%$	(7)	Luis et al.
where C_p and C_r are the permeate and retentate	$(C.V)_f = (C.V)_r + (C.V)_p$		(2018)
Mass balance calculation			
Permeation flux	$J = \frac{Q_p}{S}$	(8)	Luis et al.
flux (J)			(2018)
($L \cdot m^{-2} \cdot h^{-1}$)	where Q_p is the permeate flow rate ($L \cdot h^{-1}$), S (0.00664 m^2) is the membrane area		
Driving pressure DP	$\Delta P = \Delta P_{r-p} - \Delta \pi$	(9)	Van der Bruggen et al.,
$\Delta \pi$ is the osmotic pressure gradient (bar)	$\pi = RTC$		(2018)
C is the total concentration of ions ($molL^{-1}$)	$C = \frac{n}{V}$		
	With n as the total number of moles of ions		
$R = 0.082 \text{ L atm K}^{-1} \text{ mol}^{-1}$			
T (K)			

$$Lp = \frac{J}{\Delta P} \quad (10) \quad \text{Luis et al. (2018)}$$

Membrane Permeability

Lp is the solvent (water) permeability

($\text{L m}^{-2} \text{h}^{-1} \text{bar}^{-1}$)

4.3. Results and Discussion

4.3.1. Effect of the Metals Feed Concentration on the Membrane Performance

The removal of ions by the studied membranes is shown in Figure 4.2. using synthetic solutions with 15, 26, and 56 ppm of metals. The synthetic solutions were filtered under a pressure of 50 bar at 303.14 K for the reverse osmosis membrane, and 38 bar at 297.14 K when using the nanofiltration membranes.

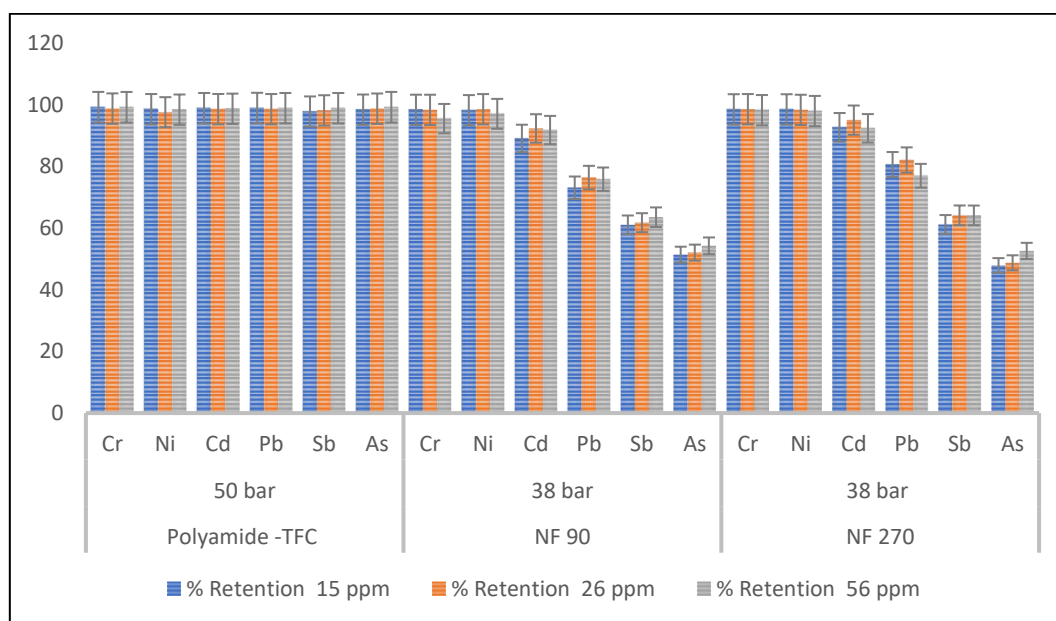


Figure 4.2. Removal of heavy metals as a function of feed concentration.

The results showed that the retention of Cr^{3+} , Cd^{2+} , Ni^{2+} , Pb^{2+} , Sb^{3+} , and As^{3+} ions was $> 98\%$ (99, 99, 99, 99, 98, and 99 %, respectively) independently of the concentration for the reverse osmosis membrane.

It was observed that the rejection of Cr^{3+} is similar to that of Ni^{2+} , although the size of Cr^{3+} is larger than that of Ni^{2+} . These two ions are larger than Cd^{2+} , Pb^{2+} , and Sb^{3+} , of which the sizes are the same. Removal of antimony was found to be $< 65\%$ and arsenic $< 55\%$ in nanofiltration. The RO membrane gave much higher ion rejection than the nanofiltration membranes. The rejection of ions by the NF 90 membrane was almost at the same level as that at NF270, except

for the 56 ppm feed solution, which was higher. Therefore, it was considered that size exclusion is not the only key factor in ion rejection. However, NF270 gave a lower ion rejection than NF90 due to its larger pore size and also due to the interaction with the membrane. The t-test on single samples shows that the difference is not significant between NF90 and NF270 (p-value: 0.6 to 0.8) for all concentrations, but it is highly significant between RO and NF (p-value: 0.001 to 0.0001). The structure of the RO and NF membranes would be the factor to influence the p-value. Very few studies have been conducted on the removal of antimony by membrane processes, unlike arsenic. Kang et al. (2000), achieved a rejection of 60% Sb (III) and 95% Sb (V) by reverse osmosis using the ES-10 membrane, and, 45 - 60% Sb (III) and 95% Sb (V) rejection using the NTR-729HF membrane. These values are lower than our research results using the reverse osmosis membrane.

Previous studies showed that 99% rejection As (V) could easily be achieved with NF and RO membranes. The Pb retention is low and ranges from 73 to 82% using nanofiltration. However, despite this difference in sizes between the ions, the retention is very high and is almost similar in reverse osmosis, due to the interaction between the ions. Similar results have been reported by Cséfalvay et al (2009); Chan et al (2008), who mentioned that Cd^{2+} can be removed at a slightly higher efficiency compared to Cu^{2+} .

In recent years (Murthy et al., 2009) many works have been devoted to the removal of heavy metal ions using nanofiltration. It was reported the application of a thin-film composite nanofiltration polyamide membrane for the rejection of nickel ions from aqueous wastewater (Bruggen et al., 2002). It was observed a nickel rejection of 98% and 92% for a feed concentration of 5 and 250 mg/L, respectively, and they studied the binary separation capacity of heavy metals (cadmium and nickel) of a commercial NF membrane starting from solutions (Seidl et al., 2001). The maximum solute rejection observed for nickel and cadmium ions was 99% and 83%, respectively, for an initial feed concentration of 5 mg/L. The nickel rejection is similar to our results, unlike cadmium, which varies between 89% to 95% and it is greater than their result. This difference would be due to the initial concentration and the pressure applied. Household and industrial wastewaters contain other heavy metal ions at various concentrations. Several research studies have been conducted on the removal of As (III) and (V) by NF (Van der Bruggen et al., 2003), and the removal of As (V) and As (III) was comparable. The low rejection of As (III) suggests that size exclusion governed their separation behavior and not charge interaction (Van der Bruggen et al., 2003). Boussouga et al (2020) confirmed the efficiency of NF to remove As (V), unlike As (III), which is much more difficult to remove, except when tight-pore NF membranes are used. Furthermore, NF membranes can only reject

<50% of As (III) at $\text{pH} \leq 8$ (Seidel et al., 2001; Oh et al., 2004; Worou et al., 2021). Sato et al. (2002), Sen et al. (2010), and Tay et al. (2002) recommended pretreatment by oxidation prior to filtration to improve As (III) rejection. These results highlight the role of charge interactions between the membrane and ions in determining the rejection performance of NF membranes, particularly for As species (Sen et al., 2010). The X-20TM type TFC PA RO membrane was efficient in removing heavy metals (at low- or high-feed solution concentration), but the permeate flux was low and the applied pressure was very high. The nanofiltration membranes showed good ion rejection at low concentrations and the permeate flux was very high, but the retention was low for high-concentration feed solutions. Many low-income countries have very limited energy resources; consequently, the application of nanofiltration becomes relevant to lift this heavy metal challenge. On the other hand, despite the excessive energy consumption in RO, it still has many advantages to removing inorganic, organic, and microplastic micropollutants (Abolfathi et al., 2020).

4.3.2. Ion removal in real waters

It was observed that the retention of ions in the real wastewater, filtered by reverse osmosis, at 50 bar pressure at 303.14 K, for Co, Al, As, Ba, Cr, Cu, Ni, Sb, Sn, Ti and Zn ions was 100% and for Fe, Mn, W ions, was 95, 99 and 89, respectively.

The retention of Co, Al, Ba, Cr, Cu, and Sn ions was also 100% by NF90 and NF270 membranes, while for As, Fe, Mn, Ni, Ti and Zn ions, it was 99.8, 82, 88, 95, 99, 97% and 98, 90, 92, 99, 99, 96%, respectively. Tungsten was not retained by NF90 but NF270 had retained it by 27%. Cd, Pb, and Sn were below the ICP-MS detection limit.

The complete elimination of toxic ions and low level of heavy metal concentration in the permeate, below the WHO standard, means that good-quality water can be obtained. Figure 4.3 below, shows the ion retention of real waters.

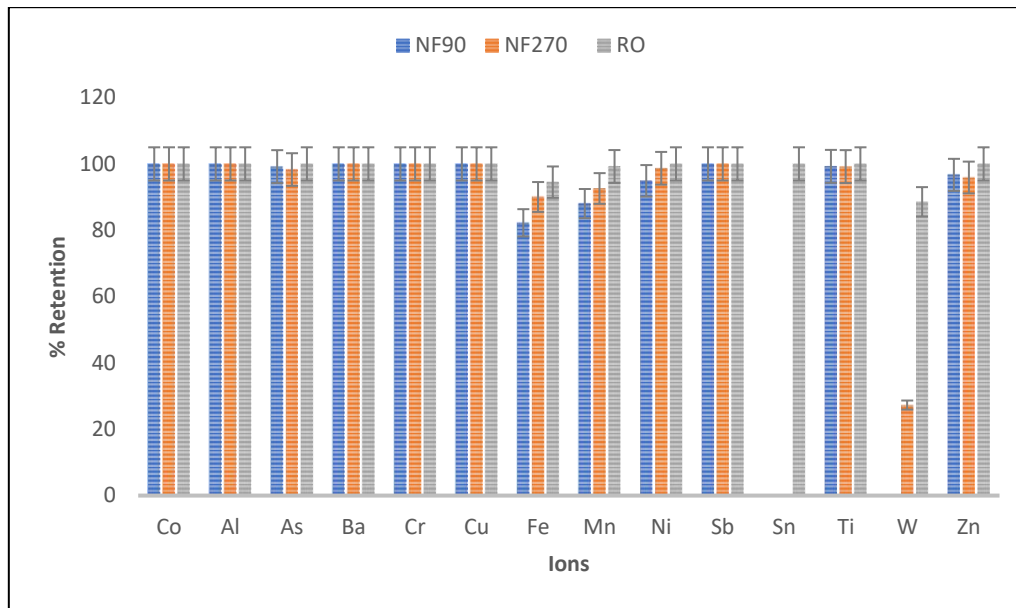


Fig 4.3. Retention of charged ions in real waters, analysis by ICP-MS

The filtration of real wastewater with RO and NF eliminated the ion concentration to the WHO standard, as shown in Table 4.5.

Table 4.5. Ions concentrations in permeate

Elements	Ions Conc (ppb)		
	NF90	NF270	RO
Co	0	0	0
Al	0	0	0.00
As	0.30	0.36	0,00
Ba	0	0	0
Cd	< 0.05	< 0.05	< 0.05
Cr	0	0	0
Cu	0	0	0
Fe	64.66	17.96	6.54
Mn	78.10	90.70	0
Ni	1.30	0.91	0
Pb	< 0.05	< 0.05	0
Sb	0	0	0
Sn	< 0.05	< 0.05	0
Ti	0.33	0.41	0
W	5.06	3.80	1.51
Zn	12.90	17.85	0.00

4.3.3. Comparison of real and synthetic water retention

Figure 4.4 shows the results of retention of the RO and NF membranes for real and mimicked surface water. It is observed that the removal of metals from the RW was higher compared to the synthetic solution. In real water, under the same feed pressures of 50 bars at 303.14 K in

RO and 38 bars at 297.14 K in NF, the Cr and Sb ions were completely eliminated from the permeate filtered by the 3 membranes. The rejection of Ni^{2+} and As^{3+} ions in NF90 was 99% and 99%, and for NF270, it was 99% and 98%, respectively. The Cd^{2+} and Pb^{2+} values in the permeate were below the detection limit by ICP-MS. No trace of these ions had been identified in the permeate after filtration by reverse osmosis. The retention of Ni^{2+} by NF90 (99%) agreed with the results obtained by Basaran et al (2016), for the same membrane.

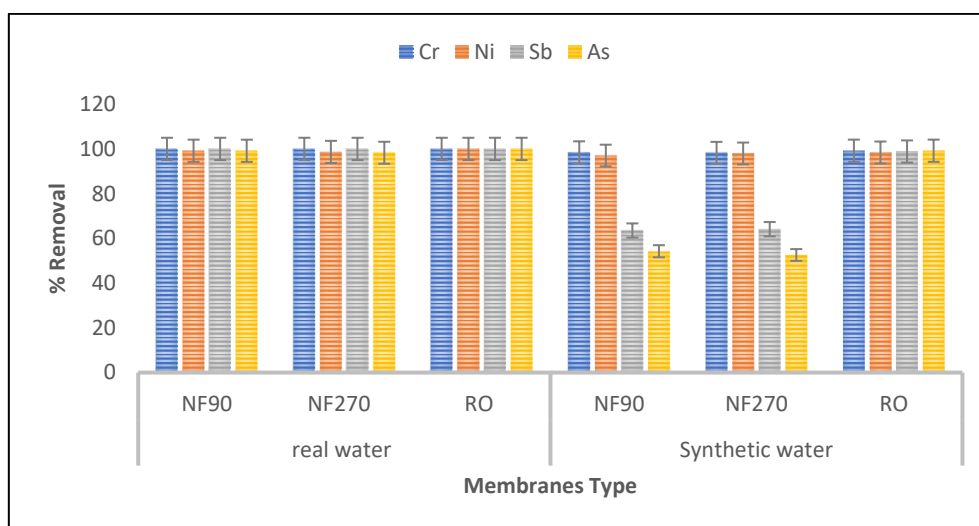


Figure 4.4. Comparison of % removal between synthetic waters and real water.

On the other hand, for the synthetic solution, the rejection of Cr^{3+} , Ni^{2+} , Sb^{3+} and As^{3+} ions were 99, 97, 64% and 54%, respectively, for NF90, followed by 98, 98, 64 %, and 53% for NF270 and finally 99, 98, 99 %, and 99% for RO. Those values are the same as the ones obtained for RW except As^{3+} and Sb^{3+} .

The high rejection of the real waters can be explained by the low concentration of the load in ppb, unlike the concentration of the synthetic solution, which was 56 ppm. Lower concentrations in the feed thus involve higher rejection. This is in agreement with the results obtained by Shigidi et al. (2022), whereby the removal of ions at 99.8% was possible in a 5 ppm feed solution and decreased to 94% in a 30 ppm feed solution and finally to 77% for the 100 ppm feed solution. Boussanga et al (2021) found that the presence of humic acids in RW can influence the removal of ions. The difference in metal ion removal between reverse osmosis and nanofiltration membranes can be explained by the porosity of NF membranes, which varies from 1 to 2 nanometers, whereas RO membranes are dense with pore sizes of less than nanometer.

Table 4.6. reviews the developments and potential applications related to nanofiltration and reverse osmosis, with a particular focus on heavy metal removal. Results from the present study

show that the application of RO without chemical pretreatment achieved a more superior removal of toxic metal ions than that obtained by Chan et al. (2008), Algureiri et al. (2016), and Shigidi et al. (2021), and was similar to other works, such as Ipek et al (2005) and Mohsen-Nia et al. (2007), whose feed solutions were chemically treated. Removal of Cr, Ni, and Cd ions was excellent in nanofiltration and like previously treated feed solutions.

Table 4.6. List of key studies on heavy metal removal by using reverse osmosis and nanofiltration.

Type	Membrane	Element	Initial Conc	Type of Water	Removal Efficiency (%)	Conditions	Conc Permeate	Ref
NF	NF270 NF90	As (V)	250 µg/L	Synthetic	82–88 93–98	Feed pressure of 20 bars		Youssef et al., 2021
RO	ES-10 NTR-729Hf	As III Sb III As V Sb V As III Sb III As V Sb V	50 µg/L	Drinking water	75 60 95 95 20–43 45.7–60.2 80–95 95	0.75 MPa	ND	Kang et al., 2000
RO NF	Polyamide	Cd ²⁺	250 ppm	Synthetic	98.5 82–97	ND	3.2 ppm 13.16 ppm	Qdais et al., 2004
RO		Ni ²⁺ Cu II	500 mg/l		99.5	Operation pressure 5 atm	2.01 ppm	Mohsen-Nia et al., 2007
RO		As III As V	<500 µg/L		20–55 91–99	3 atm	ND	Chan et al., 2008
RO		Ni ²⁺	44–169 mg/L		99.3	Operational pressure 1100 kPa		Ipek et al., 2005
NF	NF70	As III As V As III As V	ND		99 99 <30 60–90	ND	ND	Jekel et al., 2006
NF	NF90	As V	100–1000 ppb	Synthetic	>94	2-12 bars	<10	Figoli et al., 2010
NF	Polyamide	Pb II Ni II Cu II Pb II Mn II Ni II Zn II	1 ppm	Drinking water	86 93 98.72 99.61 99.31 99.11 99.51	0.75 MPa	ND	Maher et al., 2014
NF	NF90 NF270	Ni II Cr VI Ni II Cr VI		Wastewater	99.2 96.5 98.7 95.7	10, 20, and 30 bars	ND	Basaran et al., 2015
NF	AFC 80	Pb II Pb II Cd	5–250 mg/L 15 mg/L 5 mg/L	Synthetic Wastewater	>98 >99 >99	25 bars	<0.05 ppm	Gherasim et al., 2014
RO		Ni II Cu II Pb II		Synthetic	98.5 97.5 96	1 and 4 bars	ND	Algureiri et al., 2016
NF		Ni II Cu II	50 ppm 100 ppm		85 66	1, 2, 3 and 4 bars		

RO NF	TFC PA	Pb II	150–200 ppm	Synthetic	78	Operating pressures 35 bar	ND	Cséfalvay et al., 2009			
		Cu II	2 g/L		>95						
		Co II	39.4		98.6						
RO	TFC PA	Ni II	214.9	Synthetic	98.1	41 bars	ND	Ricci et al., 2017			
Mg II		242.9	98.6								
RO		Pb II	0.034	Real water	<1 ppb	1.76 MPa	<1 ppb	Thaçi, et al., 2019			
		Zn II	0.153		<0.002 ppb		<0.002				
		Cd II	0.025		<0.1 ppb		<0.1 ppb				
		Co II	0.018		<0.2 ppb		<0.2 ppb				
		Mn II	1.146		99.48		0.006 ppb				
		Ni II	0.004		<0.5 ppb		<0.5 ppb				
RO	BW30XFR	Cr VI	5 30 100	Synthetic	99.8 94.3 77.2	10, 30, and 45 bars	ND	Shigidi et al., 2022			
RO	X-20TM	Cr III	15, 26, 56 ppm		Synthetic				99	50 bars	0.15–1.02
		Ni II							98–99		
		Cd II		99							
		Pb II		99							
		Sb III		98–99							
		As III		98–99							
		Cr III		95–98							
		Ni II		97–99							
NF	NF90	Cd II	15, 26, 56 ppm	Synthetic	89–92	38 bars	0.33–22	This study			
		Pb II			73–76						
		Sb III			61–64						
		As III			51–54						
		Cr III			98–99						
		Ni II			98						
		Cd II			92–95						
		Pb II			77–82						
NF	NF270	Sb III	15, 26, 56 ppm	Synthetic	61–64	38 bars	0.11–22				
		As III			48–53						

ND: No data.

4.3.4. Transmembrane flux

The results in Sections 3.1–3.3 show that the performance of membranes is evaluated based on the concentrations of the feed solutions. Permeate flux is one of the key factors demonstrating the effectiveness of membranes in resisting fouling (Luis, 2018).

The permeate flux of synthetic solutions and real waters was determined on the three membranes. The experimental results are presented in Figure 4.5 below, under operating pressures of 48.9 bar in reverse osmosis and 36.9 bar in nanofiltration. The nanofiltration membranes exhibited a high flux and a more severe flux drop in reverse osmosis.

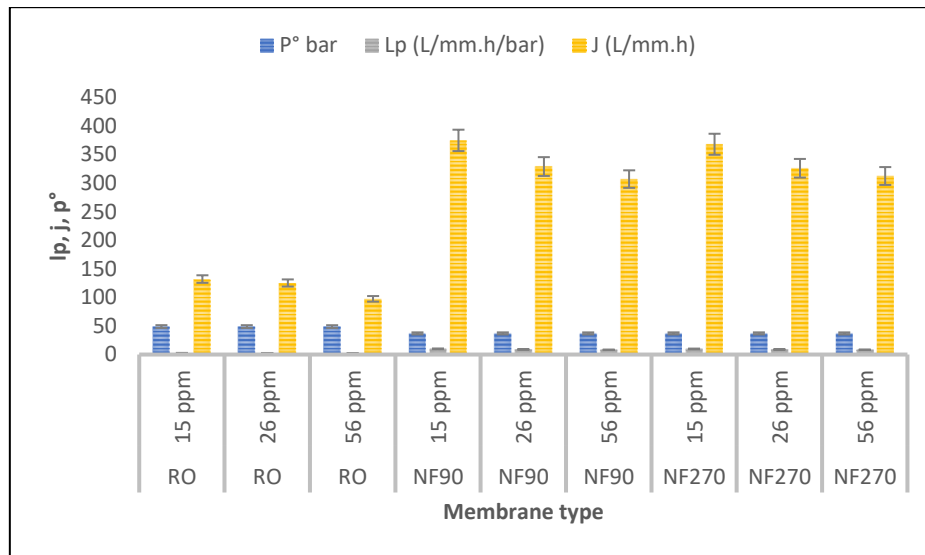


Figure 4.5. Permeability and permeate flow at constant pressure.

The water permeabilities of the nanofiltration membranes were much higher than those of the osmosis membranes and are, respectively, 2.7, 2.5, and 1.9 L/m².h/bar for RO, 10.1, 8.9, and 8.3 L/m².h/bar for NF90, and finally 9.9, 8.8, and 8.4 L/m².h/bar for NF270.

A study Yüksel et al. (2013) showed that the water permeability of NF90 and NF270 membranes were 6.05 and 14.86 L/m².h/bar and that of seawater reverse osmosis (SWRO) was 5.91 L/m².h/bar. Another study Lui et al. (2011) showed that pure water permeability and salt rejection of both NF90 and BW30 membranes were measured by crossflow permeation tests with pure water and an aqueous solution of 1000 mg/L of NaCl, and were found to be 9.4 and 4 L/m².h/bar, respectively. The difference in permeability is explained by the crossflow filtration system, the applied operating pressure, and the solute concentrations.

The increase in the concentration of solute slightly decreased the membrane permeability, and the temperature was raised from 299.14 to 303.14 K for RO, and from 296.14 to 297.14 K for NF. Low permeability due to solute concentration was observed at 15 ppm nanofiltration and 56 ppm reverse osmosis. Nanofiltration membranes had the highest water permeability and the rejection was lower than for reverse osmosis.

4.4. Conclusions

Three commercial nanofiltration and reverse osmosis membranes were successfully tested using synthetic solutions prepared in the laboratory and real water. The results showed that among the studied membranes, the reverse osmosis membranes had a lower permeability compared to nanofiltration membranes and the ion rejection was higher for the synthetic solutions. Rejection levels were higher than 98% for Cr^{3+} , Pb^{2+} , Cd^{2+} , As^{3+} , Ni^{2+} , and Sb^{3+} ions in the synthetic solutions, and 99, 99, 99, 99, 98 and 99%, respectively, in the real samples. The ion release rate in the real water was higher and met the WHO standard.

Regarding the NF membranes, the rejection of Cr, Ni, and Cd ions was interesting: 99, 98, and 92%, respectively. However, it was lower for Pb, As, and Sb ions: 78, 54 and 64%, respectively. The flux of NF was 329 to 375 $\text{L/m}^2\cdot\text{h}$, much higher than for RO membranes, which had a flux of 98 to 132 $\text{L/m}^2\cdot\text{h}$.

The elimination of heavy metals at low concentrations in the permeate implies that good-quality water can be transported to Lake Tanganyika without disturbing the ecosystem, as well as perhaps the possibility of reusing it by the local population.

In conclusion, nanofiltration is recommended for the removal of heavy metals from low-concentration wastewater, and reverse osmosis would be the best choice for a very high concentration of heavy metals in wastewater. However, commercial membranes are very expensive and present some challenges in terms of excessive energy consumption, are sometimes inefficient for removing some heavy metals, and a low flux is observed. It would therefore be interesting to develop specific low-cost membranes such as mixed-matrix membranes (MMM) based on a biodegradable polymer and nanoparticle, which combine several advantages in terms of high permeate flux, good selectivity, low manufacturing cost, and low energy consumption.

Chapter 5. Removal of toxic metal ions from surface water using biopolymer chitosan/ZIF-8 mixed-matrix membranes

In this chapter, the biopolymer chitosan (CS)/ZIF-8 selective layers were successfully deposited on two different PAN supports, namely wet PAN (PAN-w) and dry PAN (PAN-d) to fabricate mixed-matrix membranes (MMMs). PAN-w-supported MMMs exhibited better heavy metal retention properties than PAN-d-supported MMMs. Water contact angle analysis showed that all PAN-w-supported MMMs were hydrophilic. Moreover, the hydrophilicity varies with ZIF-8 loadings. Metal ion analyses were performed by standardized ICP-OES methods. The best results were obtained using a PAN-w-supported MMM with 3 wt.% CS as bio-matrix and 15 wt. % ZIF-8 as the filler, reaching a permeability of $4.6 \text{ L.m}^{-2}.\text{h}^{-1}.\text{bar}^{-1}$ at applied pressure of 30 bars. Significant rejections were also obtained: 13, 88, 98, 97, 85 and 24% for As^{3+} , Cd^{2+} , Cr^{3+} , Ni^{2+} , Pb^{2+} and Sb^{3+} , respectively, from a feed solution having a total initial concentration of 90 ppm (15 ppm per ion). Single element solutions, each containing only one element ion, were also filtered under the same operating conditions. Compared with rejection effects for the solution containing all the element ions, the rejection increased significantly for the Cd^{2+} , Pb^{2+} and Ni^{2+} ions, respectively, to 97; 96; 97% without compromising the Cr^{3+} rejection rate. Compared to commercial membranes, the economic profitability of the process of filtration can be guaranteed under a low driving pressure of 29 bar with an energy consumption of $2.10^{-3} \text{ kWh.m}^{-3}$.

5.1. Introduction

The high pressures traditionally used in reverse osmosis have resulted in a considerable energy cost and the application of reverse osmosis is limited to a certain extent (Lee et al., 2011). The membranes used in the RO process are generally thin film composite (Ismail et al., 2015; Xu et al., 2013). A very thin polyamide (PA) active layer deposited on a porous substrate is the main feature of the TFC (Ismail et al., 2015; Xu et al., 2013). Although TFC membranes have an excellent salt removal performance, there is a trade-off between permeability and selectivity (Yang et al., 2019).

Recently, reverse osmosis membranes from cellulose acetate materials operating at ultralow pressures of 1-15 MPa, have been developed (Van der Bruggen et al., 2003, Matsuura, 2001; Matsuura et al., 2001; Semiat, 2000), allowing desalination of brackish water at pressures comparable to those applied in nanofiltration. Werber et al (2016) reported that cellulose acetate membranes may have a better flux stability, improving permeability. CA membranes have their own limitations, such as high pH sensitivity and lower tolerance to high temperatures. In acidic and basic feed solutions, hydrolysis reaction will happen and lower the selectivity (Yang et al., 2019).

Polyamide-based TFC RO membranes are typically fabricated through depositing a thin separation layer on the top of the substrate of porous polysulfone via interfacial polymerization between *m*-phenylenediamine (MPD) and trimesoyl chloride (TMC) (Petersen et al., 1993). However, the presence of various inorganic and organic compounds in water affects membrane fouling, selectivity, and flux. To enhance the membrane performance, membranes that are blended/coated with various emerging (nano)materials e.g., carbon nanotubes (Farahbakhsh et al., 2019), graphene oxides (Chu et al., 2017; Chu, K., et al., 2017), MXenes (Ding et al., 2017; Han et al., 2017), or metal–organic frameworks (MOFs) (Basu et al., 2017; Denny et al., 2016), have attracted interest in the water industry due to their enhanced selectivity, hydrophilicity, and fouling resistance. Nanocomposite RO membranes, also called mixed matrix membranes or hybrid membranes, combine the advantages of organic membranes and inorganic materials, loaded with different inorganic particles are also applied in metal ion removal (Castro-Muñoz et al., 2021), successfully increasing water permeability and reduce fouling (Jeon et al., 2018; Zheng et al., 2017; Bi et al., 2018; Aligieri et al., 2021).

Among these emerging materials, metal organic frameworks (MOFs), defined as organic–inorganic hybrid microporous crystalline materials, have attracted a great deal of attention worldwide due to their great potential (Furukawa et al., 2013). Specifically, MOFs have

exceptional characteristics: (i) high tunable porosities and accessible large surface areas (Lu et al., 2014), and (ii) high capacity to combine particular species/functionalities easily without changing the framework topology (Evans et al., 2014). Among available MOFs, ZIF-8 has a high surface area, high adsorption capacity, thermochemical stability, hydrophobicity, and above all, it can be synthesized at room-temperature (Abbasi et al., 2020; Li et al., 2021).

On the other hand, chitosan (CS) is an important natural renewable biopolymer, one of the cheapest polysaccharides with inherent abundant groups (Agnihotri et al., 2004; Kumar et al., 2016; Yang et al., 2016; Lijuan Liu., 2019). It can be used as selective layer on the membrane surface due to its unique properties such as antibacterial activity, bio-compatibility, non-toxicity and good film-forming ability (Xiaowei et al., 2018; Kumar et al., 2016; Mohammad et al., 2019). Chitosan, due to the presence of hydroxyl and amine groups, has been widely used for the removal of heavy metal ions from aqueous solutions (Rodrigo et al., 2006; Sajjad et al., 2009). However, the weak mechanical properties of chitosan limit its potential (Xuejie et al., 2020). Introducing ZIF-8 into chitosan can improve the performance and properties of nanocomposite membranes, as this type of membrane combines the processability of polymers with good charge transport properties of ZIF-8 (Shalumon et al., 2010; Khosravi et al., 2012). CS/ZIF-8 MMMs have been investigated for liquid separation (Xu et al., 2022; Fazlifard et al., 2017; Kang et al., 2013; Yang et al., 2021; Won et al., 2003) and some studies have been conducted on the adsorption of U, Cr^{6+} , Pb^{2+} , rare earth elements, Congo red and Cd^{2+} . Table 5.1 presents a summary of the key studies carried out on the adsorption of ions by CS/ZIF-8.

Table 5.1. Studies on adsorption of heavy metals by CS/ZIF-8

Adsorbent	Metal ions	q_{max} (mgg^{-1})	Ref
CS/ZIF-8	U (VI)	629	Lui et al., 2019
ZIF-8/CS/melamine	U (VI)	95.47 - 129.9	Guo et al., 2022
CS/ZIF-8	Cu	165.7	Wang et al., 2022
	Pb	131.4	
CS/ZIF-8	Pb	99.95 %	Yu et al., 2022
	La III	256.4	
EDTA-CS/ZIF-8	Eu III	270.3	Feng et al., 2021
	Yb III	294.1	
CS/ZIF-8 chitosan – graphene oxide/ZIF	Congo red	987.01	Liu et al., 2000
	U (VI)	361.01	
Fe ₃ O ₄ /CS/ZIF-8	Malachite green	3.282	Zadvarzi et al., 2020
PNVCL/CS/ZIF-8	Cr(VI)	495.6	Bahmani et al., 2019
	As(V)	439.5	

However, the state-of-the-art shows that a few studies have been conducted to remove metals from wastewater by pressurized membrane filtration using MMMs combining chitosan and ZIF-8. Considering the attractive mechanical properties of CS/ZIF-8 MMMs, the main objective of this work is to synthesize low-cost biopolymer CS/ZIF-8 MMMs with good stability to remove toxic metal ions from surface water. Two PAN support materials will be evaluated (wet and dry) and the loading of ZIF-8 will be a critical parameter in the study.

5.2. Methodology

5.2.1. Materials

5.2.1.1. Characteristic of PAN supports

Two types of PAN supports were used to prepare the mixed matrix membranes, the highly porous wet PAN support (PAN-w) and the dry PAN support (PAN-d), for which the supplier did not provide full information. The wet PAN (PX-FSR-1, PX flat sheet roll) and the dry PAN supports were supplied by Synder Filtration (USA) and Mega vision (China), respectively. The specifications of both supports are included in Table 5.2.

Table 5.2. Specifications of PAN supports used in the experiments (information provided by the supplier).

Support type	Pure water flux (L/m ² .h)@0.1MPa	Pure water flux (L/m ² .h)@0.3MPa	Bubble Point	Pore Size
PAN-d	680	NA	0.012 ~ 0.02	80 KD
PAN-w	NA	NA	NA	400 KD

NA: not available

5.2.1.2. Chemicals

Zinc nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, 99%), 2-methylimidazole (97%) and dimethyl carbonate (DMC, 99%) were purchased from Alfa Aesar (Germany). The CS polymer powder (low molecular weight) was obtained from Sigma-Aldrich (US). Absolute ethanol ($\text{C}_2\text{H}_5\text{OH}$, 99.9%), ethanolamine ($\text{NH}_2\text{CH}_2\text{CH}_2\text{OH}$, 99.5%), triethylamine (TEA, $\text{N}(\text{CH}_2\text{CH}_3)_3$, 99.5%), methanol (MeOH , 100.0%), glutaraldehyde, sodium hydroxide and acetic acid (CH_3COOH , 99.8%) were provided by VWR International (Belgium). All chemicals were used as received.

5.2.1.2. Synthesis of ZIF-8 particles

Submicron-sized ZIF-8 particles were synthesized according to the aqueous room-temperature method (Nordin et al., 2014). First, 2 g of $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ were dissolved in 12.11 g of deionized water to prepare $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ solution and 3.312 g of 2-methylimidazole was dissolved in 48.45 g of deionized water to prepare 2-methylimidazole solution. Then, 3.0 ml of TEA was added into 2-methylimidazole solution. Subsequently, these two solutions were stirred for 0.5 h. The product was centrifuged at 4000 rpm for 15 min and subsequently washed with deionized water twice and with absolute ethanol once. The obtained product was dried in a vacuum oven under 120 °C for 10 h.

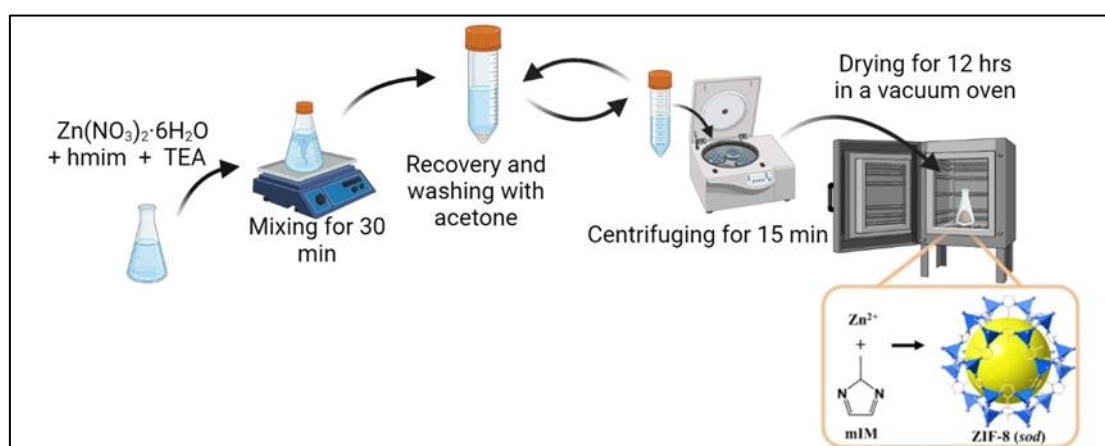


Fig 5.1. Scheme of the preparation of ZIF-8.

5.2.1.3. Membrane preparation

The MMMs were synthesized with 1.0 g of CS powders dissolved in 49.0 g of 2 wt.% CH_3COOH solution and stirred at room temperature for 2 hrs. Ethanolamine was then added to adjust the pH of the solution to $\text{pH} \geq 4$ because ZIF-8 can maintain its good crystallinity after being immersed in aqueous solution of $\text{pH} = 4$ for 3 days (Leus et al., 2016). The solution was then stirred for 24 h until chitosan and ethanolamine formed a homogeneous solution. The required amounts of ZIF-8 particles were dispersed into chitosan solution and stirred for 12 h to allow for an even distribution of ZIF-8 particles into CS solution. To prepare the membrane, the support was attached in a mold, on which the first thin layer of CS/ZIF-8 was cast, after 30 minutes of fixation on the support, the second layer was poured onto a casting table and squeegee cast with a gap of 250 μm . The membranes thickness of the thin film was measured using a Mitutoyo micrometer (TESA model, manufactured in Switzerland) and the thickness is $\pm 1 \mu\text{m}$. The loading of ZIF-8 in nanocomposite was defined as:

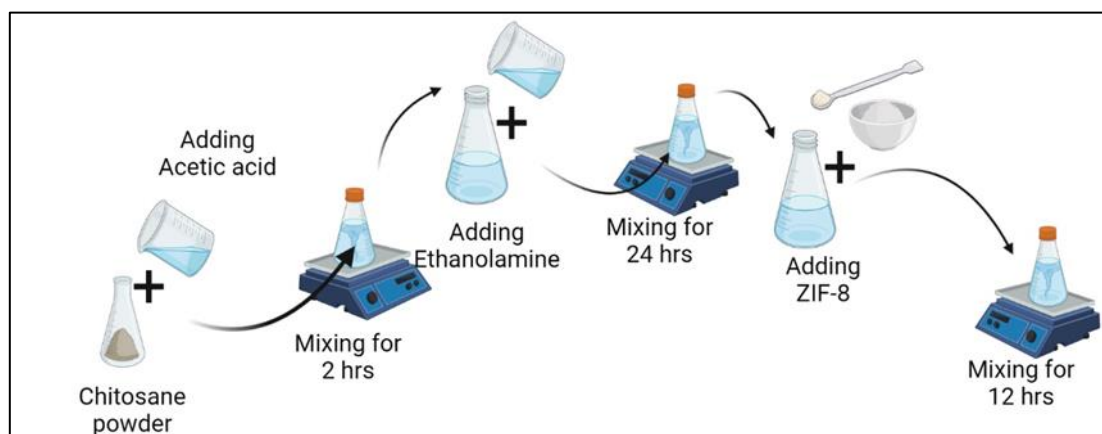
$$\omega_{ZIF-8} = \frac{m_{ZIF-8}}{m_{ZIF-8} + m_{CS}} \times 100\% \quad (11)$$

wherein w_{ZIF-8} is the ZIF-8 particle loading and m_{CS} represents the mass of chitosan.

The ZIF-8 particle loadings in the present study were varied as 0, 1, 5, 10, 15 and 30 wt%, and the resulting membranes were designated as M-0, M-1, M-5, M-10, M-15 and M-30, respectively.

The membrane was then immersed in NaOH 1.0 M solution at 25 °C for 2 h to neutralize residual acetic acid, washed repeatedly with Milli-Q water, and held in water before use. Finally, the membrane was brought under a treatment with glutaraldehyde in order to bind and couple the functional groups between the polymer chains to preserve the structural the membranes were immersed in 100 ml of glutaraldehyde solution of 0.25% (w/w) overnight (14 h) at 25 °C. Afterwards, membranes were exhaustively washed and kept in Milli-Q water at 4 °C. Figure 5.2 shows the steps of the overall procedure for membrane synthesis.

a)



b)

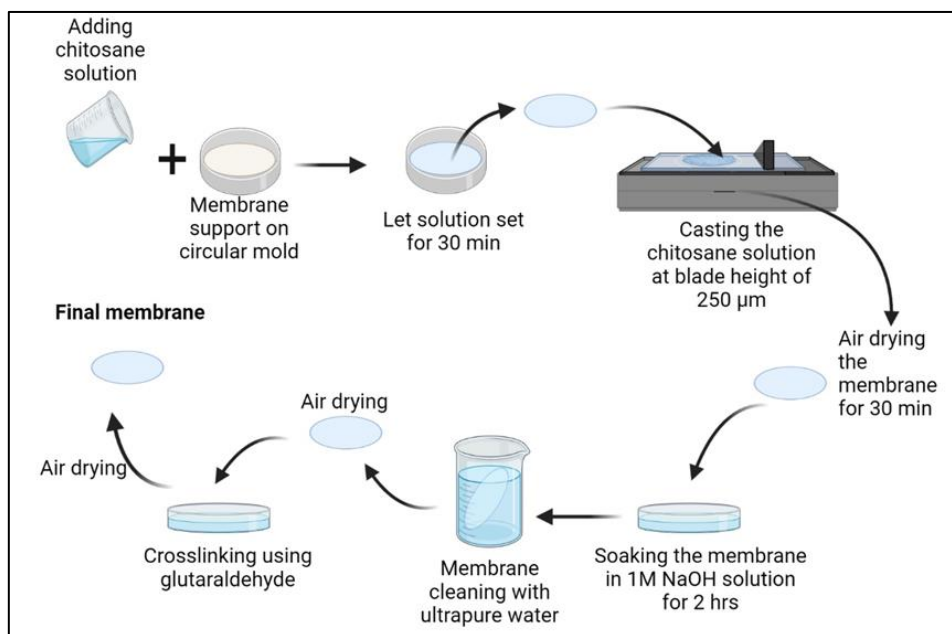


Fig 5.2. Procedure for membrane synthesis: a) Thin layer solution preparation; b) Membrane preparation.

5.2.1.4. Membrane performance evaluation

The performance of the CS/ZIF-8 nanocomposite was evaluated in a cross-flow filtration system represented in Figure 4.1 above, with an effective membrane surface area of 6.6 cm². Feed solutions containing chromium, arsenic, nickel, antimony, lead and cadmium were tested. Solutions were prepared with only one element ion, and also with the six element ions together. Synthetic samples of water loaded with chromium, arsenic, nickel, antimony, lead and cadmium were prepared at different concentrations. Feed water solutions containing different concentrations (i.e., 15 and 100 ppm of each ion) of Cr³⁺, Pb²⁺, Cd²⁺, Sb³⁺, Ni²⁺ and As³⁺, were freshly prepared from an analytical grade standard solution (Sigma Aldrich, Merck KGaA 64271 Darmstadt, Germany) containing 1000 mg L⁻¹ of As³⁺, Cr³⁺, Pb²⁺, Cd²⁺, Ni²⁺ and Sb³⁺. A standard solution (Certipur®) was used to prepare the stock solutions. The filtration of these model feed solutions was used to evaluate the rejection performance of reverse osmosis CS/ZIF-8 membranes, under a feed pressure of 30 bar. Four experiments were performed for each nanocomposite membrane. The permeation flux was calculated according to equation (8) above. The membrane permeability L_p (L/m² h/bar) to water was determined according to the formula (10) below. The driving pressure ΔP was calculated by formula (9) above.

$\Delta\pi$ is the osmotic pressure difference between retentate and permeate (bar), was calculated from formula. The ion rejection rate was calculated by the following equation (7) above. Concentrations were determined by inductively coupled plasma optical emission spectroscopy (ICP-OES, Agilent technology).

The energy consumed in reverse osmosis during a time t , E_p (kWh) was calculated as:

$$E_p (kWh) = \frac{P(Pa) \cdot Q\left(\frac{m^3}{h}\right) \cdot d}{1000 \cdot \eta_p} \quad (12)$$

with η_p as the efficiency of the pump; P : water pressure at the pump outlet (Pa); Q : water flow (m^3/s), and d : density of the liquid.

The experimental setup shown in Figure 4.1 above was used for determination of cross flow filtration performance using a range of membrane types with relatively small sample volumes.

5.2.1.5. Membrane characterization

A Scanning Electron Microscope (SEM) (Zeiss, ULTRA with Bruker energy dispersive X-ray (EDX) chemical analyzer) was employed to portray the morphology of the fabricated membranes. Membranes were beforehand immersed into liquid nitrogen for a clean cross-sectional cut. Next, a thin layer of gold was deposited under vacuum with a sputter coater (BALZERS UNION FL 9460 BALZERS SCD 030) to make the samples conductive. Contact angle measurement was measured by Contact Angle System OCA provided by DataPhysics Instruments. As for the crystallinity of ZIF-8 particles and membranes, it was investigated using X-ray diffraction (XRD, Bruker D8 Advance, Cu $K\alpha$ radiation, $\lambda=1.5406 \text{ \AA}$) in the range from 6 to 60 degree., while FT-IR spectra of ZIF-8 and membranes were obtained using a Bruker spectrometer in the range from 400 cm^{-1} to 4000 cm^{-1} in ATR mode to investigate the chemical properties of all the membrane samples.

5.3. Results and discussion

5.3.1. ZIF-8 characterization

ZIF-8 nanoparticles and fabricated membranes were characterized to obtain adequate information on their structure and properties. The size of the as-synthesized ZIF-8 particles was in the range of 100–200 nm. The XRD analysis showed a crystalline structure of ZIF-8 with characteristic peaks at 2θ values of 7° , 10° , 12° , 14° , 18° , 22° , 24° , 26° , 29° , 33° , 34° and 35° . Fig. 5.4, shows the SEM image of the as-fabricated ZIF-8 particles.

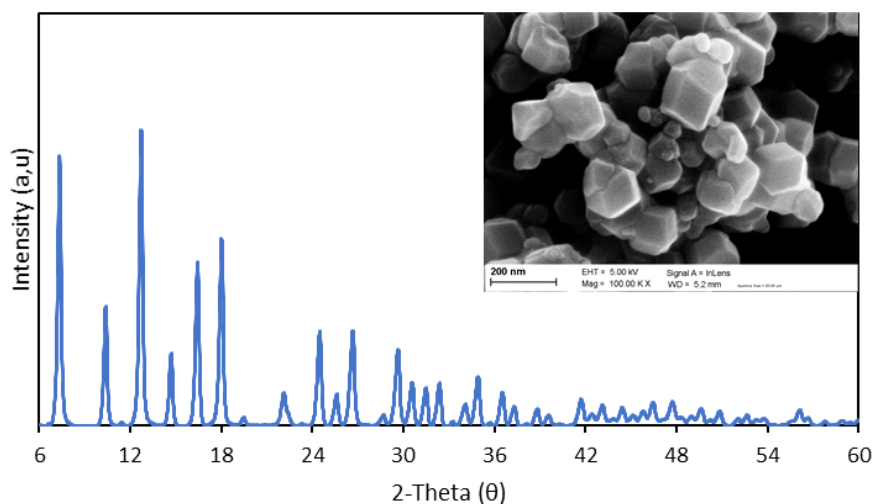


Fig. 5.3. XRD of ZIF-8 (inset image: SEM)

5.3.2. Membranes characterization

XRD analyses of PAN-w and PAN-d media are shown in Fig. 5.4 a. The two supports have a high intensity peak located at $2\theta = 25^\circ$ and a low intensity at 56° . The first intense peak of PAN-d support at $2\theta = 30^\circ$ corresponds to the diffraction of the intense peak at $2\theta = 25^\circ$. The average peak located at $2\theta \approx 20^\circ$ and another weak one at $2\theta = 23^\circ$ are considered as the reflection of the intense peak at $2\theta = 25^\circ$. The PAN-w presents the weak peaks at $2\theta \approx 30^\circ$ corresponds to the diffraction of the intense peak at $2\theta = 25^\circ$ and the two small peaks at $2\theta = 20^\circ$, 23° are considered as the reflection of the hexagonal lattice (Badawy et al., 2003; Pirlot et al., 2002; Zhang et al., 2017).

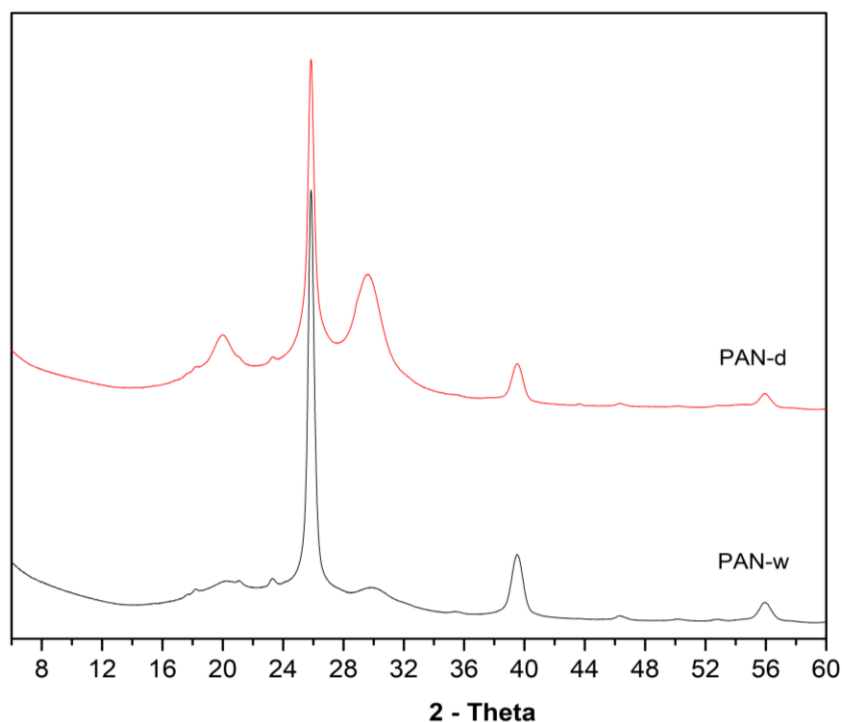


Fig. 5.4. a: XRD models of PAN-w and PAN-d brackets

To analyze the structure of ZIF-8 and CS/ZIF-8 membranes, X-ray diffraction (XRD) experiments were conducted (Fig. 5.4 b) show the characteristic diffraction peak of CS located at $2\theta = 22^\circ$. In addition, M-15 (CS/ZIF-8 nanocomposite with 15 wt% ZIF-8) show slightly at $2\theta = 21^\circ$, 32° and 35° , which belong to ZIF-8, suggesting the presence of ZIF-8 on the chitosan surface. Even though the results do not show complete evidence of the presence of ZIF-8, the difference in performance (see Figure 10) encouraged to elaborate this discussion. The disappearance of some ZIF-8 diffraction peaks in ZIF-8-based nanocomposite can be attributed to the high affinity between the lattice planes of ZIF-8 and CS polymer backbone (Khajavian et al., 2020; Zhang et al., 2018).

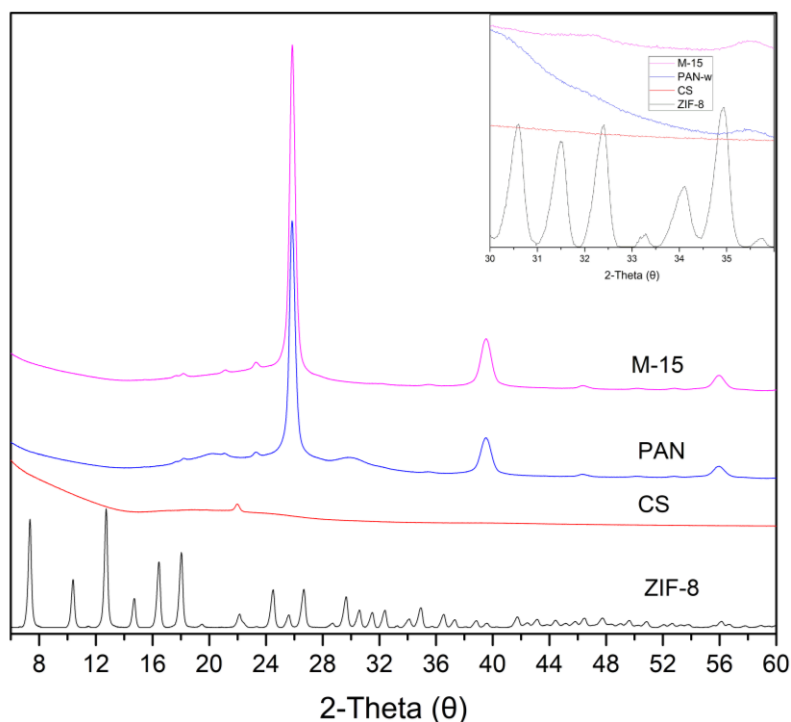


Fig. 5.4. b: CS/ZIF-8 MMM XRD models

FTIR analysis of two PAN media shows the similar peaks. A peak at 2240 cm^{-1} was observed in the FTIR spectra was related to the $\text{C}\equiv\text{N}$ group and the peak at 2940 cm^{-1} is attributed to the stretching vibration of the $-\text{CH}_2$ groups. The peaks at 1200 and 1400 cm^{-1} are attributed to the bending vibrations of the $\text{C}-\text{H}$ groups and the peak at 1730 cm^{-1} has been attributed to $\text{C}=\text{O}$ (fig 5.4 c). The $\text{O}-\text{H}$ cluster peak was broad at $3150\text{--}3690\text{ cm}^{-1}$ on the wet medium, illustrating the presence of the $\text{O}-\text{H}$ groups in the hydroxymethyl of the immobilized phenolphthalein moieties (Zhang et al., 2009). These results are similar to studies by Ahn et al. (2021 and Shoushtari et al. (2006).

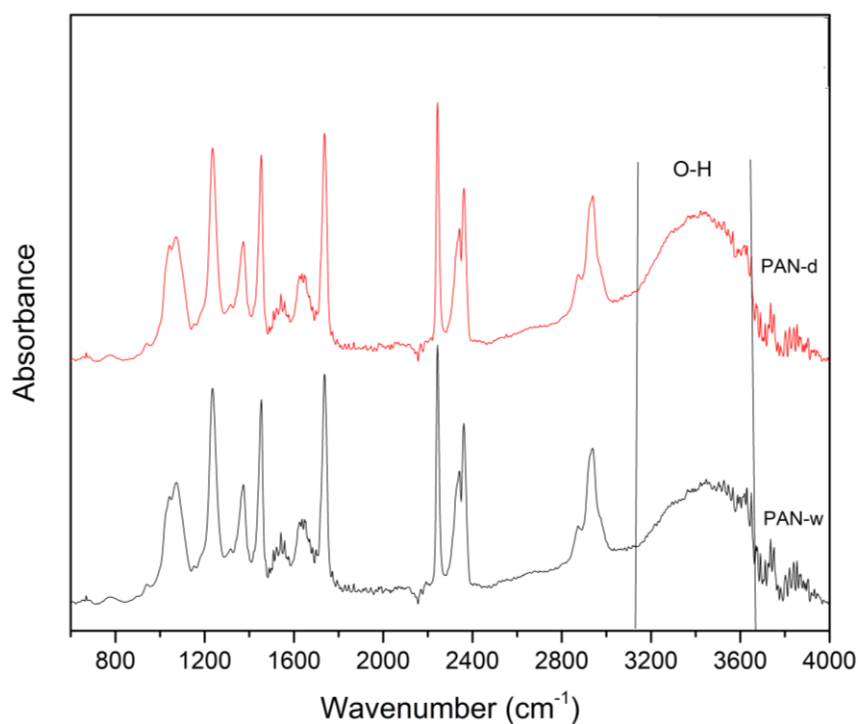


Fig. 5.4. c: FTIR spectra of PAN

FTIR spectroscopy is shown in Fig. 5.4 d, the absorption peak at 2929 cm^{-1} was attributed to the stretching vibration of the C=H bond of the methylimidazole ring of ZIF-8. The peak at 1600 cm^{-1} originated from the C=N stretching vibration. Another peak at 1584 cm^{-1} was associated with the -NH_2 vibration, while the peak at 920 cm^{-1} originates from the stretching vibration of CN (fig. 5.4 d). The characteristic peaks of the ZIF-8 crystal spectrum can be seen in Figure 5d together with the ones from the MMMs. These results are consistent with those in

the literature (Zhang et al., 2018; Rao et al., 2020; (Liu et al., 2019; Xiao et al., 2022).

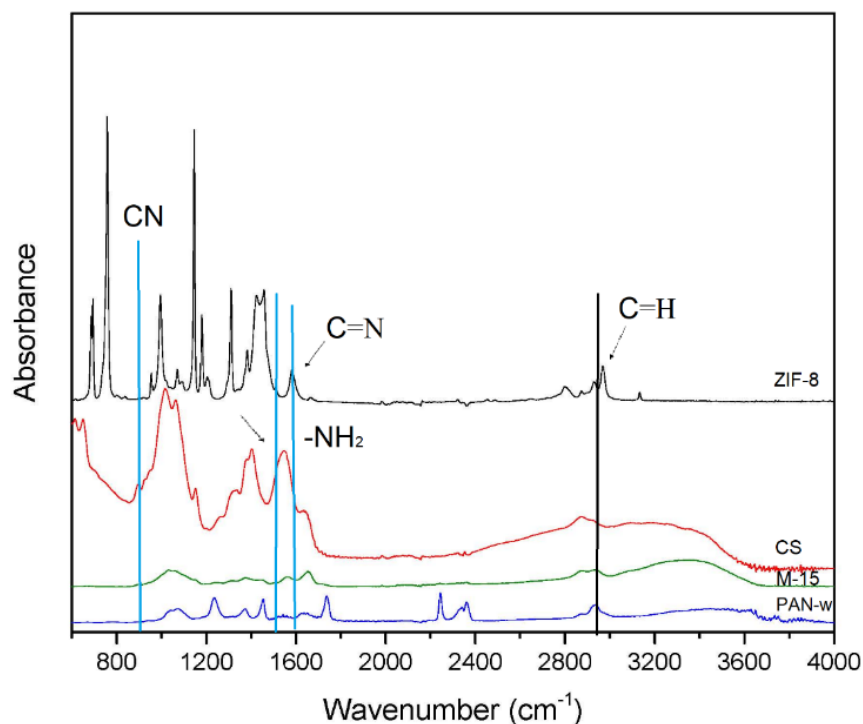


Fig. 5.4. d: FTIR spectra of ZIF-8/CS MMM

SEM images of the PAN-w supports show that the pores are large Fig. 5.5(a). The materials constituting the thick support layer are spacious Fig. 5.5(a). In contrast to PAN-d, these pores are small and very tight Fig. 5.5(b) and the support materials which are thin and tight, see cross section Fig. 5.5(b). These observations by SEM were confirmed on the description of the supports by the suppliers in Table 5.2.

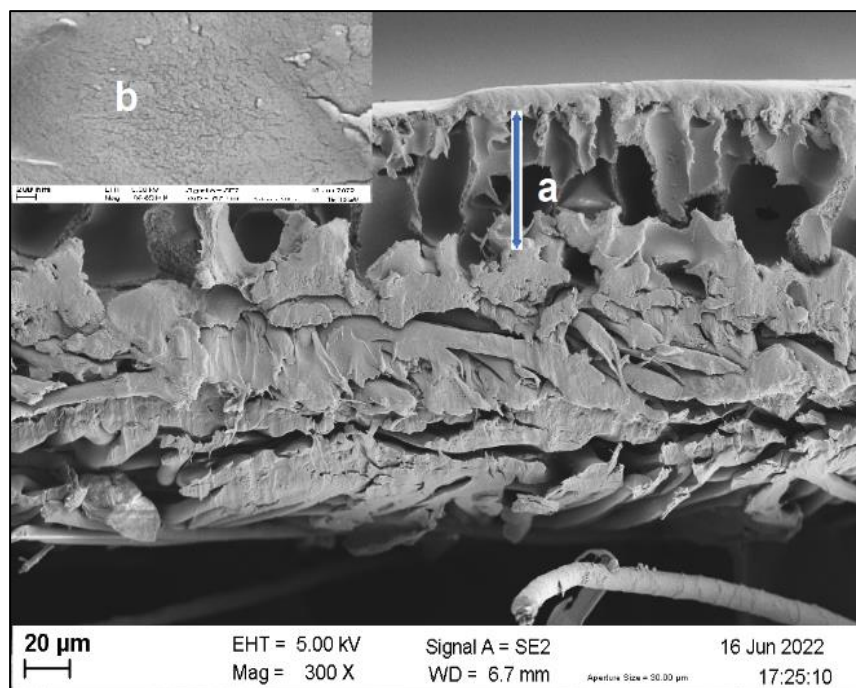
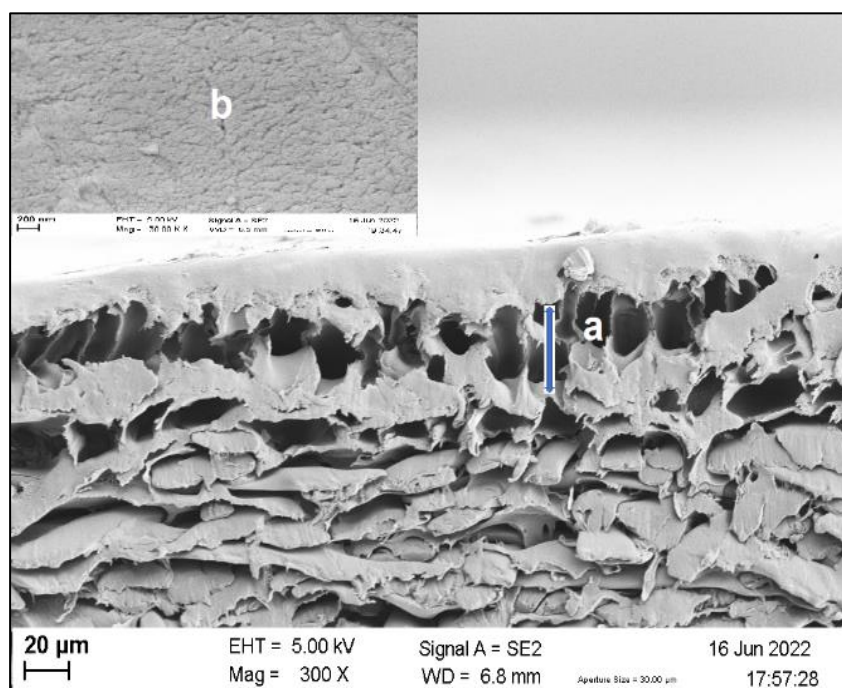


Fig. 5.5 a: SEM images of PAN-w supports;



b: SEM images of PAN-d supports

The membranes have better surface roughness. The magnified image further confirms that the PAN carrier has been fully coated with the CS/ZIF-8 dense thin layer.

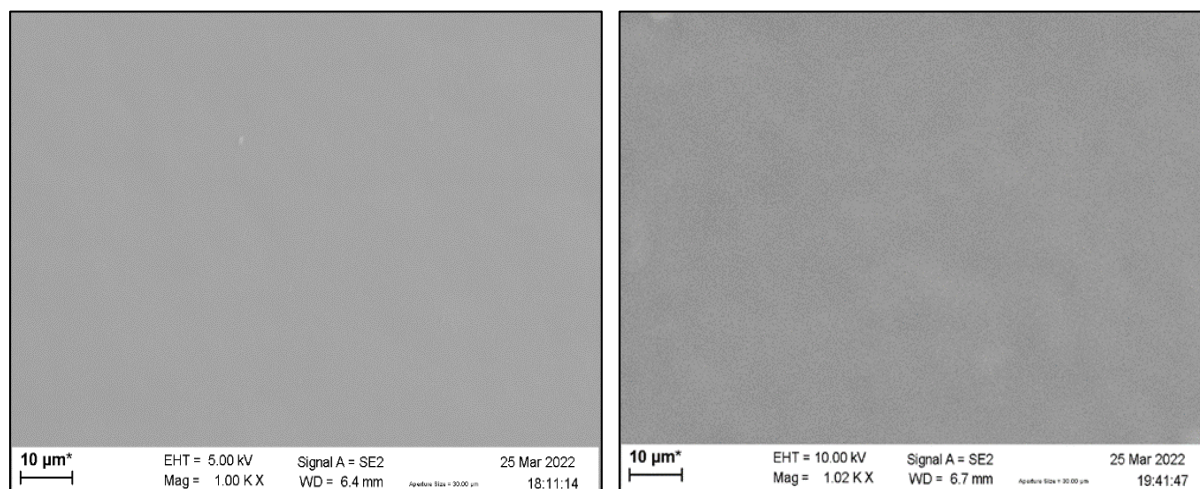


Fig. 5.5. d: SEM images of CS/ZIF-8 at different magnifications;

The cross-sectional images of the synthesized 15% by weight CS/ZIF-8 nanocomposite are illustrated in (Fig. 5.5. e, f), indicating good attachment between the upper thin layer and the PAN support. The SEM results showed that MMM CS/ZIF-8 was successfully synthesized on the PAN support.

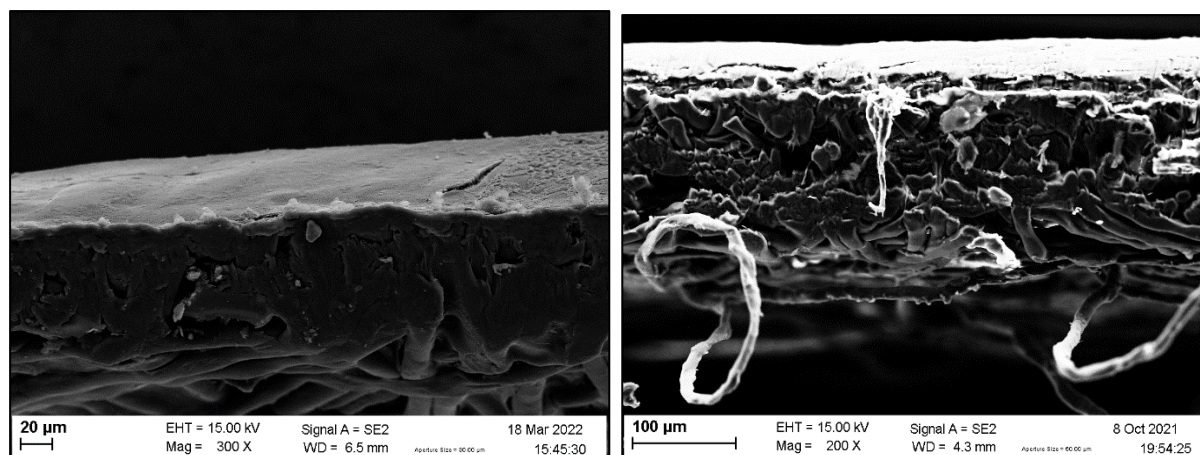


Fig. 5.5. f: Cross-section SEM image modified nanocomposite membrane.

EDX (Energy Dispersive X-ray Spectroscopy) results were obtained from SEM analysis on cross-sections of both PAN-w and PAN-d media; the spectrum confirmed the presence of elements such as C, O, N, Pd and Au, as shown in Fig. 5.6 (a, b), (c, d) in addition, for the CS/ZIF-8 MMM membranes, the spectrum confirmed the presence of Zn in ZIF-8, as well as the peaks of other elements such as C, O, N and Au were present.

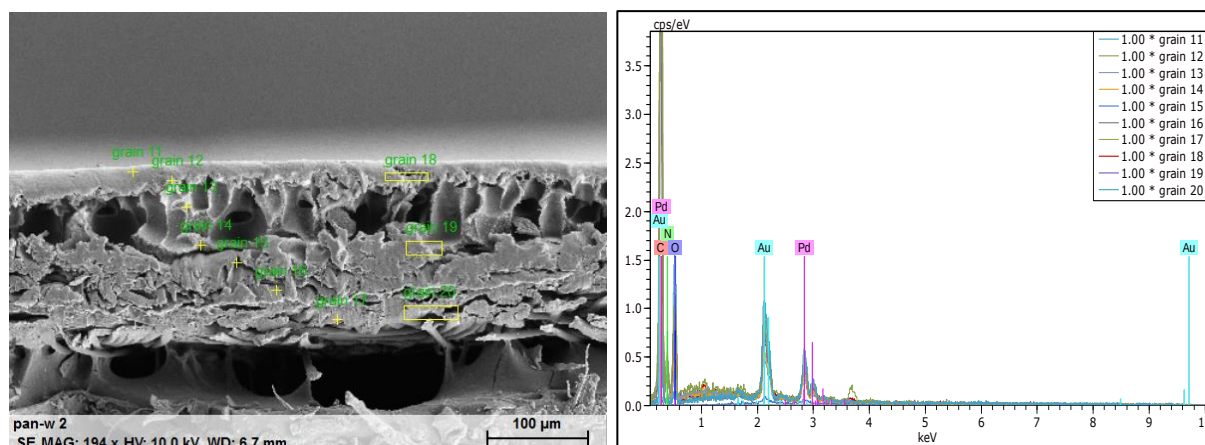


Fig. 5.6 a, b: EDX spectrum of PAN-w support;

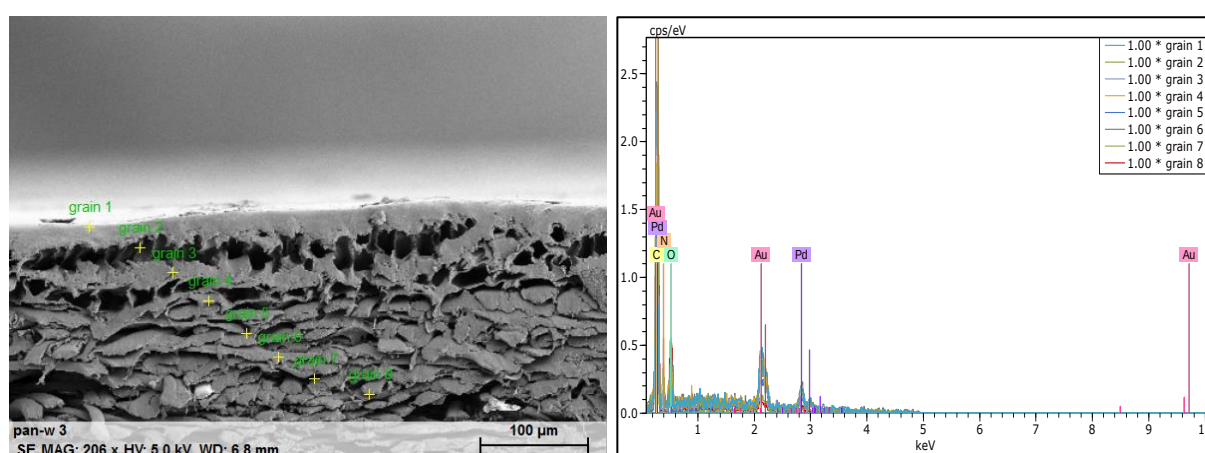


Fig. 5.6. c, d: EDX spectrum of PAN-d support

In addition, EDX (Energy-dispersive X-ray spectroscopy) results were obtained from SEM analysis; the spectrum confirmed the presence of Zn in ZIF-8 could be clearly observed as well as the peaks of other elements such as C, O, N and Au are not marked. However, the elements detected in the EDX spectrum indicate that the Zn-based MOF was successfully synthesized.

Fig. 5.6 (e, f), below shows energy dispersive X-ray spectroscopy

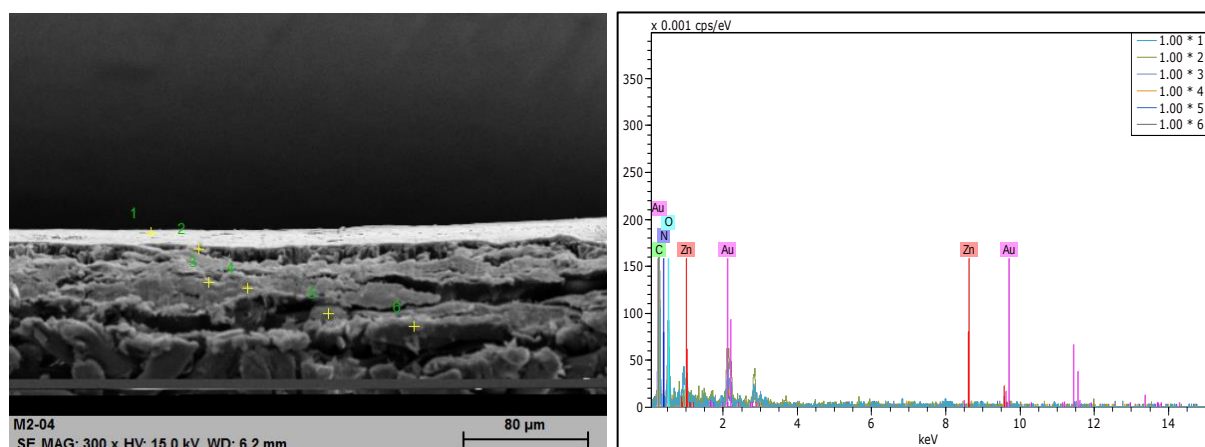


Fig. 5.6. e, f: EDX spectrum of nanocomposite membrane

5.3.3. Contact angle analysis

A quantitative analysis of the hydrophobicity of the material was made by the contact angle (CA), generally considered as hydrophobic if $CA > 90^\circ$ and hydrophilic if $CA < 90^\circ$. The contact angle of the CS/ZIF-8 nanocomposite was $<90^\circ$, which confirms the hydrophilic nature of the membranes. However, the membranes prepared on the wet support are very hydrophilic, the CA was 76.8; 65; 60.1 and 43.8° . The ZIF-8 filler increased the hydrophilicity and the membranes have a better wettability.

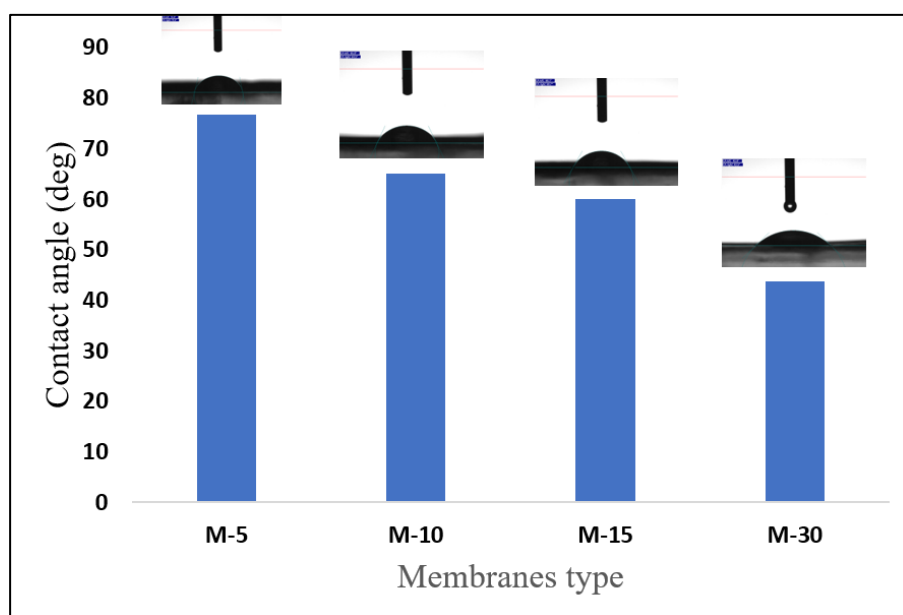


Fig. 5.7. The contact angle of the CS/ZIF-8 nanocomposite

5.3.4. Membrane performance

The MMMs were prepared on the PAN-w and PAN-d, respectively. The selective layers of MMMs with different ZIF-8 loadings were $\pm 1 \mu\text{m}$. The membrane prepared on PAN-w, without incorporated ZIF-8 nanoparticle in CS matrix (M-0), had a higher percentage removal of Cr, Cd, Ni and Pb ions, while the membrane prepared on PAN-d without ZIF-8 (M-0) displayed a higher removal of As and Sb ions, as shown in Fig. 5.8 a. These results were used as a reference to compare the variation of the rejection and the functioning of the membranes by adding ZIF-8. Ion rejection by CS/ZIF-8 MMMs is shown in fig. 5.8 a. The concentrations with 15% wt. ZIF-8 on the PAN-w were optimal for the retention of Cr^{3+} , N^{2+} , Cd^{2+} and Pb^{2+} . However, the result was compared with that obtained by Klaysom et al (2013), observing that the salt retention was almost constant at around 94% and the water permeance was $0.88 \text{ L m}^{-2} \text{ h}^{-1}$.

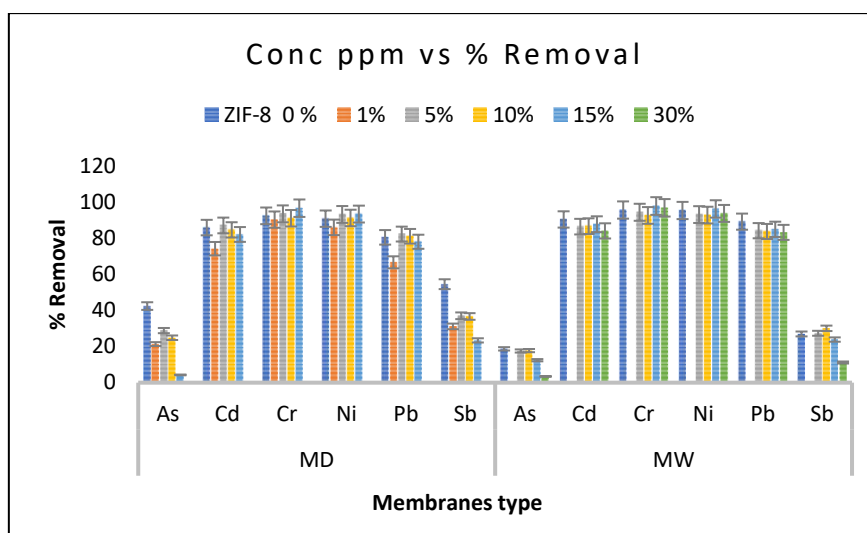


Fig. 5.8 a: % removal by CS/ZIF-8 MMMs. MD: Dry support membrane; MW: Wet support membrane

5.3.4.1. Effect of ZIF-8 on ion retention

Two types of solutions were tested, with only one ion and with all the ions together. The 15 ppm solution containing all six metal ions (Cr^{3+} , Ni^{2+} , Cd^{2+} , Pb^{2+} , As^{3+} and Sb^{3+}) was filtered under a pressure of 30 bars at 297.14 K in the reverse osmosis unit. The addition of ZIF-8 in the CS had a positive effect on increasing the removal of Cr, Ni, Cd and Pb ions to $\pm 5\%$. The results showed that the addition of ZIF-8 in the CS prepared on PAN-w, increased the removal of Cr^{3+} , Ni^{2+} , Cd^{2+} , Pb^{2+} by 98, 97, 88 and 85% respectively. The removal of Cr^{3+} , Ni^{2+} , Cd^{2+} , Pb^{2+} ions increased by 97; 94; 82 and 78.2% respectively on the membrane prepared with a PAN-d support. The removal of As^{3+} and Sb^{3+} on the CS membrane without ZIF-8, prepared on PAN-d was 43 and 55%, and was 11 and 27% on PAN-w. The addition of ZIF-8 in CS did not influence the removal of As^{3+} and the removal of Sb^{3+} had decreased on the same PAN-w. In contrast on PAN-d, the introduction of ZIF-8 greatly decreased the percentage removal to 4%. Sb^{3+} removal also decreased to 23% Fig. 5.9.

The addition of ZIF-8 into CS confirmed the MMM CS/ZIF-8 performance to remove Cr^{3+} , Cd^{2+} , Ni^{2+} , Pb^{2+} by 93, 89, 92 and 86%, respectively, from feed solutions of 100 ppm each. It is observed that the rejection of Cr^{3+} and Ni^{2+} was similar to that of the commercial membrane in RO, which was respectively 99 and 98.8% at 50 bar and 303.14 K as shown by Lumami et al (2022). While the rejection rate was high for the ions Cd^{2+} 99%, Pb^{2+} 99%, Sb^{3+} 98% and As^{3+} 99% on the same commercial membrane. Mohammed et al (2020) were the only one to conduct the research on membrane filtration of CS/ZIF-8 and obtained 96% Cr^{6+} ion rejection on PVDF/CS/ZIF-8 membrane. The PVDF/CS/ZIF-8 membrane of their work had been

modified to remove a Cr ion in solution, while in this work, MMM PAN/CS/ZIF-8 was modified to simultaneously remove Cr, Cd, Pb and Ni.

The second type of feed solution containing only one ion in the solution was filtered under the same conditions using the nanocomposite CS/ZIF-8 membranes. They exhibited high rejection of Cr^{3+} , Ni^{2+} , Cd^{2+} , Pb^{2+} similar to the commercial membrane (Lumami et al., 2022), Fig. 5.9 b and c shows the retention results. The rejection decreases in multi-ion solutions, firstly due to the high concentration of solute in the vicinity of the membrane, which could lead to concentration polarization (Koseoglu et al., 2018; Luis, 2018). Overall, the removal of heavy metals in the permeate depends on the feed concentration. it is possible that the phenomenon of concentration polarization manifests (Koseoglu et al., 2018; Luis, 2018), itself further according to the theory of solution diffusion, the water and solute molecules diffuse through the dense rejection layer independently of each other. Overall, the removal of heavy metals in the permeate depends on the feed concentration.

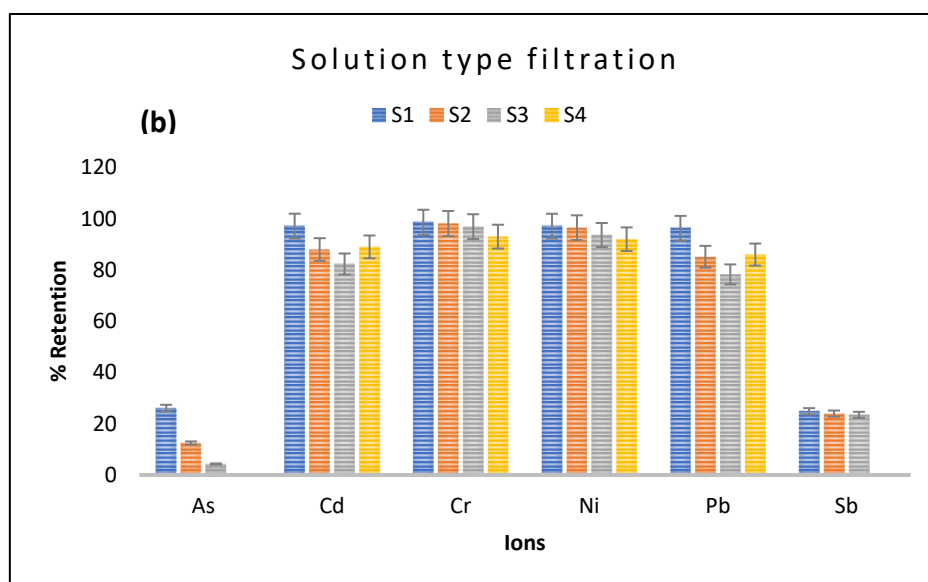


Fig. 5.9. S1: An ion in solution, filtered on PAN-w; S2: Six ions in solution, filtered on PAN-w; S3: Six ions in solutions, filtered on the PAN-d; S4: An ion in solution 100 ppm;

5.3.4.2. Interaction mechanism

Chitosan is a very interesting polymer since amino groups have a higher activity than hydroxyl or carboxyl groups (Carneiro et al., 2015). Hydroxyl and amine groups can interact with metal ions. Amino groups are highly reactive with metal ions. This is because nitrogen atoms have non-bonding doublets, giving amines a basic, nucleophilic character that can react with metal cations. The amine groups of chitosan are much more reactive than the acetamide groups of chitin (Guibal et al., 2004).

OH groups in metal binding have been cited in some studies, it is now commonly accepted that metal binding takes place primarily on amine groups. Hydroxyl groups essentially contribute to stabilizing metal binding to amine groups. Interestingly, chitosan has a very limited affinity for alkali and alkaline earth metals due to the absence of unsaturated d and f orbitals (unlike transition metals) (Carneiro et al., 2015).

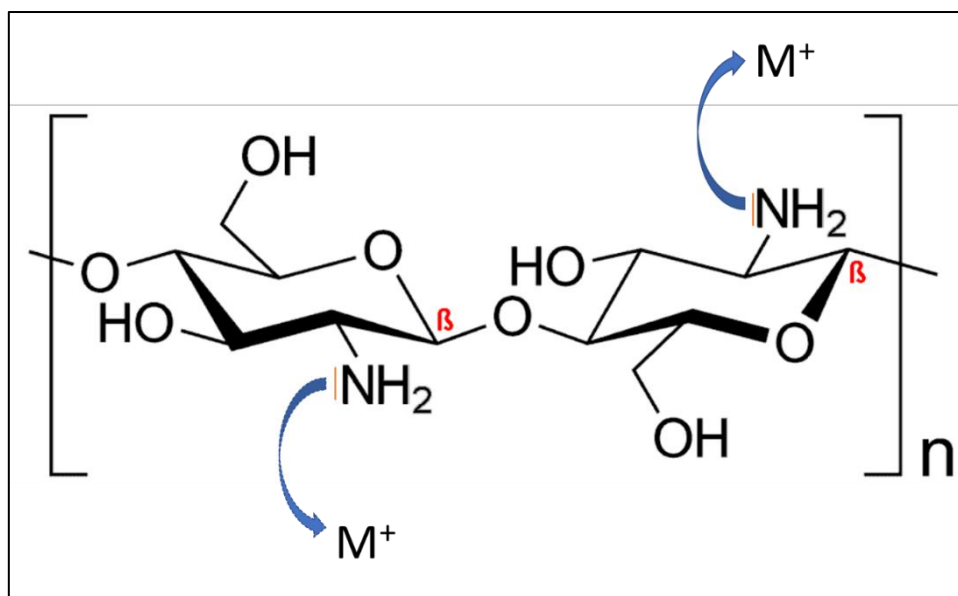


Fig 5.10. Interaction mechanism for metal ion removal (Guibal et al., 2004, Carneiro et al., 2015)

5.3.4.2. Effect of ZIF-8 on permeate flux

Permeate flux is one of the key factors for evaluating the performance of membranes, reflecting permeate quantity and species selectivity (Luis, 2018). The pristine CS membrane with zero loading showed the lowest performance in all experiments while the ZIF-loaded membranes had significant improvements in terms of permeate flux.

By introducing ZIF-8 at different concentrations, the flux increased considerably on PAN-w-based nanocomposite membranes because of its excellent porous medium. The effects of loading ZIF-8 into the CS selective layer can be clearly seen in Figure 5.9. A significant improvement in water permeate flux was found compared to the commercial RO TFC X-20TM membrane which was 132 L/m².h at 48.96 bar. The same trend was observed by other researchers, using 0.6 % ZIF-8 increased flux from 12 to 38 L/m².h (Aljundi et al., 2017). The permeate flux for each ion in solution is shown in Fig. 5.12. This flux of permeate was higher than those of 0.1M feed concentration of NaCl, MgCl₂, CaCl₂ and Na₂SO₄ tested under cross-flow conditions by the commercial RO membrane, Dow Filmtec™ BW30XFR, the flux of permeate was 83 L/m².h at a pressure of 27 bar (Jang et al., 2019).

The pressure was constant with each increase in the concentration of ZIF-8 and the membrane was found to have a water permeability of 4.6 to 6 L/m².h/bar (fig. 5.11), very high compared to commercial TFC X-20TM membranes which ranged from 2.4 to 2.7 L/m².h/bar, depending on the feed concentration for polyamide membranes (Suna et al., 2013). The permeability slightly decreased from 3.4 - 4.6 L/m².h/bar (fig. 5.12), for an ion in solution. It is reported that the addition of ZIF-8 in concentration up to 30% had improved the permeability, close to that of an NF270 nanofiltration membrane which was 9.9 L/m².h/bar

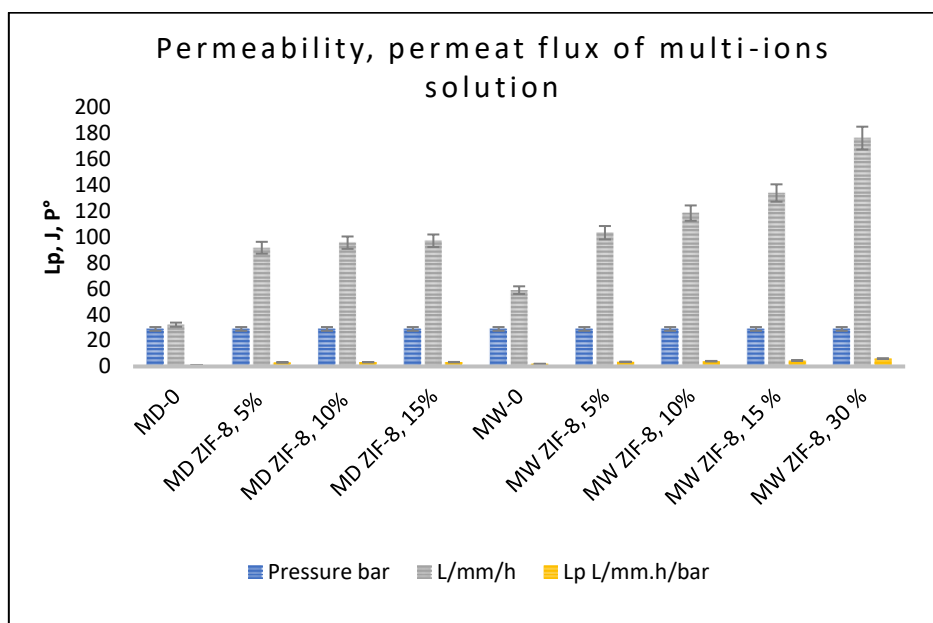


Fig 5.11: The performance of the membranes on the permeate flux of a multi-ion solution

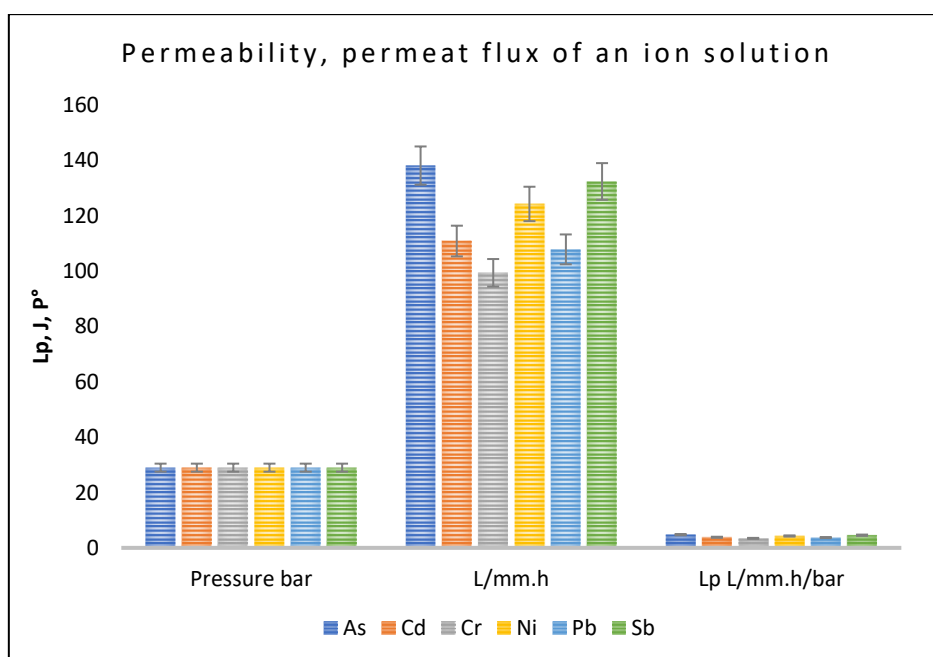


Fig 5.12: The performance of membranes on the permeate flux of an ion in solution

Table 5.3 summarize relevant key studies that show the state of the art of CS/MOF membranes with a particular focus on heavy metal removal. The cross-flow filtration on CS/ZIF-8/PAN membranes in this study show that, the salt removal is similar, while the permeate flux is higher than the results obtained by Raza et al (2019) for membrane distillation. They are also superior to the results obtained by Ma et al (2016) and Miao et al (2013), on CS/NH₂-MIL (101) (Al), CS/NH₂-MIL (101) (Cr) and CSS/PAN membranes.

Table 5.3. State of the art of previous work and for this study

Type	Membrane	HM	Removal efficiency (%)	Init Conc (ppm)	Flux (L/m ² h)	Cond	Conc Permeate	Ref
NF	PVDF/chitosan/ZIF-8	Cr VI	95.6	1000	470	1 bar	NA	Pishnamazi et al., 2020
	PVDF/chitosan/UiO-66-NH ₂		99.2	3.96	415			
	CS/NH ₂ -MIL-101(Al)	MgCl ₂	93	1000	20.8 – 60.6	15 bars	NA	Ma et al., 2016
		CaCl ₂	86.5					
		Na ₂ SO ₄	15					
		NaCl	35					
	CS/NH ₂ -MIL-101(Cr)	NaCl	33.5	2000	10.6 - 30	0.45 MPa	NA	Miao et al., 2013
	CSS/PAN	Na ₂ SO ₄	89.9		14.2 – 7.1			
		K ₂ SO ₄	93.5		18			
		MgSO ₄	45	200	20			
		KCl	48		18			
		NaCl	38		17			
		MgCl ₂	15		15			
RO	chitosan/ZIF-8	NaCl	99.5	3500	2.3	1.25 bar	NA	Raza et al., 2019
RO	Chitosan/ZIF-8	Cr	98	90	134	30 bars	0.58	This study
		Ni	96.5				1.23	
		Cd	88				2.94	
		Pb	85				3.27	
		As	13				7.92	
		Sb	24				8.19	
RO	Chitosan/ZIF-8	Cr	99	15	99	30 bars	0.45	This study
		Ni	97		124		0.61	
		Cd	97		111		0.58	
		Pd	96		108		0.65	

NA: not available

The energy consumption in RO was evaluated at 2.10^{-3} kWh/m³, it is lower than that of 1.6 k.Wh.m⁻³ produced by (Davey et al., 2022). The energy consumption is lower compared to sea water reverse osmosis (SWRO), and it has undergone huge improvements over the past five decades, specific energy consumption of SWRO has been reduced by 20 kWh.m⁻³ in 1970 to only 2.5 kWhm⁻³ in 2010. It has been estimated that 0.69 - 0.79 kWhm⁻³ could be saved by intelligent process integration with intrinsic heat recovery (Kiho et al., 2020). The low energy consumed in waste water RO has been confirmed by Van der Bruggen (2021).

5.4. Conclusions

In the present study, a TFN membrane was developed by coating an ultra-thin layer containing chitosan/ZIF-8 on wet and dry PAN supports. The presence and fixation of the thin layer on the support were revealed by SEM, EDX, FTIR and XRD analysis. The CS/ZIF-8/PAN-w MMMs were tested by three series of feed solutions of different concentrations. The TFN membranes confirm its high performance and guarantee the economic efficiency of the filtration process compared to commercial membranes. The removal of Cr^{3+} , Cd^{2+} , Ni^{2+} and Pb^{2+} ions was 98, 97, 97 and 96% respectively. The results obtained in this study confirm that CS/ZIF-8 nanocomposite membranes show an efficient performance and can be easily scaled up to industrial scale for the rejection of Cr^{3+} , Cd^{2+} , Ni^{2+} , Pb^{2+} , but are not good for the rejection of As^{3+} and Sb^{3+} .

Chapter 6. General conclusions and future perspectives

6.1. General conclusions

Two main aspects of research were of interest this thesis: The pollution and toxicological risk of pollutants on the northwestern slope of Lake Tanganyika, and the treatment of domestic and industrial wastewater by commercial membranes and a prepared membrane. To achieve this, both qualitative and quantitative analysis of the pollutant loads of effluents discharged into Lake Tanganyika were determined during which three emitters of the pollution were identified to be the central prison of Uvira - biggest emitter of organic loads with a chemical oxygen demand (COD) of 42%, followed by the general hospital (30%) and finally households (28%). The general pollutant load of Lake Tanganyika varied daily from 0.65 to 10.5 kg/d COD; 0.65 to 4.2 kg/d BOD₅; 0.05 to 0.13 kg/d ammonia nitrogen; 0.17 to 0.25 kg/d total nitrogen; 0.11 to 0.41 kg/d total phosphorus; 9.73 to 15 kg/d chloride; and 5.67 to 17 kg/d suspended solids.

Heavy metal concentrations were high for Hg and Pb in the lake with values of 1.21 and 1.42 µg/L respectively while results of effluent analysis revealed toxicity values of the effluent from the collector to be: Pb: 8; Sb: 14; Zn: 16; Cr: 7; Co: 3; Ni: 4; Cu: 2; As: 7; Sn: 0.1 and Hg: 1 µg/L, which were very high.

These potentially harmful effluents also contain volatile organic compounds originating in high concentrations from the pharmaceutical discharges of the Uvira General hospital, which include toluene, ethylbenzene, m-xylene/p-xylene, o-xylene and chloroform. Other components such as benzene, bromodichloroethane and 1,1-dichloroethane were found to be present, but at a concentration below 0.05 ppb.

An toxicology experiment conducted on wastewater samples showed lethal pollutant concentrations of the order of 0.0055 mL/mL, which killed at least 50% of fish (LC₅₀), confirming the high toxicity of the wastewater.

The commercial reverse osmosis membrane tested in this study (TFC PA membranes of the X-20™ type) showed efficiency in terms of rejection of inorganic micropollutants (at low or high concentration in the feed solution) and excessive energy consumption, but the permeate flux a little low. On the other hand, the nanofiltration membranes exhibited good ion rejection at low feed concentration, but the retention was low for high concentration feed solutions. However, the power consumption was low, and the flux was high. The modified CS/ZIF-8 MMM was considered better as it exhibited similar metal ion removal efficiency to the X-20™ TFC PA membrane, high water permeability and energy consumption was low (2.10⁻³ kWh.m⁻³) compared to the TFC PA membrane, but similar to nanofiltration.

The membrane characterization by SEM, EDX, FTIR and XRD confirms that the membrane was successfully prepared with good performance which encourage the scaling up to industrial scale.

6.2. Futures perspectives

6.2.1. Techno-economic assessment and determination of real viability in Uvira/DRC

6.2.1.1. Geo-security situation of the city of Uvira

The city of Uvira is located at the northwestern end of the Lake Tanganyika basin in the eastern Democratic Republic of Congo in the province of South Kivu. Uvira is the second largest coastal city in the province after the city of Bukavu. It is bounded to the West by the Mitumba Mountains, to the East by Lake Tanganyika, to the North by the Kawizi River located at 03° 17' 03.767" 'S; 29° 09' 37.830" E and to the South by the Sangeza River located at 029 07' 52.0" E; -03 26' 37.4" S. It was created by decree N° 13/029 of June 13, 2013 conferring the status of the city and the communes to certain agglomerations of the South Kivu Province.

The security situation in the town of Uvira is relatively calm. It is not unlikely that such situations will lead to the migration of people to the town of Uvira, which could destabilize the town economically and make it difficult to assess demographic trends.

6.2.1.2. Socio-technical aspects

The DRC is committed to achieving the Millennium Development Goals, including the goal of halving the number of people without access to adequate sanitation. It is therefore a signatory to the Ethekwini Declaration, which calls for 0.5% of GDP to be devoted to sanitation (UNEP, 2013). The lack of sanitation infrastructure in the DRC in general and South Kivu in particular requires the application of adapted and innovative technologies. In this context, the use of membrane technologies will allow the provision of a stable quality of permeate that could be locally reused or discharged into water bodies (Hasar et al., 2009; Renou et al., 2008; Cingolani et al., 2018).

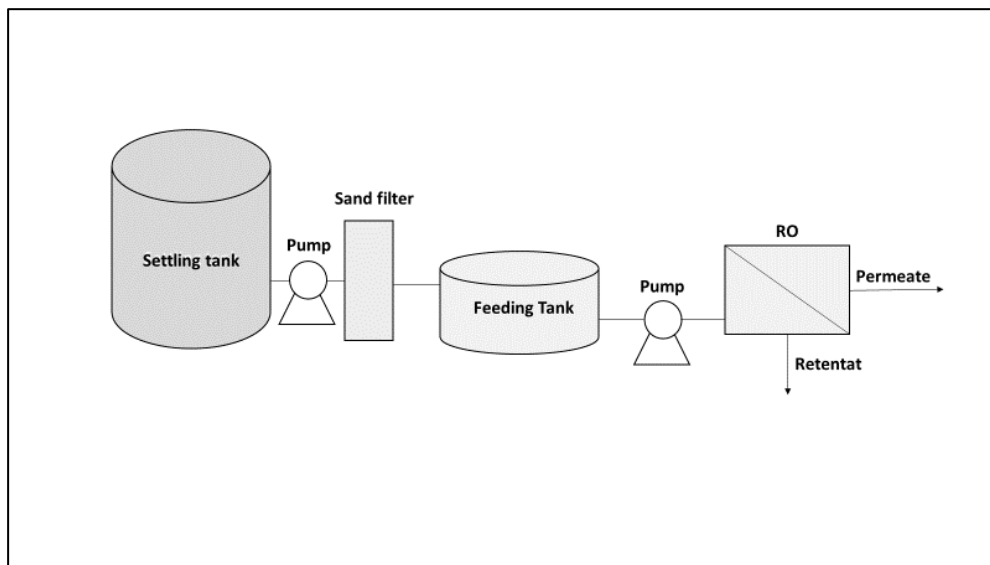
6.2.1.3. Description of the process

The industrial scale-up of the process will consist of a membrane filtration module. Upstream, the plant will be structured with a pre-treatment of raw water. The effluents are deposited in a 70 m³ tank for the decantation of large particles. After settling, the supernatant is sent to the first treatment which consists in filtering it on a sand device containing the 20 µm screen. Downstream, the filtrate will be subjected to the second treatment on the reverse osmosis membrane at a maximum pressure of 30 bars to depollute it in organic and inorganic loads.

Judd (2017) noted that multi-stage reverse osmosis improves water recovery and reduces SEC when fewer than five stages are used. The overall operation of the system is automatic and does not require much human intervention (outside of routine maintenance operations).

The membrane module will ultimately consist of one inlet (the feed) and two outlets, the portion of the fluid that has passed through the membrane (the permeate) and the portion that has been retained (the retentate).

In these operations, it is most common to work with a continuous fluid flow tangentially to the membrane to limit the accumulation of material on the surface (clogging) which is the main phenomenon antagonistic to the separation. This mode of operation (tangential filtration) allows continuous operation with a constant permeation flow but requires additional energy to ensure the flow and overcome the pressure drops ($P_r - P_a$); the tangential mode ensures high productivity (limitation of clogging) at the cost of higher investment (additional pump). Figure 6.1 shows a schematic diagram of a possible treatment process.



❖ *Technical Analysis*

These costs are estimated. They correspond to a preliminary design approach and are exclusive of taxes and fees. They do not take into account the supplies and works necessary for the supply of utilities, the services of the project owner's personnel, and the project management mission (IRH, 2010).

❖ *Principle*

The pressure applied for reverse osmosis is generally in the range of 6 to 85 bars (SUEZ, 2017):

- 55 to 85 bars for sea water type salinities,
- 15 to 40 bars for brackish water type salinities,
- 6 to 15 bars for low pressure osmosis.

Since we treat wastewater, we use a maximum pressure of 30 bars. The reverse osmosis feed rate is set at 60 m³/h

The conversion rate T_c obtained in the laboratory scale devices, can then be used to calculate the flow rates and the surface area A at industrial scale with a different driving force

T_c : Conversion rate

Q_2 : Permeate flow

Q_1 : Feed flow

$$T_c = \frac{Q_2}{Q_1} = 80 \% = 0,8 \quad (13)$$

$$Q_2 = Q_1 \times T_c = 48 \text{ m}^3/\text{h}$$

$$\frac{Q_2}{A} = T_c \times \Delta P = A = \frac{Q_2}{T_c \times \Delta P} = \frac{48 \text{ m}^3/\text{h}}{0,8 \times 28,9} = 2,07 \text{ m}^2$$

$$E_p (\text{kWh}) = \frac{P(\text{Pa}) \cdot Q_1 - Q_2 \left(\frac{\text{m}^3}{\text{h}} \right) \cdot d}{1000 \cdot n_p}$$

$$E_p (\text{kWh}) = \frac{2897890 \times 12 \times 0,99742}{1000 \times 3000} = 11,5 \text{ kWh}$$

6.2.1.4. Managing reverse osmosis retentate

Reverse osmosis retentate can have a number of environmental impacts. However, the rational management and reuse of retentate is recommended. Retentate recycling depends mainly on the feed water characteristics and chemical composition of the retentate. For industrial wastewater loaded with heavy metals, there is no recycling of heavy metals in general, but recycling by sector. The best organized processes are obviously those with the largest volumes to be treated in order to recover valuable compounds.

The study considers two retentate treatment options: The first level is recycling for the use of heavy metals, which requires additional investment in additional treatment by a Metallise electrolysis reactor. Metals can be recovered by a reactor that uses the principle of electrolysis to cause charged particles, such as heavy metals, to agglomerate on the cathode. The metal agglomerate can then be recovered and recycled (LENNTECH). The second level is the option of considering the circular economy of the process. For example, the use of retentate as mixing water to produce calcio-sulfoaluminate cement bricks for construction is recommended (Valdés et al., 2012).

RO retentate from organic matter feedwater, represents a non-toxic, cost-effective and nutrient-rich growth medium for algal culture (Rana et al., 2020).

6.2.1.5. Chemical cleaning of membranes

Fouling is one of the phenomena that slows down wastewater treatment using membrane processes (Ang et al., 2011), it leads to a drop in productivity. Membrane cleaning is extremely important for removing undesirable matter from the membrane surface and renewing its functionalization (Wilson et al., 2022). In the literature review, very little research is devoted to membrane cleaning (Porcelli et al., 2009). Qi et al (2016) showed that a solution of ethylenediaminetetraacetic acid (EDTA), sodium hydroxide (NaOH) and citric acid was better at cleaning the RO membrane and had good chemical stability.

The chemical stability of the membrane was further confirmed by cleaning with a solution of citric acid, NaOH and EDTA (Porcelli et al., 2009). Ngai et al (2011) proposed that the addition of sodium chloride (NaCl) to the solution sodium hydroxide (NaOH), ethylenediaminetetraacetic acid (EDTA) and sodium dodecyl sulphate (SDS) was ideal for cleaning the membrane resulting from the treatment of alkaline solutions, metal chelating agents, surfactants and salt. It was concluded that cleaning performance increased with increasing pH of the cleaning solution.

6.2.1.6. *Management of end-of-life of reverse osmosis membranes*

The lifetime of the RO membrane depends on several factors such as the RO membrane model, the type of pre-treatment of the plant, the quality of the feed water, the operating conditions, the location of the RO membrane and the frequency of chemical cleaning (Kharraz et al., 2021). In view of the above factors, the average life has been estimated at between 5 and 10 years for pre-treated feedwater (Kharraz et al., 2021), but it is shorter at around 12 months when the feed solution is too loaded and if there is no pre-treatment before-hand (Chang et al., 2006).

At the end of RO membrane life, reuse in MF and NF has been recommended by researchers. Khoo et al (2021) converted RO membranes to end-of-life by modifying its microfiltration properties with KMnO_4 treatment and showed that the end-of-life of RO membranes should increase considerably in the coming years. The membrane converted to MF had a NaCl rejection of around 80% and the permeability to pure water was $172.6 \text{ L/m}^2 \cdot \text{h} \cdot \text{bar}$. In addition, RO has been cited as the process that generates environmental impacts following the production of solid waste (Luiza et al., 2021).

Senán-Salinas et al (2021) have estimated that by 2025 two million membrane modules will be discarded worldwide by the desalination sector in developed countries. Kharraz et al (2021), reveal that membranes consist of plastic parts and their recycling will reduce landfill disposal. In addition, these membranes are made from petroleum-based polymers and are responsible for greenhouse gas emissions (Senán-Salinas et al., 2021). Rattanukul et al (2012) has shown that RO membranes contain 80% recycled components. However, some plastics such as polyethylene terephthalate (PET), high-density polyethylene (HDPE) and polypropylene (PP) are recyclable and represent a step forward in reducing the harmful impact on the environment.

6.2.1.7. *Economic evaluation*

The cost assessment model was developed considering both capital and operating costs. The investment costs of RO membranes were evaluated by IRH (2010) as part of a study conducted for the Agence de l'Eau Rhône Méditerranée Corse (Record, 2018).

Table 6.1. Investment costs and operating costs.

Flow to be processed (m^3/j)	Investment (k€)
< 10	60 – 90
10 – 30	90 – 130
30 – 50	130 – 180
50 – 100	180 – 250
100 – 200	250 – 350
200 – 300	350 – 450

The investment costs are related to the treatment capacity, ranging from 7.000 €/m³ /d for low capacities (< 10 m³ /d) to about 1500 €/m³ /d for flows of 200 to 300 m³ /d. To these budgets, it is necessary to integrate about 25 to 30% for installation and start-up costs, etc (IRH, 2017).

Investment costs related to the treatment capacity: 60 m³ x 250 k€ = 15000 k€.

Installation and start-up costs: 30% = 4500 k€

The cost of electricity for business in the Democratic Republic of Congo is set at 0.097 US dollars per kWh and the average price in the world is 0.129 US dollars per kWh (SNEL, 2022).

This amount has been converted at the current exchange rate equivalent to 1\$ to 1.01€

Cost of electricity consumption 0.129 \$/kWh = 0.130 €/kWh: 11.5 kWh x 0.130 €/kWh = 1.49€

The global estimate for the industrial scale-up of the RO membrane process to filter a volume of 60 m³ is worth 19501 k€.

6.2.1.8. *Financing*

The study presents the low cost of energy due to the availability of energy in the Democratic Republic of Congo, but the investment cost is too high for industrial scale-up in a low-income country like the Democratic Republic of Congo. In terms of rural and urban water supply, almost all investments are made by international donors.

Given the biological richness of Lake Tanganyika, it attracts great international scientific curiosity and is the reservoir of drinking water and domestic uses of these four riparian countries.

The governments of these four riparian countries had agreed to create an organization called "Lake Tanganyika Authority ALT", its main mission is to ensure the protection and conservation of biological diversity and sustainable use of natural resources of Lake Tanganyika and its basin, based on integrated management and cooperation between the contracting states. This project can be financed by the EU through its partner Belgian Development Agency "Enabel" which supports governments of low-income countries in international development. This support can be extended to the town halls in large cities in the DRC.

6.2.1.9. *Surface modifications for other applications*

Future prospects should focus on improving membrane performance, including modification and composite methods, to enhance the retention of As and Sb ions by the membranes. Surface functionalization is a challenge and requires the rational design of the sizes, types, and grafting

methods of functional groups. Covalent bonding of functional groups onto mechanically stable metal oxides or carbon surfaces can endow the materials with high mechanical strength, thus improving their applicability for real-world applications;

6.2.1.10. Access to sanitation in Africa and DRC

In terms of sanitation coverage, Africa has the lowest rate and includes 13% of the world's population without access to improved sanitation. In Sub-Saharan Africa, improved sanitation coverage is 30%. 70% of people without access to an improved sanitation facility live in rural areas. 51% of the rural population uses improved sanitation facilities.

The Democratic Republic of Congo (DRC) has more than 52% of Africa's water supply, but despite this tremendous potential, access is limited. 33 million people in rural areas do not have access to quality water. Despite ongoing efforts, only 52% of the population has access to an improved water source and 29% to improved sanitation facilities (Unicef DRC Report 2018, Water, Sanitation and Hygiene).

Sustainable management of water, sanitation and hygiene for health and human development, appear in several international policy forums. Among the most recent is the adoption of the United Nations Sustainable Development Goals, including indicators for Good Health and Well-Being; Clean Water and Sanitation; Sustainable Cities and Communities; and Water Life. In addition, a new report by the World Health Organization (WHO), UNICEF and the World Bank, published in October 2022, on investments in access to safe drinking water should be increased fourfold to achieve this sustainable development goal by 2030, because water is not a luxury, but a human right.

In order to remedy the problem of poor access to better quality water, the implementation of the Water and Sanitation Master Plan will enable urban and rural centers to achieve the Millennium Development Goals, with the aim of increasing the rate of access to drinking water. The installation of separate sewerage networks to the treatment center and the monitoring of effluent characterization would allow the effective scaling up of pressure treatment.

It would be interesting to conduct studies on human toxicity because it is not the same as toxicity in fish. It is important to put in place and strictly adhere to certain regulations for that use of phytosanitary that would guarantee the purity of Lake Tanganyika. Regular monitoring of the water pollution level is also recommended. Measures to prevent the sale of products containing highly toxic and poorly biodegradable compounds would be an asset.

07. APPENDICES**APPENDIX 1. CHAPTER 2**

Chapter 2. Status of the pollution of the northwestern slope of Lake Tanganyika in the Democratic Republic of Congo

1. Physical-chemical characteristics of wastewater and tributaries

Site	pH	T°	Dissolved O2	Saturation	Turbidity	STD	Conductivity
1	7.25	28.25	0	0	4650	2910	5825
1	7.42	27.43			3440	1185	2378
1	7.48	28.41	2.8	40.6	399.6	1317	2630
1	7.9	27.21	0	0	5634	1550	3075
1	8.27	25.61	0.58	7.7	4410	1266	2536
1	9.97	25.96	1.82	27	4500	1256	2514
1	7.47	27.04	2.76	39	3005	1281	2559
2	7.25	28.45	0	0	61.4	1653	3305
2	6.94	29.56			55.9	714	1427
2	7.29	28.26	2.49	35.3	61.5	580	1166
2	8.55	29.98	0.85	5.8	3438	897	1652
2	7.81	25.28	0.93	13	65.9	782	1551
2	9.69	25.66	2.36	27.9	1278	788	1577
2	6.92	27.01	1.35	18.9	34.6	846	1690
3	7.22	28.53	0	0	2715	1879	3758
3	7.55	32.93			138.5	1098	2197
3	7.3	28.34	2.76	40.8	99.9	1073	2150
3	8.19	31.89	0	0	65.7	1078	2159
3	8.7	27.75	0.59	9.3	666	1161	2337
3	9.72	29.27	2.67	40.1	558	1085	2169
3	7.13	27.14	1.21	16.4	3385	1241	2480
4	7.21	28.59	0	0	456	2210	4421
4	7.07	29.21			421	925	1853
4	7.32	28.32	2.53	37.2	99.9	665	1331
4	8.08	27.53	0	0	66.4	862	1718
4	8.68	26.68			85.3	818	1640
4	8.98	26.85	0.95	12.5	77	867	1738
4	7.5	27.75	1.2	16.6	75.9	997	1994
5	7.2	28.39	1.44	21.1	27.7	1718	3665
5	6.91	25.99			4.9	892	1781
5	7.26	28.19	3.07	43.1	16.9	685	1370
5	8.58	28.61	0.62	8.1	13.6	918	1835
5	9.03	28.74	1.69	24.9	5.6	931	1857
5	9.42	28.41	3.16	45.7	8.5	439	677
5	7.16	27.15	2.9	41.6	7.3	969	1936

Site	pH	Temp	Dissolved O2	Saturation	Turbidity	STD	Conductivity
1	8.42	24.22	2.79	40.1	492	231	463
1	9.11	23.5	0	0	2310	1247	2495
1	10.7	25.58	3.15	43.3	306	994	1992
1	8.85	31.11	0	0	3200	2340	4780
1	7.57	34.54	2.7	43.3	172	2663	5325
1	7.77	31.74	0	0	87	1232	2467
1	7.25	28.25	0	0	4650	2910	5825
1	7.42	27.43	0	0	3440	1185	2378
1	7.48	28.41	2.8	40.6	3996	1317	2630
1	7.9	27.21	0	0	5634	1550	3075
1	8.27	25.61	0.58	7.7	4410	1266	2536
1	9.97	25.96	1.82	27	4500	1256	2514
1	7.47	27.04	2.76	39	3005	1281	2559
2	8.5	24.1	0.55	7.5	72.8	335	675
2	6.88	28.09	2.66	38.4	66	1099	2196
2	10.51	25.04	3.5	49.1	66	980	1961
2	7.98	25.8	0.7	11.6	148	1164	2308
2	8.2	23.2	0	0	18	1143	2297
2	8.8	31.6	0	0	25	1745	3490
2	7.25	28.45	1.2	24	614	1653	3305
2	6.94	29.56	1.3	23	55.9	714	1427
2	7.29	28.26	2.49	35.3	61.5	580	1166
2	8.55	29.98	0.85	5.8	3438	897	1652
2	7.81	25.28	0.93	13	65.9	782	1551
2	9.69	25.66	2.36	27.9	1278	788	1577
2	6.92	27.01	1.35	18.9	34.6	846	1690
3	8.2	24.1	3.1	43.5	153	254	508
3	8.56	23.3	1.7	39.8	8.2	1061	2123
3	8.02	24.14	1.2	24	7.5	1125	2263
3	8.9	24.03	1.6	28	9.1	960	1920
3	6.79	28.19	2.43	34.7	7.1	1252	2503
3	8.93	31.87	0.59	9	2623	1842	3987
3	7.22	28.53	0	0	2715	1879	3758
3	7.55	32.93	1.6	40	138.5	1098	2197
3	7.3	28.34	2.76	40.8	99.9	1073	2150
3	8.19	31.89	0	0	65.7	1078	2159
3	8.7	27.75	0.59	9.3	666	1161	2337
3	9.72	29.27	2.67	40.1	558	1085	2169
3	7.13	27.14	1.21	16.4	3385	1241	2480
4	8.1	24.2	2.8	41	263	87	814
4	8.02	24.3	0	0	28	969	1928
4	8.65	24.58	0	0	17	1127	2262
4	8.96	23.24	5.5	79.1	11	822	1645
4	6.55	23.77	4	53.4	8.2	1987	3975
4	8.82	29.6	0	0	102	2001	4003
4	7.21	28.59	0	0	456	2210	4421
4	7.07	29.21	2.8	41	421	925	1853

4	7.32	28.32	2.53	37.2	99.9	665	1331
4	8.08	27.53	0	0	66.4	862	1718
4	8.68	26.68	1.6	40	85.3	818	1640
4	8.98	26.85	0.95	12.5	77	867	1738
4	7.5	27.75	1.2	16.6	75.9	997	1994
5	7.6	26.1	3.1	45.1	8.2	838	876
5	8.66	25.96	4.64	64.2	16	734	1569
5	7.82	24.23	3.8	48.3	12.4	953	1907
5	7.28	27.21	4.5	63.3	11.7	929	1859
5	8.6	27.9	3.6	47	9	910	1624
5	8.8	30.6	5.03	75.7	11	1751	3504
5	7.2	28.39	1.44	21.1	27.7	1718	3665
5	6.91	25.99	2.6	36	4.9	892	1781
5	7.26	28.19	3.07	43.1	16.9	685	1370
5	8.58	28.61	0.62	8.1	13.6	918	1835
5	9.03	28.74	1.69	24.9	5.6	931	1857
5	9.42	28.41	3.16	45.7	8.5	439	677
5	7.16	27.15	2.9	41.6	7.3	969	1936

Time	Flow	Ammoniacal N	Total N	Total P	SM	Chloride
	Ls ⁻	mgL ⁻	mgL ⁻	mgL ⁻	mgL ⁻	mgL ⁻
7	0.78	12.96	25.46	18.03	196	1320.6
10	1.09	11.4	21.1	60.26	128	1427.1
13	1.34	12.96	23.13	69.26	5650	1508.7
16	0.8	13.2	25.26	6.69	320	1295.75
7	0.67	6.48	26.06	7.82	230	1178.6
10	0.73	4.03	11.54	15.83	230	1327.7
13	0.79	4	11.1	10.99	220	1246.05
16	0.7	9.8	21.07	19.65	180	1256.7
7	0.92	9.02	19.98	12.16	1110	1164.4
10	0.66	16.75	26.12	5.54	790	1065
13	0.65	15.84	24.88	11.11	400	1260.2
16	0.64	10.7	18.34	29.43	660	1207

2. Characteristics of the organic load of liquid effluents

	Site 1		Site 2		Site 3		Site 4		Site 5	
	COD	BOD ₅	COD	BOD ₅	COD	BOD ₅	COD	BOD ₅	COD	BOD ₅
	2215	950	1205	730	1056	880	1198	510	479	870
	3652	1782	3401	1824	3722	2112	3883	1892	586	710
	713	250	360	30	561	48	604	15	386	47
	650	200	421	120	531	110	597	250	323	140
	713	110	360	90	561	70	604	300	386	90
	3125	1428	2987	1324	2561	1476	2827	1782	352	524
	197	1540	356	154	553	270	734	156	409	580
	4869	2775	924	2700	1627	1400	2885	2675	601	57
	10480	8200	3020	1550	1627	1400	3740	1750	361	1080
	8870	2800	6980	5750	4000	5050	2300	980	1730	920
	5130	2100	4750	8050	3210	4250	8520	8550	3740	1750
Average	3692.18	2012.27	2251.27	2029.27	1819	1551.45	2535.63	1714.54	850.27	615.27
COD/BOD ₅	1.83		1.10		1.17		1.47		1.38	
BOD ₅ /COD	0.54		0.90		0.85		0.67		0.72	

APPENDIX 2. CHAPTER 3

Chapter 3. Determination of the Toxicological Risk of Urban Waste from the City of Uvira Dumped into the North-Western Coast in Lake Tanganyika (Democratic Republic of Congo)

3.1 Monitoring of physico-chemical parameters in aquariums

lake water aquarium

Day	Temp [°C]	pH [Units]	DO% [Sat]	DO [mg/l]
1 st	25.1	9.04	103	8.5
2 nd	25	9.55	104.8	8.5
3 rd	24.73	9.86	99.8	8.27
4 th	24.73	9.13	96.8	8.02
Aquarium 10%				
1 st	24.54	9.91	97.5	8.11
2 nd	24.25	9.54	101	8.33
3 rd	24.73	9.86	99.8	8.27
4 th	24.73	9.14	66.1	5.48
Aquarium 20%				
1 st	24.23	9.88	92.1	7.7
2 nd	24.24	9	89.1	7.45
3 rd	24.25	9.88	86.2	7.14
4 th	24.26	9.92	91.6	7.68
Aquarium 30%				
1 st	24.61	9.86	80.1	6.66
2 nd	24.61	9.78	72.6	6.03
3 rd	24.55	9.87	73	6.07
4 th	24	9.7	73	6.07
Aquarium 40%				
1 st	24.61	9.97	69.4	5.77
2 nd	24.54	9.81	64.1	5.33
3 rd	24.73	9.2	65.1	5.4
4 th	24.73	9.14	66.1	5.48
Aquarium 50%				
1 st	26.04	9.9	59.4	4.82
2 nd	24.73	9.14	66.1	5.48
3 rd	24.73	9.2	65.1	5.4
4 th	24.82	9.65	55	4.55
Aquarium 60%				
1 st	24.25	9.5	60.1	4.98
2 nd	24.69	9.98	50.9	4.22
3 rd	29.8	9.62	50.9	4.22
4 th	24.54	9.74	50	4.16
Aquarium 70%				
1 st	25.11	9.82	56.1	4.62

2 nd	25.11	9.8	52	4.28
3 rd	25.11	9.76	52.4	4.31
4 th	25.1	9.67	55	4.53
Aquarium 80%				
1 st	24.66	9.79	55.4	4.59
2 nd	24.94	9.29	52.7	4.35
3 rd	24.94	9.28	52.6	4.34
4 th	24.82	9.65	55	4.55
Aquarium 90%				
1 st	24.82	9.65	46.2	3.82
2 nd	24.68	9.35	43.6	3.62
3 rd	24.74	9.64	42	3.48
4 th	24.97	9.51	47.5	3.92
Aquarium ww				
1 st	24.97	9.51	47.5	3.92
2 nd	24.97	9.57	39.2	3.24
3 rd	24.97	9.53	47.6	3.93
4 th	24.97	9.57	42.5	3.5

3.2. Description of acute toxicity test on *Oreochromis Niloticus* in aquaria expressed in mg.kg⁻¹

g Al/kg	g Cr/kg	g Mn/kg	g Ni/kg	g Cu/kg	g Zn/kg	g Pd/kg	g Ag/kg	g Cd/kg	g Pb/kg
0.107	0.001	0.028	0.002	0.011	0.137	0.00005	0.00005	0.00003	0.00131
0.048	0.001	0.023	0.002	0.002	0.069	0.00004	0.00001	-0.00001	0.00020
0.018	0.000	0.012	0.001	0.003	0.031	0.00001	0.00001	-0.00002	0.00021
1.272	0.001	0.030	0.003	0.004	0.081	0.00088	0.00014	0.00006	0.00086
0.025	0.000	0.050	0.003	0.003	0.050	0.00005	0.00002	-0.00001	0.00025
0.315	0.002	0.057	0.002	0.004	0.037	0.00003	0.00002	-0.00001	0.00095
0.762	0.002	0.070	0.002	0.003	0.039	0.00006	0.00001	0.00001	0.00059
0.480	0.001	0.041	0.003	0.004	0.058	0.00041	0.00008	0.00002	0.00066

APPENDIX 3. CHAPTER 4

Chapter 4. Evaluation of commercial reverse osmosis and nanofiltration membranes for the removal of heavy metals from surface water in the Democratic Republic of Congo

4.1. Removal of heavy metals as a function of feed concentration.

		RO			NF90			NF270		
HM	Init Conc (ppm)	P	R	R%	P	R	R%	P	R	R%
As	15	0.36	39	99.08	10	18	44.44	9.12	18	49.33
Cd		0.53	38	98.61	2.31	36	93.58	1.56	36	95.67
Cr		0.54	39	98.62	0.61	39	98.44	0.11	38	99.71
Ni		0.81	38	97.87	0.72	39	98.15	0.24	37	99.35
Pb		0.42	32	98.69	6.48	32	79.75	5.04	32	84.25
Sb		0.57	31	98.16	10	27	62.96	8.7	26	66.54
		RO			NF90			NF270		
HM	Init Conc (ppm)	P	R	R%	P	R	R%	P	R	R%
As	15	0.5	34	98.53	10	16	37.5	1.2	27	95.56
Cd		0.49	37	98.68	5.29	26	79.65	1.6	32	95
Cr		0.52	39	98.67	4.19	32	86.91	1.76	35	94.97
Ni		0.85	42	97.98	4.47	31	85.58	1.75	34	94.85
Pb		0.45	34	98.68	6.64	25	73.44	1.59	32	95.03
Sb		0.53	32	98.34	11	18	38.89	1.35	29	95.34
		RO			NF90			NF270		
HM	Init Conc (ppm)	P	R	R%	P	R	R%	P	R	R%
As	15	0.38	36	98.94	11	17	35.29	10	17	41.18
Cd		0.51	36	98.58	3.56	31	88.52	2.67	34	92.15
Cr		0.51	37	98.62	0.49	41	98.8	0.67	42	98.4
Ni		0.91	40	97.73	0.63	39	98.38	0.75	40	98.13
Pb		0.47	36	98.69	7.33	29	74.72	6.12	31	80.26
Sb		0.55	30	98.17	9.36	20	53.2	9.02	22	59
		RO			NF90			NF270		
HM	Init Conc (ppm)	P	R	R%	P	R	R%	P	R	R%
As	26	0.38	36	98.94	10	18	44.44	9.12	18	49.33
Cd		0.51	36	98.58	2.31	36	93.58	1.56	36	95.67
Cr		0.51	37	98.62	0.61	39	98.44	0.11	38	99.71
Ni		0.91	40	97.73	0.72	39	98.15	0.24	37	99.35
Pb		0.47	36	98.69	6.48	32	79.75	5.04	32	84.25
Sb		0.55	30	98.17	10	27	62.96	8.7	26	66.54
		RO			NF90			NF270		
HM	Init Conc (ppm)	P	R	R%	P	R	R%	P	R	R%
As	26	0.41	37	98.89	17	28	39.29	17	31	45.16
Cd		0.55	38	98.55	4.53	51	91.12	2.77	49	94.35
Cr		1.02	40	97.45	0.43	61	99.3	0.33	55	99.4
Ni		0.63	42	98.5	0.64	60	98.93	0.37	55	99.33
Pb		0.7	39	98.21	11	44	75	8.84	44	79.91

Sb		0.45	35	98.71	15	33	54.55	13	34	61.76
		RO			NF90			NF270		
HM	Init Conc (ppm)	P	R	R%	P	R	R%	P	R	R%
As	56	0.29	71	99.59	22	47	53.19	22	45	51.11
Cd		0.26	75	99.65	7.46	73	89.78	5.44	68	92
Cr		0.2	82	99.76	3.62	82	95.59	0.97	79	98.77
Ni		0.54	82	99.34	3.38	81	95.83	1.21	78	98.45
Pb		0.35	98	99.64	16	64	75	15	63	76.19
Sb		0.58	56	98.96	15	40	62.5	14	38	63.16
		RO			NF90			NF270		
HM	Init Conc (ppm)	P	R	R%	P	R	R%	P	R	R%
As	56	0.79	68	98.84	23	54	57.41	23	52	55.77
Cd		1.57	69	97.72	2.76	44	93.73	2.98	41	92.73
Cr		1.27	95	98.66	3.72	77	95.17	2.17	95	97.72
Ni		1.86	74	97.49	1.3	89	98.54	2.22	89	97.51
Pb		1.37	73	98.12	15	74	79.73	16	72	77.78
Sb		0.82	66	98.76	19	56	66.07	20	57	64.91

4.2. Retention of charged ions in real waters

	RO			NF270			NF90		
HM	P	R	R%	P	R	R%	P	R	R%
Co	-6.31	41.98	100	-6.2	53.55	100	-6.25	20.36	100
Al	-5.62	3.55	100	-3.19	< 1		-0.39	40.81	100
As	-0.23	8.48	100	-0.11	11	100	0.08	9.32	99.18
Ba	-4.78	46.92	100	-1.19	33.7	100	-1.19	43.95	100
Cd	-5.72	< 0.05		-5.37	< 0.05		-5.54	< 0.05	
Cr	-5.33	1.18	100	-3.25	0.71	100	-2.39	0.21	100
Cu	-6.42	11.39	100	-1.32	10.22	100	-4.68	6.59	100
Fe	10.34	92.85	88.86	8.93	128.92	93.07	25.3	142.51	82.25
Mn	7.12	454.41	98.43	68.85	929.28	92.59	89.56	748.54	88.04
Ni	-2.33	79.04	100	3.13	118.27	97.35	15.14	148.51	89.81
Pb	-4.38	< 0.05		-4.22	< 0.05		-4.37	< 0.05	
Sb	-0.96	5.64	100	-1.55	8.7	100	-0.42	10.49	100
Sn	-5.4	0.29	100	-5.28	< 0.05		-5.15	< 0.05	
Ti	-3.9	54.33	100	-0.97	59.17	100	0.16	44.07	99.63
W	2.67	16.97	84.25	2.27	4.57	50.29			
Zn	-16	363.86	100	2.31	373.84	99.38	23.8	359.59	93.38

4.3.Calculation of osmotic pressure and permeability of commercial membranes

Type	Init Conc (ppm)	p° bar	HM	Feed Conc	Ret	Per	R	T°C	T°K	Pi Ret	Pi Per	$\frac{\Delta \Pi}{\text{(atm)}}$	kpa	P Ret	P Per	Pr - Pp	PM	J	Lp			
				mol/l	mol/l * % mass	mol/L	R*T*C				$(\Pi_r - \Pi_p)$	bar		atm	atm	atm	kpa	kpa	bar	L/hm²	L/hm²/bar	
PA TFC	15	50	Cr	0.000289	0.000287	0.0000021	0.082	26	299.14	0.026	0.00034	0.0257	2.6	50	49.35	1	48.35	4898.66	4896.06	48.96	132.2	2.7
			Pb	0.000072	0.000072	0.0000008	0.082	26	299.14													
			Cd	0.000133	0.000132	0.0000015	0.082	26	299.14													
			As	0.0002	0.000197	0.0000032	0.082	26	299.14													
			Ni	0.000256	0.000252	0.0000037	0.082	26	299.14													
			Sb	0.000123	0.000121	0.0000027	0.082	26	299.14													
			somme	0.001074	0.00106	0.0000139																
NF 90	15	38	Cr	0.000289	0.000284	0.0000048	0.082	23	296.14	0.0204	0.00569	0.0147	1.49	38	37.5	1	36.5	3698.36	3696.87	36.98	375	10.14
			Ni	0.000072	0.000071	0.0000013		23	296.14													
			Cd	0.000133	0.000119	0.0000146		23	296.14													
			Pb	0.0002	0.000146	0.0000539		23	296.14													
			Sb	0.000256	0.000156	0.0000997		23	296.14													
			As	0.000123	0.000063	0.0000599		23	296.14													
			somme	0.001074	0.00084	0.0002341																
NF 270	15		Cr	0.000289	0.000285	0.0000043	0.082	23	296.14	0.0208	0.00528	0.0155	1.57	38	37.5	1	36.5	3698.36	3696.79	36.96	368.2	9.96
			Ni	0.000072	0.000071	0.0000011																
			Cd	0.000133	0.000124	0.0000097																
			Pb	0.0002	0.000162	0.0000388																
			Sb	0.000256	0.000157	0.0000994																
			As	0.000123	0.000059	0.0000643																
			somme	0.001074	0.000857	0.0002176																
Polyamide -TFC	26	50	Cr	0.000501	0.000495	0.0000062		26	299.14	0.0449	0.00074	0.0442	4.478	50	49.35	1	48.35	4898.66	4894.18	48.94	125.3	2.56
			Pb	0.000125	0.000124	0.0000016		26	299.14													
			Cd	0.000231	0.000228	0.0000033		26	299.14													
			As	0.000347	0.000343	0.0000044		26	299.14													

Chapter 7 Appendices

			Ni	0.000444	0.000433	0.0000107		26	299.14														
			Sb	0.000214	0.00021	0.0000039		26	299.14														
somme				0.001862	0.001832	0.0000302																	
NF 90	26	38	Cr	0.000501	0.000493	0.0000083	0.082	23	296.14	0.0359	0.00928	0.0267	2.701	38	37.5	1	36.5	3698.36	3695.66	36.95	329.21	8.91	
			Ni	0.000125	0.000124	0.0000018																	
			Cd	0.000231	0.000214	0.0000178																	
			Pb	0.000347	0.000265	0.000082																	
			Sb	0.000444	0.000274	0.0001697																	
			As	0.000214	0.000111	0.0001025																	
somme				0.001862	0.00148	0.0003821																	
NF 270	26		Cr	0.000501	0.000494	0.0000072	0.082	23	296.14	0.0358	0.0094	0.0264	2.677	38	37.5	1	36.5	3698.36	3695.69	36.95	326.2	8.83	
			Ni	0.000125	0.000123	0.0000021																	
			Cd	0.000231	0.00022	0.0000116																	
			Pb	0.000347	0.000285	0.0000622																	
			As	0.000444	0.000216	0.0002274																	
			Sb	0.000214	0.000137	0.0000766																	
somme				0.001862	0.001475	0.0003871																	
Polyamide -TFC	56	50	Cr	0.001079	0.00107	0.0000086	0.082	30	303.14	0.0986	0.00109	0.0975	9.88	50	49.35	1	48.35	4898.66	4888.78	48.88	97.5	1.99	
			Cd	0.00027	0.000267	0.0000036		30	303.14														
			Pb	0.000498	0.000493	0.0000056		30	303.14														
			As	0.000748	0.000742	0.0000059		30	303.14														
			Ni	0.000956	0.00094	0.0000151		30	303.14														
			Sb	0.00046	0.000455	0.0000052		30	303.14														
somme				0.004011	0.003967	0.0000439																	
NF 90	56	38	Cr	0.001079	0.00103	0.0000486	0.082	24	297.14	0.0773	0.02039	0.0569	5.77	38	37.5	1	36.5	3698.36	3692.59	36.92	307.22	8.32	
			Ni	0.00027	0.000262	0.0000079																	
			Cd	0.000498	0.000457	0.0000409																	
			Pb	0.000748	0.000567	0.0001804																	
			Sb	0.000956	0.000607	0.0003485																	
			As	0.00046	0.00025	0.0002105																	

Chapter 7 Appendices

somme			0.004011	0.003174	0.0008368	0.082	24	297.14	0.0783	0.01939	0.059	5.97	38	37.5	1	36.5	3698.36	3692.39	36.92	312.6	8.47
NF 270	56	Cr	0.001079	0.00106	0.0000189																
		Ni	0.00027	0.000265	0.0000055																
		Cd	0.000498	0.00046	0.000038																
		Pb	0.000748	0.000576	0.0001721																
		Sb	0.000956	0.000613	0.000343																
		As	0.00046	0.000242	0.0002182																
somme			0.004011	0.003215	0.0007957																

4.4. Flow and permeate flux

RO	Volume ml	Time (minute)	Q L/h		A	J (L/hm ²)
15 ppm	340	22	927.27	0.927	0.00664	139.61
26 ppm	300	20	900	0.9		135.54
56 ppm	324	21	925.71	0.925		139.31

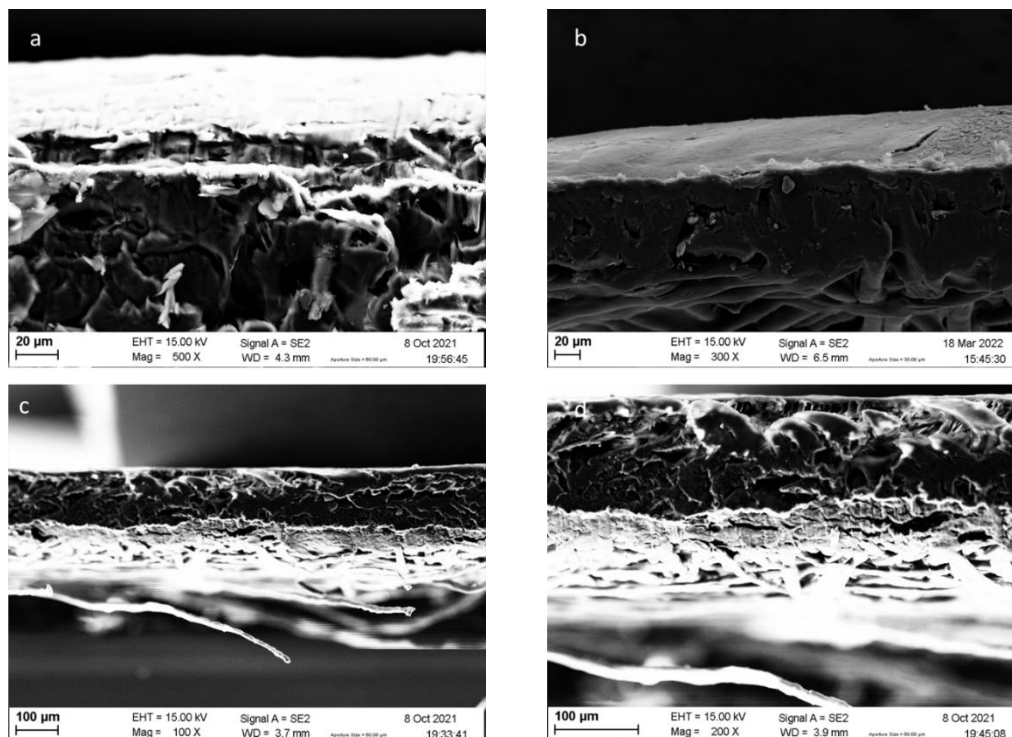
NF90	Volume ml	Time (minute)	Q L/h		A	J (L/hm ²)
15 ppm	332	8	2490	2.49	0.00664	375
26 ppm	328	9	2186.67	2.186		329.22
56 ppm	340	10	2040	2.04		307.23

NF270	Volume ml	Time (minute)	Q en l/h		A	J (L/hm ²)
15 ppm	326	8	2445	2.445	0.00664	368.22
26 ppm	325	9	2166.67	2.166		326.20
56 ppm	346	10	2076	2.076		312.65

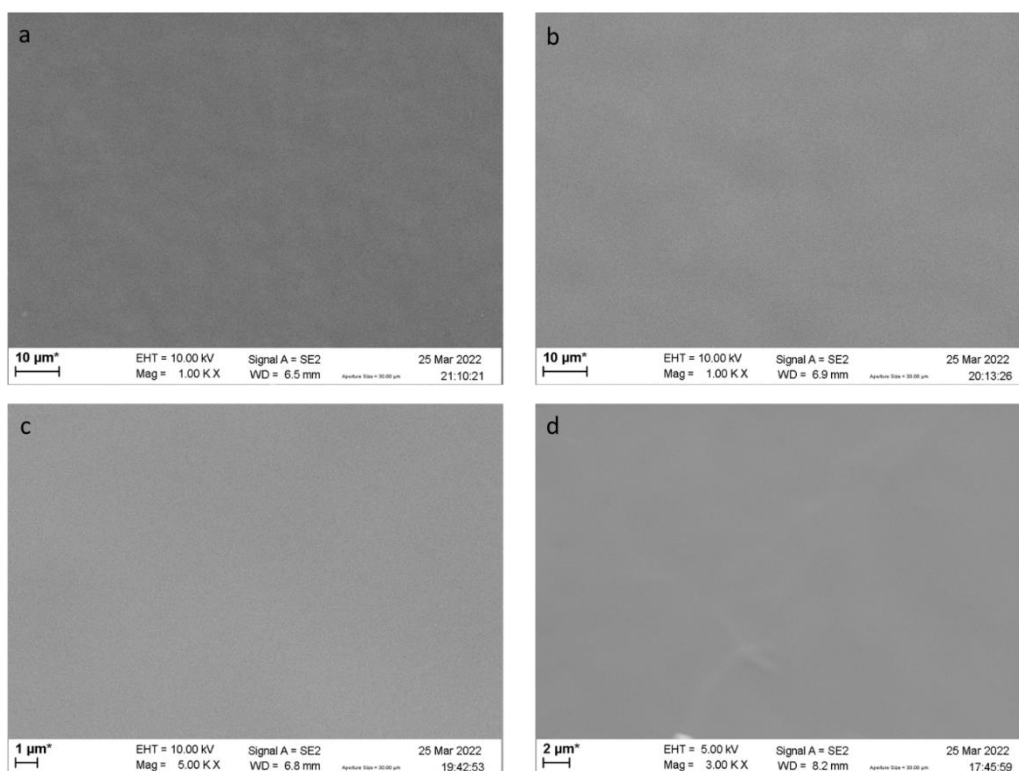
APPENDIX 4. CHAPTER 5

Chapter 5. Removal of toxic metal ions from surface water using biopolymer chitosan/ZIF-8 mixed-matrix membranes

5.1. Transversal section and Morphology



5.2. Membrane surface morphology



Membrane morphology: a: CS membrane without ZIF-8; b: CS/ZIF-8 membrane 10%; c: CS/ZIF-8 membrane 15%; d: CS/ZIF-8 membrane 30%.

5.1. Optimization of CS concentrations

CS 2%. No ZIF-8. Time: 30'			
HM	P	R	R %
As	13	15	13.33
Cd	14	21	33.33
Cr	13	27	51.85
Ni	13	25	48
Pb	14	21	33.33
Sb	14	16	12.5

CS 3%. No ZIF-8. Time: 30'			
HM	P	R	R %
As	7.47	13	42.54
Cd	3.19	23	86.13
Cr	1.91	26	92.65
Ni	2.41	27	91.07
Pb	4.05	21	80.71
Sb	5.89	13	54.69

CS 4%. No ZIF-8. Time: 30'			
HM	P	R	R %
As	10	16	37.5
Cd	4.54	29	84.34
Cr	2.62	34	92.29
Ni	3.15	34	90.74
Pb	5.92	27	78.07
Sb	9.4	17	44.71

CS 4%. No ZIF-8. Time: 60'			
HM	P	R	R %
As	10	17	41.18
Cd	5.18	30	82.73
Cr	3.84	35	89.03
Ni	4.19	36	88.36
Pb	6.64	28	76.29
Sb	10	18	44.44

CS 4%. No ZIF-8. Time: 60'			
HM	P	R	R %
As	11	15	26.67
Cd	6.76	25	72.96
Cr	4.2	36	88.33
Ni	5.15	32	83.91
Pb	9.06	26	65.15
Sb	12	18	33.33

CS 3%. Time : 90'			
HM	P	R	R %
As	11	16	31.25
Cd	5.33	28	80.96
Cr	2.86	38	92.47
Ni	3.35	35	90.43
Pb	7.45	28	73.39
Sb	11	18	38.89

5.2. Testing of dry-supported membranes

CS 3% ZIF-8 5 %			
HM	P	R	R %
As	4.87	5.3	8.11
Cd	1.66	10.4	84.04
Cr	1.12	11.4	90.18
Ni	1.22	12.7	90.39
Pb	1.85	10.6	82.55
Sb	3.78	5.29	28.54

CS 3% ZIF-8 5 %			
HM	P	R	R %
As	5.34	7.76	31.19
Cd	1.33	14.9	91.07
Cr	0.66	13.8	95.22
Ni	0.97	20.9	95.36
Pb	1.78	15	88.13
Sb	4.81	7.81	38.41

CS 3% ZIF-8 5 %			
HM	P	R	R %
As	5.29	7.4	28.51
Cd	1.25	15.6	91.99
Cr	0.46	14.1	96.74
Ni	0.65	19.3	96.63
Pb	1.69	15	88.73
Sb	5.02	7.58	33.77

CS 3% ZIF-8 5 %			
HM	P	R	R %
As	9.19	12	23.42
Cd	3.38	20	83.1
Cr	1.73	24	92.79
Ni	2.03	24	91.54
Pb	4.62	20	76.9
Sb	8.43	13	35.15

CS 3% ZIF-8 7.5 %			
HM	P	R	R %
As	5.99	8	25.13
Cd	2.53	16.9	85.03
Cr	2.19	19.6	88.83
Ni	2.22	19.9	88.84
Pb	2.98	16.8	82.26
Sb	5.38	8.46	36.41

CS 3% ZIF: 7.5%			
HM	P	R	R %
As	12.3	13.8	10.86
Cd	6.73	27.1	75.16
Cr	3.52	35.5	90.08
Ni	4.25	34	87.5
Pb	7.98	25.1	68.2
Sb	12	17.1	29.82

CS 3% ZIF-8 10 %			
HM	P	R	R %
As	4.02	5.94	32.32
Cd	2.06	10	79.4
Cr	1.54	9.1	83.08
Ni	2.13	15.3	86.08
Pb	2.42	10.3	76.5
Sb	3.32	5.36	38.06

CS 3% ZIF-8 10 %			
HM	P	R	R %
As	4.66	5.65	17.52
Cd	1.32	10.7	87.66
Cr	0.71	9.23	92.31
Ni	1.14	13.9	91.8
Pb	1.51	10.7	85.89
Sb	3.43	5.74	40.24

CS 3% ZIF-8 10 %			
HM	P	R	R %
As	5.82	7.59	23.32
Cd	1.63	14.5	88.76
Cr	0.82	16.3	94.97
Ni	0.98	17.2	94.3
Pb	2.11	14.3	85.24
Sb	5.49	8.25	33.45

CS 3% ZIF-8 10 %			
HM	P	R	R %
As	11	15	26.67
Cd	4.42	27	83.63
Cr	1.78	35	94.91
Ni	2.01	32	93.72
Pb	6.06	27	77.56
Sb	11	17	35.29

CS 3% ZIF-8 10%			
HM	P	R	R %
As	12.7	13.7	7.29
Cd	7.28	26.1	72.1
Cr	2.77	35.6	92.21
Ni	4.2	33.2	87.34
Pb	8.44	24.4	65.4
Sb	12.3	16.4	25

5.3. Testing of wet-supported membranes

CS 3% ZIF-8 5%			
HM	P	R	R%
As	5.45	6.67	18.29
Cd	1.2	12.3	90.24
Cr	0.24	13.8	98.26
Ni	0.65	20.1	96.77
Pb	1.48	12.1	87.77
Sb	5.15	6.95	25.9

CS 3% ZIF-8 5%			
HM	P	R	R %
As	12.2	13.9	12.23
Cd	6.19	27.7	77.65
Cr	1.66	36.8	95.48
Ni	2.67	35.4	92.45
Pb	7.55	25.9	70.84
Sb	12.1	16.6	27.1

CS 3% ZIF-8 5%			
HM	P	R	R %
As	5.75	6.92	16.91
Cd	2.39	14.1	83.05
Cr	1.45	16.1	90.99
Ni	1.66	16.5	89.94
Pb	2.71	14.3	81.05
Sb	5.3	7.46	28.95

CS 3% ZIF-8 7.5%			
HM	P	R	R%
As	6.6	7.51	12.12
Cd	2.18	15.5	85.94
Cr	0.99	18.2	94.56
Ni	1.37	20.4	93.28
Pb	2.5	15.2	83.55
Sb	6.15	8.12	24.26

CS 3% ZIF-8 7.5%			
HM	P	R	R %
As	12.3	13.9	11.5
Cd	5.61	27	79.2
Cr	1.73	35.3	95.1
Ni	2.47	33.7	92.7
Pb	6.97	25	72.1
Sb	11.8	16.6	28.9

CS 3% ZIF-8 7.5 %			
HM	P	R	R %
As	6.44	7.53	14.48
Cd	2.27	17.1	86.73
Cr	1.43	20	92.85
Ni	1.5	20.4	92.65
Pb	2.76	17.1	83.86
Sb	5.46	8.13	32.84

CS 3% ZIF-8 10%			
HM	P	R	R%
As	7.05	7.73	8.8
Cd	2.32	16.6	86.02
Cr	0.79	20.5	96.15
Ni	1.12	21.4	94.77
Pb	2.82	16.3	82.7
Sb	6.48	8.28	21.74

CS 3% ZIF-8 10%			
HM	P	R	R %
As	13.1	14	6.42
Cd	7.3	27.7	73.64
Cr	2.02	38.3	94.72
Ni	3.48	35.8	90.27
Pb	8.6	26.4	67.42
Sb	12.8	16.5	22.42

CS 3% ZIF-8 15%			
HM	P	R	R%
As	6.88	7.86	12.47
Cd	2.09	17.4	87.99
Cr	0.41	21.6	98.1
Ni	0.76	21.6	96.48
Pb	2.51	16.9	85.15
Sb	6.48	8.52	23.94

CS 3% ZIF-8 15%			
HM	P	R	R %
As	7.92	8.29	4.46
Cd	2.94	20.2	85.44
Cr	0.58	27.7	97.9
Ni	1.23	25.5	95.17
Pb	3.27	19.2	82.96
Sb	8.19	10.6	22.73

CS 3% ZIF-8§ 15 %			
HM	P	R	R%
As	7.18	7.79	7.83
Cd	2.90	17.1	83.04
Cr	0.67	21.4	96.87
Ni	1.30	20.6	93.69
Pb	3.05	16.0	80.94
Sb	5.85	7.97	26.60

CS 3% ZIF-8 30%			
HM	P	R	R%
As	5.86	6.07	3.46
Cd	1.76	11.2	84.29
Cr	0.37	12.8	97.11
Ni	0.86	14.3	93.99
Pb	1.81	10.8	83.24
Sb	5.14	5.79	11.23

5.4. Calculation of the osmotic pressure and the permeability of MMM

Membrane	Init Conc (ppm)	HM	(Conc	Ret	Per	T°C	T°K	Pi Ret	PI Per	$\Delta\Pi$ (atm)	kpa	P Ret	P Per	Pr - Pp		PM		J	Lp	
			mol/l	mol/l * % mass	mol/L	23	296.14	R*T*C		($\Pi_r - \Pi_p$)		bar	atm	atm	atm	kpa	kpa	bar	L/hm ²	L/hm ² /bar
CS 2% No ZIF-8. 30'	15	As	0.0002	0.000026	0.0001736	23	296.14	0.0093	0.0168	0.0075	0.756	30	29.6	1	28.6	2897.89	2897.13	28.97	37.95	1.31
		Cd	0.000133	0.000044	0.000089	23	296.14													
		Cr	0.000289	0.000149	0.0001393	23	296.14													
		Ni	0.000256	0.000122	0.0001331	23	296.14													
		Pb	0.000072	0.000024	0.0000483	23	296.14													
		Sb	0.000123	0.000015	0.0001078	23	296.14													
			0.001074	0.000383	0.0006912															
CS 3% No ZIF-8. 30'	15	As	0.0002	0.000069	0.000131	30	303.14	0.0191	0.0076	0.0114	1.155	30	29.6	1	28.6	2897.89	2896.74	28.97	32.22	1.11
		Cd	0.000133	0.000106	0.0000274	30	303.14													
		Cr	0.000289	0.000261	0.0000277	30	303.14													
		Ni	0.000256	0.000223	0.0000323	30	303.14													
		Pb	0.000072	0.000052	0.0000196	30	303.14													
		Sb	0.000123	0.000054	0.000069	30	303.14													
			0.001074	0.000767	0.000307															
CS 4% No ZIF-8. 30'	15	As	0.0002	0.000075	0.0001252	30	303.14	0.0198	0.0069	0.013	1.317	30	29.6	1	28.6	2897.89	2896.57	28.97	42.46	1.47
		Cd	0.000133	0.000112	0.000021	30	303.14													
		Cr	0.000289	0.000266	0.0000225	30	303.14													
		Ni	0.000256	0.000232	0.0000238	30	303.14													
		Pb	0.000072	0.000056	0.0000159	30	303.14													
		Sb	0.000123	0.000055	0.0000682	30	303.14													
			0.001074	0.000797	0.0002766															
CS 4%. No ZIF-8. 90'	15	As	0.0002	0.000074	0.0001262	32	305.14	0.0186	0.0082	0.0104	1.053	30	29.6	1	28.6	2897.89	2896.84	28.97	33.13	1.14
		Cd	0.000133	0.000105	0.000028	32	305.14													
		Cr	0.000289	0.000248	0.0000405	32	305.14													

Chapter 7 Appendices

		Ni	0.000256	0.000217	0.0000384	32	305.14													
		Pb	0.000072	0.000052	0.0000195	32	305.14													
		Sb	0.000123	0.000046	0.0000764	32	305.14													
			0.001074	0.000745	0.000329															
CS 4%. No ZIF-8. 60'	15	As	0.0002	0.000082	0.0001182	32	305.14	0.0196	0.0072	0.0124	1.256	30	29.6	1	28.6	2897.89	2896.63	28.97	27.83	0.96
		Cd	0.000133	0.00011	0.0000231	32	305.14													
		Cr	0.000289	0.000257	0.0000318	32	305.14													
		Ni	0.000256	0.000225	0.0000307	32	305.14													
		Pb	0.000072	0.000055	0.0000174	32	305.14													
		Sb	0.000123	0.000054	0.0000685	32	305.14													
			0.001074	0.000784	0.0002897															
CS 3% No ZIF-8. 90'	15	As	0.0002	0.000062	0.0001378	32	305.14	0.0193	0.0076	0.0116	1.175	30	29.6	1	28.6	2897.89	2896.72	28.97	25.75	0.89
		Cd	0.000133	0.000108	0.0000255	32	305.14													
		Cr	0.000289	0.000267	0.000022	32	305.14													
		Ni	0.000256	0.000231	0.0000246	32	305.14													
		Pb	0.000072	0.000053	0.0000193	32	305.14													
		Sb	0.000123	0.000047	0.0000754	32	305.14													
			0.001074	0.000769	0.0003046															
CS 3%. ZIF 0.5%. 30'	15	As	0.0002	0.00004	0.0001602	28	301.14	0.0175	0.0091	0.0084	0.851	30	29.6	1	28.6	2897.89	2897.04	28.97	109.93	3.79
		Cd	0.000133	0.000098	0.0000354	28	301.14													
		Cr	0.000289	0.000265	0.000024	28	301.14													
		Ni	0.000256	0.00022	0.0000351	28	301.14													
		Pb	0.000072	0.000047	0.0000249	28	301.14													
		Sb	0.000123	0.000036	0.000087	28	301.14													
			0.001074	0.000707	0.0003666															
CS 3%. ZIF 1%. 30'	15	As	0.0002	0.000042	0.0001574	26	299.14	0.0175	0.0089	0.0086	0.871	30	29.6	1	28.6	2897.89	2897.02	28.97	134.18	4.63
		Cd	0.000133	0.000099	0.0000342	26	299.14													
		Cr	0.000289	0.000263	0.000026	26	299.14													
		Ni	0.000256	0.00022	0.0000358	26	299.14													

Chapter 7 Appendices

		Pb	0.000072	0.000048	0.000024	26	299.14													
		Sb	0.000123	0.000038	0.0000848	26	299.14													
			0.001074	0.000712	0.0003623															
CS 3%. ZIF 5%	15	As	0.0002	0.000057	0.0001432	32.5	305.64	0.0198	0.0072	0.0126	1.276	30	29.6	1	28.6	2897.89	2896.61	28.97	34.78	1.2
		Cd	0.000133	0.000116	0.0000169	32.5	305.64													
		Cr	0.000289	0.00027	0.0000182	32.5	305.64													
		Ni	0.000256	0.000239	0.0000169	32.5	305.64													
		Pb	0.000072	0.000059	0.0000127	32.5	305.64													
		Sb	0.000123	0.000045	0.0000775	32.5	305.64													
			0.001074	0.000788	0.0002854															
CS 3%. ZIF 10%	15	As	0.0002	0.00005	0.0001502	32	305.14	0.0191	0.0078	0.0113	1.144	30	29.6	1	28.6	2897.89	2896.75	28.97	37.5	1.29
		Cd	0.000133	0.000113	0.00002	32	305.14													
		Cr	0.000289	0.000263	0.000026	32	305.14													
		Ni	0.000256	0.000232	0.000023	32	305.14													
		Pb	0.000072	0.000058	0.0000138	32	305.14													
		Sb	0.000123	0.000045	0.0000776	32	305.14													
			0.001074	0.000763	0.0003107															
CS 3% ZIF 5% M H	15	As	0.0002	0.000036	0.0001642	27	300.14	0.0187	0.0078	0.0109	1.104	30	29.6	1	28.6	2897.89	2896.79	28.97	103.16	3.56
		Cd	0.000133	0.000116	0.0000173	27	300.14													
		Cr	0.000289	0.000274	0.0000145	27	300.14													
		Ni	0.000256	0.000238	0.0000179	27	300.14													
		Pb	0.000072	0.00006	0.0000116	27	300.14													
		Sb	0.000123	0.000033	0.00009	27	300.14													
			0.001074	0.000758	0.0003155															
CS 3% ZIF 7.5%	15	As	0.0002	0.000034	0.0001662	27	300.14	0.0184	0.008	0.0104	1.053	30	29.6	1	28.6	2897.89	2896.84	28.97	95.78	3.31
		Cd	0.000133	0.000114	0.0000187	27	300.14													
		Cr	0.000289	0.000265	0.0000231	27	300.14													
		Ni	0.000256	0.000235	0.0000205	27	300.14													
		Pb	0.000072	0.00006	0.0000123	27	300.14													

Chapter 7 Appendices

		Sb	0.000123	0.000038	0.000085	27	300.14													
			0.001074	0.000748	0.0003259															
	15	As	0.0002	0.000036	0.0001642	25	298.14	0.0185	0.0078	0.0107	1.084	30	29.6	1	28.6	2897.89	2896.81	28.97	118.29	4.08
		Cd	0.000133	0.000116	0.0000173	25	298.14													
		Cr	0.000289	0.000268	0.0000202	25	298.14													
CS 3%		Ni	0.000256	0.000238	0.0000179	25	298.14													
ZIF 10%		Pb	0.000072	0.00006	0.0000116	25	298.14													
		Sb	0.000123	0.000037	0.0000863	25	298.14													
			0.001074	0.000756	0.0003176															
	15	As	0.0002	0.000024	0.0001762	27	300.14	0.0187	0.0077	0.011	1.114	30	29.6	1	28.6	2897.89	2896.78	28.97	133.78	4.62
		Cd	0.000133	0.000117	0.000016	27	300.14													
		Cr	0.000289	0.000283	0.0000058	27	300.14													
CS 3%		Ni	0.000256	0.000245	0.0000102	27	300.14													
ZIF 15 %		Pb	0.000072	0.000061	0.0000109	27	300.14													
		Sb	0.000123	0.000029	0.0000937	27	300.14													
			0.001074	0.000761	0.0003128															
	15	As	0.0002	0.000022	0.0001782	27	300.14	0.0178	0.0087	0.0091	0.922	30	29.6	1	28.6	2897.89	2896.97	28.97	91.56	3.16
		Cd	0.000133	0.000105	0.000028	27	300.14													
CS 3%		Cr	0.000289	0.000271	0.0000173	27	300.14													
ZIF 5%		Ni	0.000256	0.000232	0.000023	27	300.14													
MS		Pb	0.000072	0.000052	0.0000195	27	300.14													
		Sb	0.000123	0.000037	0.0000863	27	300.14													
			0.001074	0.000721	0.0003525															
	15	As	0.0002	0.000022	0.0001782	29	302.14	0.0176	0.009	0.0086	0.901	30	29.6	1	28.6	2897.89	2896.99	28.97	78.31	2.7
		Cd	0.000133	0.000102	0.0000307	29	302.14													
CS 3%		Cr	0.000289	0.000268	0.0000202	29	302.14													
ZIF 7.5%		Ni	0.000256	0.00023	0.0000256	29	302.14													
MS		Pb	0.000072	0.00005	0.0000217	29	302.14													
		Sb	0.000123	0.000035	0.0000875	29	302.14													

Chapter 7 Appendices

		0.001074	0.00071	0.000364																
CS 3% ZIF 10% MS	15	As	0.0002	0.00014	0.0000601	26	299.14	0.0199	0.0065	0.0134	1.357	30	29.6	1	28.6	2897.89	2896.53	28.97	95.48	3.3
		Cd	0.000133	0.000097	0.000036	26	299.14													
		Cr	0.000289	0.000268	0.0000202	26	299.14													
		Ni	0.000256	0.000227	0.0000282	26	299.14													
		Pb	0.000072	0.000047	0.0000249	26	299.14													
		Sb	0.000123	0.000029	0.0000937	26	299.14													
			0.001074	0.000811	0.0002631															
CS 3% ZIF 15% MS	15	As	0.0002	0.00008	0.0001202	25	298.14	0.0194	0.0068	0.0126	1.276	30	29.6	1	28.6	2897.89	2896.61	28.97	76.95	2.66
		Cd	0.000133	0.000109	0.000024	25	298.14													
		Cr	0.000289	0.00028	0.0000087	25	298.14													
		Ni	0.000256	0.00024	0.0000154	25	298.14													
		Pb	0.000072	0.000056	0.0000159	25	298.14													
		Sb	0.000123	0.000028	0.0000949	25	298.14													
			0.001074	0.000795	0.000279															
CS 3% ZIF-8 30%	15	As	0.0002	0.000068	0.0001322	0.082	21	0.0187	0.0072	0.0115	1.165	30	29.6	1	28.6	2897.89	2896.73	28.97	176.13	6.08
		Cd	0.000133	0.000112	0.0000214	0.082	21													
		Cr	0.000289	0.00028	0.0000087	0.082	21													
		Ni	0.000256	0.00024	0.0000154	0.082	21													
		Pb	0.000072	0.00006	0.0000123	0.082	21													
		Sb	0.000123	0.000013	0.0001097	0.082	21													

8.5. Calculation of the energy consumption in RO

Parameter	Value	Energy
Pressure	2896770	204. 10^{-5}
Q	10263. 10^{-7}	
d	0.99742	
np	1450	
Constant	1000	

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List of publications

Lumami, K.V., Alvarez M.G., Sefidi V.S., Tamungang, E.B.N., Ndikumana, T., Musibono, D.D., Van der Bruggen B., Luis, P. Evaluation of Commercial Reverse Osmosis and Nanofiltration Membranes for the Removal of Heavy Metals from Surface Water in the Democratic Republic of Congo. *Clean Technologies*. 2022; 4(4):1300-1316. <https://doi.org/10.3390/cleantechnol4040080>

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Research papers under preparation

Lumami, K.V., Xu, X., Hartanto, Y., Chergaoui, S., Van der Bruggen, B., Luis, P. Removal of heavy metals from surface water using biopolymer chitosan/ZIF-8 mixed-matrix membranes

Lumami, K.V., Luis, P. Removal of heavy metals from surface water using reverse osmosis, article submitted

Communication in international conferences

Participation in the conference Micropollutants in water, a challenge for the living, which took place on July 11, 2022 online, BORDEAUX, France.

Lumami, K.V., Musibono, D.D., Luis, P. Removal Evaluation of commercial reverse osmosis and nanofiltration membranes for the removal of heavy metals in water sources from the Democratic Republic of Congo, Water Symposium (Aula Magna, Louvain-La-Neuve (BE), du 31/03/2022 au 01/04/2022).

Lumami, K.V., Alvarez, M.G., Sefidi, V.S., Tamungang, E.B.N., Ndikumana, T., MUSIBONO, D.D., Van der Bruggen, B., Luis, P. Evaluation of commercial reverse osmosis and nanofiltration membranes for the removal of heavy metals in water sources from the Democratic Republic of Congo. Participation and presentation of a poster at the third international conference "AMSIC 3" on membrane processes, November 2-5, 2021 in Senegal

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Curriculum Vitae (with publication list)

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EDUCATION

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July 2012 - 2014: Masters in Environmental Science; Orientation: Prevention and remediation of pollution, Université du Burundi.

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LIST OF PUBLICATIONS

Lumami Kapepula Vercus, Mar García Alvarez, Vida Sang Sefidi, Estella Buleng Njoyim Tamungang, Théophile Ndikumana, Dieu-Donné Musibono, Bart Van Der Bruggen, and Patricia Luis. 2022. "Evaluation of Commercial Reverse Osmosis and Nanofiltration Membranes for the Removal of Heavy Metals from Surface Water in the Democratic Republic of Congo" *Clean Technologies* 4, no. 4: 1300-1316. <https://doi.org/10.3390/cleantechnol4040080>.

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RESEARCH PAPERS UNDER PREPARATION

Lumami, KV.; Xu, X.; Hartanto, Y.; Sara Chergaoui, S.; Der Bruggen, BVD.; Patricia Luis, P. Removal of heavy metals from surface water using biopolymer chitosan/ZIF-8 mixed-matrix membranes.

Lumami, KV.; Luis, P. Removal of heavy metals from surface water using reverse osmosis, The book chapter.

INTERNATIONAL CONFERENCES

Participation in the conference Micropollutants in water, a challenge for the living, which took place on July 11, 2022 online, BORDEAUX, France.

Lumami, KV.; Xu, X.; Hartanto, Y.; Chergaoui, S.; Bruggen, BVD.; Luis, P. Removal of heavy metals from surface water using biopolymer chitosan/ZIF-8 mixed-matrix membranes, Presented to the second international symposium on Nanomaterials and Membrane Science for Water, Energy and Environment and Reuse in the face of water scarcity, Tangier-Morocco June 1st & 2nd 2022.

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Participation and poster presentation on evaluation of commercial reverse osmosis and nanofiltration membranes, used for the release of heavy metals in wastewater, at the international congress on Membrane and Filtration Technologies for Sustainable Development from 2-5 November 2021 in Dakar / Senegal.

Participation in the 1st International Symposium on the Water, Energy-Food-Habitat nexus from December 12 to 14, 2018, Ouagadougou –BURKINA FASO.

Participation in the International Symposium on the Quality and Uses of Water Resources in Central and East Africa: Impacts on Ecosystems and Health, October 21 to 26, 2019, Conference Room of the CEMAC Palace, Bangui, Central African Republic.

Water resources quality and uses in Central and Eastern Africa: the impacts over ecosystems and health. December 12-17, 2017, Conference Room of the CEMAC Palace, Bangui, Central African Republic.

International Water-Environment-Climate'2018 Symposium and Training (E²C-2018): Water Resources, Environment and Climate Change (Water Sciences, Climate and Environment (STEE'2018) from September 30 to October 02 2019 Hammamet (Tunisia).