Study of Ultrafiltration and Reverse Osmosis Membrane Processes for Advanced Treatment and Recycling of Textile Effluent in Bangladesh

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Certification

We, the undersigned, certify that Mesbah Ahmad, candidate for the degree of Master of Science in Chemical Engineering, has presented his thesis on the subject "Study of Ultrafiltration and Reverse Osmosis Membrane Processes for Advanced Treatment and Recycling of Textile Effluent in Bangladesh". The thesis is acceptable in form and content. The student demonstrated a satisfactory knowledge of the field covered by this thesis in an oral examination held on June 13, 2021.

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Candidate's Declaration

It is hereby declared that this thesis or any part of it has not been submitted elsewhere for the award of any degree or diploma.

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Dedication

I dedicate this thesis work to my late mother, Latifa Ali, the wisest and smartest woman I have ever known.

Abstract

Textile and apparel industries are the major role-players in the fast-growing economy of Bangladesh. However, the textile sector consumes a large amount of water for various wet processing operations, and hence, has a high water-footprint. Currently 98% of the water used by local textile factories is groundwater, which is causing depletion of ground water levels at a high rate. Considering the gravity of groundwater crisis in future, Bangladesh Government and international brands and retailers are advocating local textile factories to reuse textile effluents and implement ZLD (zero liquid discharge) option in the upcoming years. However, the existing study about the technical and investment aspects of using advanced treatment technologies does not provide enough insights for reusing textile effluents or implement ZLD. In this work, the performance of ultrafiltration and reverse osmosis was studied as advanced treatment processes for recycling textile effluents of Bangladesh. For advanced treatment, a mobile setup of ultrafiltration (UF) and reverse osmosis (RO) unit was used at six selected factory premises to further treat ETP (effluent treatment plant) treated water. The advanced treatment was carried out for three different permeate to reject flowrate ratios to observe changes in the permeate and reject water quality and contaminants removal efficiency. A time dynamic analysis was also done to further demonstrate the performance of advanced treatment of conventional ETP treated water of a denim washing factory. The corresponding technical and economic issues of water recycling and reusing were also analyzed. The study showed that the treated water quality was well within the standards of groundwater quality that is being used as process water in the local factories. It also showed that it was possible to reduce blue water footprint up to 80% based on the operating configuration of the RO unit. It also demonstrated that it was possible to reduce grey water footprint from 154.6 m³/day (7.4%) to 15568.6 m³/day (95%) for the selected industries. Finally, the study showed that the advanced water treatment cost by ultrafiltration and reverse osmosis ranged from 24.06 BDT/m³ to 24.29 BDT/m³ for the selected textile industries.

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Chapter 1: Introduction

1.1 Background of the Study

Textile industries have been the backbone of the economy of Bangladesh for the last three decades. Bangladesh exports almost 750 textile and readymade garment products in global market, which includes USA, Europe, China and Japan's market [1]. The ready-made garment (RMG) industry has occupied a unique position in the economy of Bangladesh. It is the largest exporting industry in Bangladesh and in the last few decades, it has experienced phenomenal growth [2].

Textile industries in Bangladesh consume a huge amount of water every day which is extensively used in wet processing operations. But The gap between the water demand of textile wet processing factories and availability of high-quality water is continuously increasing. Currently, 98% of the water used by these industries is groundwater [3]. Recently It has been reported that in Dhaka city, a permanent declining trend of groundwater level was observed due to excessive withdrawal for city water supply and around Dhaka city for industrial withdrawal [4]. And the situation of other industrial zones near to Dhaka is not any different. So, some alternative sources of freshwater would be beneficial for these areas.

In 2016, textile industries in Bangladesh produced about 1.80 million metric tons of fabrics and generated around 217 million m³ of wastewater. In 2021, textile industries in Bangladesh will produce about 2.91 million metric tons of fabrics and around 349 million $m³$ of wastewater will be produced using conventional dyeing practices [2]. Local textile factories use traditional chemical, physico-chemical and biological effluent treatment plants (ETPs) to comply with local and international standards for treated water parameters. Textile industries of Bangladesh treat their wastewater with effluent treatment plants (ETP) and then release them in the environment according to the guideline given in ECR 1997. But instead of completely discharging this huge volume of water into the environment, reusing this effluent water in the industries as process water would certainly help to decrease the dependency on groundwater.

Nowadays, membrane processes have become the technology-of-choice as tertiary treatment of ETP treated water and to provide reusable water to factories. Ultrafiltration, Nanofiltration and reverse osmosis membranes are becoming popular as tertiary treatment units. Ultrafiltration membranes are used for many applications. They can remove dissolved compounds like colloids, carbohydrates, proteins, etc. that have high molecular weight. UF membranes do not remove sugar or salt. However, they can remove viruses and can produce rinse water of high purity in the industries [5].

Reverse osmosis is a technology that uses a semipermeable membrane to remove contaminants from water. It can remove dissolved solids, organics, pyrogens, submicron colloidal matter, colour, nitrate and bacteria from water. Feedwater is delivered under pressure through the semi-permeable membrane, where water permeates the minute pores of the membrane and is delivered as purified water called permeate water [5].

Membrane processes having pressure as driving force such as ultrafiltration (UF), reverse osmosis (RO) and nanofiltration (NF) are increasingly being used to produce high quality water and have been applied over a wide variety of fields such as the pharmaceutical industry, dairy and food technology, pulp and paper industry, textile industry, and a wide diversity of other new applications. A study showed that by combining recycled wastewater and other water resources in management, the combined ecological footprint of water, sewage and drainage systems could be potentially reduced by more than 25% [6]. The application of membrane separation processes like ultrafiltration (UF) and reverse osmosis (RO) provide the industries with a technology to achieve water quality limits, and produce reusable water and has proved to be an effective process in concentrating the bulk of pollutant into small liquid volume for further disposal [7].

1.2 Significance of the Study

Due to easy access to pumped wells, groundwater production for municipal, industrial, and agricultural supplies has accelerated worldwide in the last half-century. But groundwater depletion is the inevitable and natural consequence of withdrawing water from an aquifer. As depletion continues worldwide, its impacts worsen, signifying the need for analysis of the problem and its possible solutions [8]. Currently, the whole world is suffering from the crisis of fresh water. Groundwater is considered a suitable source of freshwater worldwide. But the groundwater level is continuously declining all over the world because of excessive consumption. The whole world is looking for a suitable alternative or management of existing water resources so that the challenge can be mitigated. Bangladesh is pumping up a lot of groundwater for industrial and sanitary use

purposes at an alarming rate. So, it is certain that we will be facing extreme adversities in the future to collect freshwater for various uses and face global climate change like the rise of seawater level. This study was conducted keeping this picture in mind. If even a small portion of the industryeffluent water can be reused that is continuously being discharged into the environment, it will make a huge impact in conserving groundwater level. In this study, the potential of UF and RO as tertiary treatment units for ETP treated water and their performance for producing reusable water in wet processing operations were analyzed. This study will allow us to understand how successfully effluent water from textile industries can be reused as process water. It will also show the capital and operating costs associated with the advanced treatment of the ETP treated water from the textile industries. Considering the gravity of the groundwater crisis in the future, international brands and retailers are advocating local textile factories to reuse textile effluents and implement ZLD (zero liquid discharge) option in the upcoming years. So, the understanding from this study will help factory owners and policymakers to plan and set a target to implement water recycle and reuse options to reduce water consumption and water footprint for textile wet processing.

1.3 Research Objectives

The aim of this research work was to study ultrafiltration and reverse osmosis processes for advanced treatment of textile effluents and recycle the effluent water in the industries as process water. The objectives of the present work can be summarized as follows:

- 1. Experimental analysis of UF and RO as tertiary treatment units for textile effluents from dyeing and washing factories (knit, woven, denim)
- 2. Analyzing hydraulic performance, capital expenditures and operational costs of UF and RO membrane technology for advanced treatment and recycling of textile effluents.

The possible outcome of this research is as follows:

1. Data will be generated to guide local textile factories and national regulatory bodies using recent industry-based data to understand the technical and economic issues associated to tertiary (UF and RO) membrane treatment and reusing textile effluents.

1.4 Thesis Overview

The thesis consists of five chapters. Chapter 1 contains an introduction to the research topic. Chapter 2 contains a detailed literature review that provides further insights to the research work. It gives basic information about the textile industries of Bangladesh and how they manage the textile effluents. It also gives information about the groundwater condition in Bangladesh and how advanced water treatment processes like Ultrafiltration and Reverse Osmosis can help to reduce the load on groundwater demand. It further shows that this huge load on groundwater can be decreased by further treating the textile effluents after being treated in the effluent treatment plants. In Chapter 3, materials and methods are discussed along with the functions of the used chemicals and reagents. In Chapter 4, obtained results were shown, analyzed and then further discussed. Chapter 5 which is the final chapter concludes and summarizes the findings of the study and gives recommendations for future work.

Chapter 2: Literature Review

2.1 Textile industries of Bangladesh

For a variety of purposes, textiles have long been an important part of Bangladesh's economy. Ready-to-wear apparel accounts for 80% of the country's \$24 billion in annual exports and 15% of GDP. Bangladesh's garment exports could double in the next ten years, according to consulting firm McKinsey and Company. Bangladesh is one of the largest exporters of textile products in Asia, employing a significant percentage of the country's workforce. Currently, the textile industry employs 45 percent of all factory workers in the country and generates 5% of total national income [9]. Not only that, Bangladesh has set a target of \$50 billion by 2030 and has already established itself as the world's second largest textile producer. [10].

Bangladesh currently has more than 5,000 garment factories, according to a private estimate. However, according to the Bangladesh Garment Manufacturers and Exporters Association (BGMEA), Bangladesh has over 4000 garment factories. [11]. Table 2.1 summarizes the latest comparative statement on RMG export and total export of Bangladesh (from 2000-01 to 2019-20) [12]. The table shows that RMG exports have steadily risen over the last 20 years. However, due to the recent pandemic triggered by COVID 19, exports were forced to decline in 2019-20. Nonetheless, the proportion of RMG exports to total export did not differ significantly. Since the factories have reopened and are fully operational, it is expected that RMG exports will resume soon.

Local factories are thought to export 95% of their goods to other countries, with the remaining 5% consumed in the local market. Production volume varies depending on product price and weight, which can be determined from export data and a prediction of a production pattern produced. Shirts, T-shirts, Hoodies and sweatshirts, Sweaters, Denim, Trousers, Blouses, and other RMG items are manufactured in Bangladesh. A study showed real and expected textile production from 2011 to 2021, based on the high volume of shirt, T-shirt, trouser, and sweater production in Bangladeshi RMG industries. In one study, export data from Bangladesh's textile industries was analyzed from 2006 to 2016, and a 10% annual growth rate was used to forecast textile production from 2017 to 2021 [13]. This is represented in figure 2.1. It appears that since the 1980s, woven items have been making up the majority of textile production. It also predicts that production in 2021 will exceed 2.9 million tons of fabric, which is 1.61 times more than what was produced in 2016.

Figure 2.1: Annual textile production in Bangladesh (2011–2021) [13]

2.2 Textile subsectors in Bangladesh

Bangladesh textile sector comprises of the following sub-sectors: spinning mills, weaving and knitting factories and dyeing and finishing units. Despite major constraints such as global recession, strong foreign competition, inadequate infrastructure, and insufficient power supplies, all sub-sectors of the textile industry are rising [14].

2.2.1 Spinning subsector

Bangladesh has a more than four-decade-old spinning industry. Bangladesh's spinning mills have made a major contribution to the country's readymade garment industry. With 12.50 million spindles, Bangladesh's spinning industry can meet 95% of the garment industry's yarn demand. Imported fabrics and yarns are used in the majority of sweaters, and home textiles, and the spinning industry can supply 40% of their yarn needs. Bangladesh's knit and denim garment industry has already established a large backward linkage industry — spinning, knitting, weaving, dyeing, and finishing — which is primarily made of cotton and cotton blends of various forms. Cotton accounts

for 60–70% of a spinning mill's total expense. Bangladesh imported 7.20 million bales of cotton in 2018, making it the world's largest cotton importer. Spinning is a power-hungry industry that relies on captive gas generators for the majority of its production. It is important for industry to have long-term access to reliable power at a fair cost [15].

2.2.2 Weaving subsector

At present, Bangladesh has 400 modern textile weaving factories with an annual production capacity of 1,600 million meters of woven fabrics for the export-oriented readymade garment industries. However, this amount of woven fabric production only meets 40% of the demand of the country's rapidly expanding readymade garment industries. Around 80,000 people are employed in these new textile factories. Apart from these weaving mills, Bangladesh has 1,065 advanced textile and power-loom mills that produce about 400 million meters of woven fabrics for export and domestic use. This sub-sector of the country's textile industry employs approximately 45,000 employees. The majority of Bangladesh's woven apparel exports contain 50-70% imported material, despite the textile sector's continued expansion over the last decade, although not at the same rate as the apparel sector [14].

2.2.3 Knitting subsector

Bangladesh is home to one of the world's largest knit garment manufacturing industries. For major fast fashion brands, the sector produces high-quality knit garments. In terms of both capabilities and competitiveness, Bangladesh is one of the world's most powerful knit garment manufacturers. The country is well-known for producing high-quality goods at low prices. Bangladesh has become a heaven for the manufacture of knit garments for foreign brands. They always get the best deal here, having the best quality for the least amount of money.

The overall export in this sector was \$13.76 billion in 2016-17, accounting for around 6.5 percent of the global market share. Bangladesh has a much larger market share in terms of sales. Bangladesh accounted for 12% of the global t-shirt industry in 2016.

The country has one of the world's largest knit garment manufacturing capacities. Knit factories in Bangladesh have expanded their capacity at a rapid rate over the last decade, and they are now very large volume producers of knit garments [16]. However, the COVID-19 pandemic has disrupted global fashion apparel demand, causing apparel prices to plummet and markets to contract. The apparel manufacturing countries, especially Bangladesh, have a significant negative effect. According to data from the Export Promotion Bureau (EPB), earnings from Bangladesh Knitwear Garments exports were USD 10.89 billion in July-May of FY 2019-20, a 5.17 percent decrease [17].

2.2.4 Handlooms subsector

The handloom industry is the largest handicraft industry in our country, and after agriculture, it is the second largest source of rural jobs. Since the beginning of time, the traditional handloom industry has been Bangladesh's largest nonfarm economic activity, providing enormous employment opportunities for the rural poor, especially women. Around 1.5 million weavers, dyers, hand spinners, embroiderers, and other allied artisans have been putting their artistic talents to work on more than 0.30 million working looms per year, producing around 620 million meters of fabric. It contributes 63 percent of the country's total fabric output for domestic use, meeting 40 percent of the country's fabric demand. Furthermore, it employs a million rural residents, half of whom are women. Indirectly, another half-million workers work in the industry. As a value addition, it adds more than 10 (ten) billion taka to the national exchequer per year. While many countries have seen traditional industries disappear due to the introduction of modern production methods and technology, Bangladesh's handloom industry has not only survived, but has recently shown a positive growth trend in terms of total jobs and output. According to available data, the handloom industry has also been active in exporting to India a high-fashion variety of women's wear known as Jamdani saree. The traditional handloom industry has long been the most important industry in Bangladesh's rural areas, and handloom products such as muslin were well-known in Asia and Europe. [18].

2.2.5 Dyeing, printing and finishing subsector

All fabric processes that aren't used in fiber production, yarn production, or fabric forming are referred to as textile finishing. Finishing effectively entails enhancing or beautifying the products.

Bangladesh's textile dyeing industry contributes significantly to the country's economic and environmental growth. The textile dyeing industry has been labeled as one of the most polluting industries in the world. In Bangladesh, there are numerous dyeing industries, the majority of which are concentrated in the Gazipur and Narayanganj industrial areas [19]. The main goal of dyeing is to create uniform shades on different textiles. However, with the careful application of technological or mechanical improvements, as well as changes in fiber form or structure, we can be able to create an infinite number of fancy dyeing results. Simple spot marks to progressive lighter or darker colors, woven dyeing, tie and dye, cross, and ring effects, surface dyeing, space dyeing, ombré dyeing, and other effects are all possible. A similar emphasis has been put on finishing, with the sole goals being not only routine physical or functional changes, but also a range of creative effects such as calendaring, brushing, and differential washing effects, as well as specialty chemicals that enhance the look of clothing and appeal to consumers.

Reactive, scattered, indanthrene, and other dyes are used to color textiles. Chemicals of different kinds are used in these dyes. Because of compound chemical reactions, the amounts of chemical compounds in the effluent from this process often differ, and the effluent is colored. High levels of color impurities are produced during the printing process. This is the final step in the wet processing phase [19].

It's worth noting that designs created by various processes (such as weaving, printing, dyeing, and finishing) have a few fundamental differences; artistic requirements and appearance are largely incomparable. Dyeing and finishing designs can produce a wide range of results, and the techniques used to create them are numerous [20].

2.3 Impact of textile wastewater on the environment and health

Bangladesh's textile industries, especially the dyeing industries, produce a large amount of wastewater each year. Figure 2.1 and 2.2 show actual and projected textile production, as well as the wastewater volume produced by textile dyeing, from 2011 to 2021.

Figure 2.2: Yearly wastewater volume produced by textile industries using conventional dyeing technology [13]

Figure 2.1 indicates that total fabric production in 2021 will be about 2.9 million metric tons, which is around 1.61 times than what it was in 2016. A rise in output of this magnitude would result in 349 million $m³$ of wastewater (2021). The effluent volume and pollution load produced by textile dyeing industries in 2021 are expected to be 1.61 times higher than in 2016. [13]. Because of its effect on environmental health and safety, water contamination caused by industrial effluent discharges has become a worrying phenomenon.

2.3.1 Environmental Impacts

In terms of adhering to the color specifications, the environmental issues associated with residual color in treated textile effluents are always a concern for each textile operator that directly discharges, both sewage treatment works and commercial textile operations. [21].

Textile wastewaters from various stages of textile production contain large quantities of contaminants that, if released without treatment, are extremely harmful to the environment. The amount of contamination caused by dye bath water is extremely high. The contamination of the environment caused by the release of a variety of azo dyes by industrial wastewater is a serious problem today. The textile industry includes a large number of mechanical and chemical processes, each of which has a different environmental effect. The effluent is extremely poisonous due to the presence of sulfur, naphthol, vat dyes, nitrates, acetic acid, soaps, chromium compounds, heavy metals such as copper, arsenic, lead, cadmium, mercury, nickel, and cobalt, and auxiliary chemicals. The mill effluent usually has a high temperature and pH, which are both extremely harmful. Additionally, the accumulation of color reduces sunlight penetration, disrupting the receiving water's ecosystem.

Furthermore, enabling this effluent to circulate in the fields clogs the soil pores, resulting in a loss of soil productivity. The texture of the soil becomes tougher, preventing root penetration. The sewerage pipes are corroded and incrusted by the wastewater that flows down the drains. When wastewater is permitted to flow freely in drains and rivers, it contaminates drinking water from hand pumps, rendering it unfit for human use. Watercourse color is viewed as an aesthetic concern rather than an eco-toxic threat. As a result, the public tends to tolerate rivers that are blue, green, or brown in color, but "abnormal" colors like red and purple generally cause the most concern. Wastewater also induces drain leakage, which increases the cost of drain cleaning. Other environmental concerns include air pollution, especially volatile organic compounds (VOCs), excessive noise or odor, and workplace safety [22].

2.3.2 Health impacts

The textile industry consumes roughly a quarter of all chemical production worldwide. Textile processing uses up to 2000 different chemicals, many of which are considered to be hazardous to

human (and animal) health [23]. As a result, releasing textile wastewater directly into water bodies such as rivers pollutes the water and damages the flora and fauna [24]. Dyes may have acute and/or chronic effects on exposed species depending on exposure period and dye concentration. The most significant consequence of textile waste is the loss of dissolved oxygen in water, which is important for marine life. This also makes water's self-purification process more difficult.

Under anaerobic conditions, dyes used in the textile industry may be converted to toxic and/or carcinogenic materials, posing a health risk. Many dyes are harmful to fish and mammals; they hinder microbial growth and have an effect on flora and fauna. Aside from that, a number of dyes and their decomposition derivatives have been found to be harmful to aquatic life (plants, microorganisms, fish, and mammals). They're also carcinogenic, which means they can cause intestinal cancer and cerebral defects in a developing fetus. Textile dyes can cause allergies including contact dermatitis and respiratory diseases, as well as allergic reactions in the eyes, skin irritation, and mucous membrane and upper respiratory tract irritation [22].

Many of these dangerous chemicals may be absorbed through the skin, lungs, or digestive system. Carcinogenic and mutagenic chemicals are found in some of these compounds. The mutagenicity of azo dyes, as well as their ability to cause cancer, have been investigated. It was discovered that painters who had been exposed to azo dyes for a long time developed bladder cancer [25].

In the 1950s and 1960s, allergic contact dermatitis caused by formaldehyde in textiles was widely published in the literature. In recent years, the textile industry has attempted to reduce the amount of free formaldehyde in clothing and bedding fabrics, and reports of formaldehyde-induced contact allergy to textiles have decreased [26].

Aside from the health risks associated with textile wastewater and textile materials, there are many hazards in the textile industry's workplace. Powerful acids, such as nitric and sulphuric acid, or a combination of the two known as mixed acid, are the first danger we encounter in the dye industry. These are used to nitrate benzene, toluene, and other chemicals. Because of their toxic nature to human tissues, every safeguard is taken to keep these acids from getting on the skin of the workers. Workers in the textiles industry face a variety of dangers and threats, ranging from noise and hazardous chemicals to manual handling and operating with unsafe machinery. Workers are exposed to hazards at any point of the processing process, from material production to

manufacturing, finishing, coloring, and packaging, and some of these are especially hazardous to women's health [19].

Fabric manufacturing, as well as the products and chemicals used in it, wreak havoc on the environment and human health. Analyzing chemicals and other substances in fabrics is critical, and further research into this area is needed [25].

2.4 Management of Textile Chemicals and Wastewater

2.4.1 Textile chemicals management

Chemical management regulations provided by buyers are crucial to the textile industry. There is no existing government policy on the safety and security of textile chemicals. Chemical management regulations provided by multinational apparel brands, also known as buyers, are extremely important to local textile industries. There are strict regulations focused on numerous private and global chemical management systems, laws, treaties, and mechanisms that primarily address chemical safety and occupational health and safety, but seldom address chemical security [27]. But there exists strict government policy in Bangladesh to impactfully manage textile wastewater.

2.4.2 Textile wastewater management

No industrial unit or project can be established or undertaken without first obtaining an Environmental Clearance Certificate (ECC) from the DOE, according to Section 12 of the Bangladesh Environment Conservation Act 1995 (ECA 1995). An applicant for ECC in the Orange B and Red categories cannot do so without first building an Effluent Treatment Plant (ETP) [28]. Textile industries fall under the Red category due to their huge pollution load.

An Environmental Management Plan (EMP) must be submitted as part of the ECC application for Orange-B and Red group industries. The EMP should provide a Process Flow Diagram, a Layout Plan that includes the position of the Effluent Disposal System, and the Effluent Treatment Plant's plan and design, as well as comprehensive details about its effectiveness. A post-project tracking

software should be included in the EMP [28]. So, without establishing an ETP, no textile industrial unit can be established or undertaken.

Bangladeshi laws aim to ensure that wastewater is discharged without damaging the ecosystem. Until discharging into the atmosphere, the Environmental Conservation Rules of 1997 include specific guidance and require companies to uphold those water quality parameter values. Table 2.2 shows standards for waste from industrial units or projects standards waste for inland surface water, which is taken from Schedule 10 of the ECR 1997. (Selected parameter values are shown). Standards for sector wise industrial effluent discharge are also included in ECR 1997. The discharge standards for composite textile plants and large processing units are shown in Table 2.3, which is taken from Schedule 12 of the ECR 1997 [29].

Table 2.2: ECR 1997 Standards for waste from industrial units or projects waste for inland surface water [29]

Parameter	Inland surface quality standards (mg/l)
Temperature $(^{\circ}C)$	40
Biological Oxygen Demand (BOD ₅) at 20°C	50
Chemical Oxygen Demand (COD)	200
Dissolved Oxygen (DO)	$4.5 - 8$
Total Dissolved Solids (TDS)	2,100
pH	$6-9$
Suspended Solid (SS)	150
Nitrate	10
Arsenic	0.2
Lead	0.1
Chloride	600
Iron	$\overline{2}$
Manganese	5
Oil & Grease	10

Table 2.3: ECR 1997 discharge standards for composite textile plant and large processing unit effluent [29]

2.4.3 Zero liquid discharge (ZLD) management system

Zero liquid discharge (ZLD) is a wastewater management strategy that ensures that no industrial wastewater is discharged into the environment. It is accomplished by recycling wastewater and then recovering and reusing it for industrial purposes. As a consequence, ZLD is a closed loop circuit with no discharge. Despite the fact that ZLD is an expensive operation, it provides economic benefits by recovering salts and other chemical compounds [30]. It is a completely new concept for Bangladesh textile sector. So, it should involve itself with the ongoing research on achieving zero liquid discharge to further know the strength and opportunities associated with this system.

The textile effluent is immune to biodegradation due to two major contaminants: color and total dissolved solids. Though physico-chemical methods can effectively remove color, they produce a large amount of sludge as a by-product. Biological treatment approaches have no effect on the salt. Apart from its non-biodegradability, the dyeing effluent is strongly colored and imparts its color to the receiving water bodies into which it is discharged. The regulatory authorities, having

recognized the severity of the water contamination, are insisting on treating the wastewater in order to reuse it in the process and reach "zero discharge" [5]. In order to protect the environment in the future, the Bangladesh government should investigate this advanced management scheme.

2.5 Groundwater condition and its' consumption in Bangladesh

Groundwater is a vital resource for billions of people's livelihoods and food security, especially in Asia's booming agricultural economies. Groundwater supplies nearly half of the existing potable water supplies, 40 percent of agricultural water demand, and 20 percent of irrigation water globally [31].

Groundwater is the lifeblood of agriculture in India, Northern Sri Lanka, Pakistan's Punjab, Bangladesh, and the Northern China Plain due to scarcity and inconsistency of surface water sources. Groundwater provides about 60% of total agricultural water use in India, accounting for more than half of the total irrigated area [32]. Groundwater extraction accounts for 65 percent, 70 percent, 50 percent, and 50 percent of total agricultural water supply in the North China plains, respectively, for the provinces of Beijing, Hebei, Nanan, and Shandog. Tube well irrigation has aided India and China in achieving food security for their large populations and improving the rural poor's livelihoods. Groundwater supplies more than half of the total crop water requirements in Pakistan's Punjab province, which produces 90% of the country's grain [33].

2.5.1 Groundwater consumption by irrigation

Bangladesh has a total length of 22,000 kilometers of rivers. The annual renewable water supplies within the country are projected to be $105,000$ Mm³. This includes approximately 21,000 Mm³ of groundwater supplies and 84,000 Mm³ of surface water provided internally as stream flows from rainfall [34]. Groundwater is derived in part from the infiltration of surface water with an external source. Annual cross-border river flows and entering groundwater are estimated to be 1,121,600 Mm^3 , taking the total sustainable water resources to 1,226,600 Mm^3 per year.

In Bangladesh's agricultural economy, rice production is the single most important activity. Farmers in northern Bangladesh have been extracting groundwater for irrigation, commercial, and domestic purposes due to a lack of surface water and easy access to groundwater through shallow tube wells. Farmers were encouraged to grow irrigated boro rice during the dry winter season due to increased water availability. Currently, groundwater (both shallow and deep tubewells) irrigates around 4.2 million ha of land, whereas surface water (low lift pumps) irrigates just 1.03 million ha (BADC 2013).

Figure 2.3: Area irrigated with (a) groundwater and (b) surface water in Bangladesh [35]

The area irrigated by surface water declined from 76% in 1981 to 23% in 2012, whereas for the same period, area irrigated by groundwater has jumped to 80% from 16% as shown in figure 2.3.

2.5.2 Groundwater consumption by the textile industries

The majority of industrial water comes from groundwater (estimated at 98 percent), and factories must have a groundwater abstraction license or pay the industrial or commercial rate for municipally supplied water. Around 90% of the water used in cities comes from underground sources.

However, due to a considerable amount of unrecorded industrial water abstraction, either from unregistered abstraction, unmetered abstraction, or a lack of control and compliance, the figure above is an underestimation of actual water usage.

The demand for textile water can only be measured based on a few proxy measures because there are no official estimates on how much groundwater the textile industry absorbs annually. According to a report, the textile and RMG industries used about 1.5 billion m^3 (BCM) of water per year in 2009. Over-extraction of groundwater has resulted from a heavy reliance on groundwater as the primary water source for all sectoral water usage, as evidenced by a rapid decline in groundwater table, especially in areas with textile clusters [36].

Bangladesh's textile industry uses about 1,500 billion liters of groundwater per year for washing and dyeing fabrics, according to a 2014 study published by partners for Water Programme of the Netherlands in collaboration with the government. Since the majority of garment factories are located in Dhaka, Gazipur, Savar, and Narayanganj, groundwater use in these areas is comparatively higher, despite the fact that high groundwater usage is prevalent in all hubs with RMG and textile manufacturing units. [37].

2.5.3 Groundwater level depletion in Bangladesh

According to the zoning map of the Bangladesh Agricultural Development Corporation (2004 and 2010), groundwater levels are steadily decreasing, which has become a major source of concern for everyone [37]. Over the decade 2001-2010, there was a major drawdown of aquifers due to over-exploitation of groundwater. It has been calculated using data from the Bangladesh Water Development Board (BWDB) that decline has increased significantly over time in areas with water tables less than 8 meters deep. Between 1998 and 2002, this region accounted for just 4% of the country's total, but it grew to 11% in 2008 and 14% in 2012. The north-west (e.g., Braind Tract) and north-central (e.g., Madhupur Tract) regions are the most severely affected. These are areas where extensive boro cultivation is taking place, with long-term groundwater patterns showing a downward trend. Water tables are slowly decreasing in the northwest, but at a slower rate (0.1-0.5 m per year), rendering the use of STWs tapping shallow aquifers for extensive boro irrigation unsustainable.

The groundwater table in Dhaka has fallen by about 10 meters as a result of excessive usage for domestic and industrial purposes between 2000 and 2010, which is much higher than in other parts of Bangladesh. If this trend continues, serious water risks will exist not only for industry, but also for other sectors of water use, especially domestic water use [36].

2.6 Membrane-based technologies for treating wastewater

Filtration is the method of separating colloids and particulates from liquid. The range of particle sizes is extended in membrane filtration to include dissolved compounds (typically from 0.0001 micron to 1 micron). The membrane's function is to act as a selective barrier, allowing some constituents to move through while retaining others found in the liquid.

Microfiltration, Ultrafiltration, Nanofiltration, and Reverse Osmosis are all membrane processes [38]. The disparity between traditional and membrane filtration processes is typically shown in Figure 2.4. When comparing the two, there are two main differences to remember. The first is that, unlike traditional coagulation-based processes, MF and UF processes achieve particle separation by physical removal, effectively size exclusion, and do not need physiochemical treatment prior to media filtration to achieve the required degree of particle removal. The second distinction of membrane filtration is that the pore size is highly uniform, allowing for exceptionally high, or "absolute," particle or microorganism elimination. [39].

Figure 2.4: Basic diagram of mass transport in membranes [39]

2.6.1 Microfiltration

Microfiltration (MF) is a pressure-driven separation process for concentrating, purifying, or separating macromolecules, colloids, and suspended particles from a solution. The nominal pore sizes of MF membranes are usually on the order of 0.1–1.0 m. In the food industry, MF processing is commonly used for wine, juice, and beer clarification, wastewater treatment, and plasma separation from blood for therapeutic and industrial purposes. Cell recycling and harvesting, isolation of recombinant proteins from cell debris, and purification of process streams are all examples of MF applications in the biotechnology industry.

In a cross-flow configuration, MF is usually run at low TMPs (4 bar or 0.4 MPa). To avoid cake formation and therefore membrane fouling, the feed stream flows tangentially to the membrane surface. Membrane fouling caused by suspended solids in the feed stream often limits the activity of cross-flow MF. As the stored particles settle on and inside the membrane, the permeate flux decreases over time. External fouling (cake formation) of cells, cell debris, or other rejected particles on the membrane surface is normally reversible, while internal fouling (deposition and adsorption of small particles or macromolecules inside the internal pore structure of the membrane) is often irreversible. [40].

2.6.2 Ultrafiltration

The UF membrane is similar to the MF membrane, but it allows smaller molecules to pass through. Macromolecules, colloids, and solutes with molecular weights greater than 10,000 Da are isolated using UF membranes. As a driving force, TMPs ranging from 100 to 1000 kPa are used. The difference in solute size, membrane properties, and hydrodynamic conditions all contribute to the selectivity of the UF membrane. The phase inversion method is an easy way to prepare this membrane, which results in an asymmetric porous structure [41]. This method is effective for removing suspended matter through direct filtration or biological treatment in place of a sedimentation device. It is highly dependent on the type of material that makes up the membrane. According to one analysis, this process may be useful as a reverse osmosis pre-treatment tool. It could be used to get rid of the higher molecular weight components of leachate that clog reverse osmosis membranes. The use of ultrafiltration alone resulted in a COD removal of 50%. Another

study showed that using a combination of bioreactors and membrane technology, ultrafiltration membranes have been successfully used in full-scale membrane bioreactor plants. Using this process, high levels of landfill leachate treatment have been achieved [42].

2.6.3 Nanofiltration

A nanofiltration (NF) membrane is a pressure-driven membrane process that is intermediate in complexity to reverse osmosis (RO) and ultrafiltration (UF). It has a pore size of 0.2–2 nm and a molecular weight cut-off of 200–1000 Da. Normally, the membrane surface is negatively charged to increase the efficiency of the membrane in separating dissolved ions. Currently, NF membrane applications include desalination as a pretreatment step for feed, the removal of heavy metals and pesticides from groundwater, the separation of organic compounds in wastewater treatment, and water recycling in a number of industrial processes [43]. Nanofiltration membranes, which lie between pure RO and pure UF membranes, are often referred to as loose RO, low-pressure RO, or, more generally, NF membranes. NF membranes usually reject between 20% and 80% of sodium chloride and have MWCOs of 200–1000 Da for dissolved organic solutes. These characteristics fall between those of RO membranes with a salt rejection of greater than 90% and a MWCO of less than 50 and those of UF membranes with a salt rejection of less than 5%. Since the neutral NF membrane rejects salts in proportion to their molecular size, the order of rejection is straightforward that is $CaCl₂ > Na₂SO₄ > NaCl [44].$

2.6.4 Reverse Osmosis

The RO membrane market is largely based on thin-film composite (TFC) polyamide membranes, which usually consist of a polyester web used as a support, a microporous interlayer, and a top ultrathin barrier layer with selectivity properties and low resistance to mass transfer of permeate. The selective layer is usually made of 1,3-phenylenediamine (also known as 1,3-benzenediamine) and tri-acid chloride of benzene, which can provide salt rejection of greater than 99.5 percent to NaCl. Historically, cellulose acetate (CA) membranes were used in RO technology. Despite their higher chlorine resistance (which is often used as a disinfectant to prevent bacterial growth on membranes), asymmetric CA or triacetate hollow-fibre modules are much less commonly used in

RO plants due to their lower rejection capacity. The boron rejection of any commercially available membrane is still inadequate (93 percent) to meet WHO water drinking requirements by one-pass RO method, which is one of the unresolved challenges. [45].

2.7 Recycling textile effluent by Ultrafiltration and Reverse Osmosis

Ultrafiltration facilitates water clarification and disinfection in a single phase with consistent permeate content and no by-products. Reverse osmosis is a membrane method that has a long history of industrial applications for eliminating salts from solutions and creating nearly deionized water [46]. Both processes are being used as advanced water treatment processes for a long time. After treating wastewater by these processes, the permeate water can be reused in the industries such as in textile industries [46] [7], metal finishing industries [6], dairy industries [47], tanneries [48], semiconductor plants [49], refineries, [50] etc. In this way, yearly requirement of fresh water for these industries can be reduced to a great extent. Although the production of this reusable water by advanced treatment UF and RO is lot more costly than the conventional source of freshwater (groundwater), it will become a must to conserve our groundwater and other freshwater resources within a few decades.

The research was carried out in one study by treating textile wastewaters from a pilot plant and replicating a separation method based on ultrafiltration and reverse osmosis on a smaller scale. The final separation step, reverse osmosis at 8 bar pressure, provided a permeate of significantly higher quality than the current process water. As a result, the generated permeate can be reused in all production phases, including the most stringent water quality requirements [46].

Another paper looks at a case study of a full-scale effluent treatment plant upgrade in one of the textile units. Based on treatability studies, the existing treatment facility was upgraded by introducing membrane separation processes that included ultrafiltration and reverse osmosis units to produce recyclable boiler feed water quality. At full scale, the advanced treatment facility achieved reusable effluent characteristics with SS , $BOD₅$, and COD concentrations below detectable limits and TDS concentrations of 40 mg/l (average) [7].

Another work concerns the treatment of textile plant effluent after conventional biological processing. The effluent from the textile plant was treated with a biological treatment process, but the consistency was not good enough to reuse on site. The RO and NF membrane processes were successfully used to obtain a permeate quality that enabled water reuse in the dyeing process, but this process required physicochemical pretreatment [51].

In another study, textile wastewater was treated for water reuse. To improve the quality and recovery rate of reused water, a two-stage combined water reuse treatment was developed. A flocculation and sedimentation system, two sand filtration systems, an ozonation unit, an ultrafiltration (UF) system, and a reverse osmosis (RO) system were all part of the primary treatment. An ozonation unit, a sand filtration unit, and UF and RO systems were used in the second treatment. For chemical oxygen consumption (COD), color, NH3-N, hardness, Chloride, SO_4^2 , turbidity, Fe^{3+} , and Cu^{2+} parameters, the treated, reused water met the reuse requirement and satisfied the drinking water standard rates. [52].

The experimental results of a pilot-scale application of membrane technologies to textile wastewater advanced treatment, downstream of a biological activated sludge process, aimed at water reuse in textile technology processes, are described in another paper. The final permeate from the two-stage membrane system (UF+RO or MF+NF) has a high quality and can be used as process water in the textile industry, according to the studies. Around 60–65 percent of mill effluents can be recycled using this form of advanced treatment method [53].

All these studies indicate that membrane processes, especially ultrafiltration and reverse osmosis, have the potential to reuse textile effluents. For Bangladesh, this is still a novel idea. Consequently, there is plenty of possibility to learn about these advanced processes. The outcomes of the learning can be implemented in the textile industry to preserve groundwater.
Chapter 3: Materials and Methods

3.1 Instruments, Chemicals and Reagents

3.1.1 Instruments

A Pre-treatment filter was used to treat the effluent coming out of the ETP which contained five standard 40'' string wound filter cartridges. The pore size of the pre-treatment filter was 100 microns. CLRO20 filter made by Hydromaster which is a combination of ultrafiltration and reverse osmosis system along with the pre-treatment filter was used for this study. The technical details of the membrane systems are given in table 3.1. Figure 3.1 represents the real time setup of the mobile filtration unit.

Particulars	Ultrafiltration	Reverse osmosis
Model	U850	UP-LRO 8040
Material	Hydrophilic modified PAN,	Thin-film composite,
	PVDF or PTFE (Teflon)	Polyamide
Module	Hollow fiber	Spiral wound, FRP wrapping
Pore size	0.02 micron	0.001 micron
Surface area	35 m^2 /membrane	37.1 m^2
Number	$\overline{2}$	

Table 3.1: Technical details of ultrafiltration and reverse osmosis units

Other instruments used in this work and their functions are tabulated in table 3.2.

Table 3.2: Instruments/Materials used in this work and their functions

Figure 3.1: Setup of the mobile filtration unit

3.1.2 Reagents

Reagents used in this work and their functions are listed below in table 3.3.

3.2 Experimental procedures

3.2.1 Selection of textile industries and numbering

Cluster of textile industries are located at Gazipur and Narayanganj of Dhaka city. Among these, several ones were selected to bring different types of industries in the study. The selected ones include two knit dyeing, two woven dyeing and two denim washing factories. These factories are numbered in this work as follows:

Knit dyeing factory 1 as KD#1, Knit dyeing factory 2 as KD#2, Woven dyeing factory 1 as WD#1, Woven dyeing factory 2 as WD#2, denim washing factory 1 as DW#1 and denim washing factory 2 as DW#2.

3.2.2 Characterization of Wastewater and ETP treated water

Untreated wastewater and ETP treated water samples were collected from the inlet and outlet respectively of the effluent treatment plant of the selected textile industries located at Dhaka. The main parameters that were used to characterize the wastewater were pH, total dissolved solids (TDS), total suspended solids (TSS), colour, biological oxygen demand (BOD5) and chemical oxygen demand (COD). pH was measured using a pH meter from HANNA. TDS was measured using TDS meter from HACH. TSS, colour and COD were measured using DR 6000 spectrophotometer from HACH. COD was measured using standard method 5220 D. BOD₅ was measured using standard method 5210 B.

3.2.3 RO Permeate and Reject Water analysis

For each industry, a part of the effluent from the ETP was stored in a tank and then taken into the filtration system using a submersible pump. The pump flows the water through the pre-treatment filter. Water from this filter was then passed to the ultrafiltration membranes. Water flowed through the membranes in parallel and the combined permeate was passed to the reverse osmosis unit. The maximum pressure of the feed in the reverse osmosis unit was 145 psi (10 bar). Figure 3.2 represents the schematics of the advanced treatment process of the ETP treated water.

Figure 3.2: Schematics of the applied advanced treatment of the ETP treated water

The reverse osmosis unit of the mobile filtration unit can be configured to change the flowrate of reject (i.e. saline water) or permeate (i.e. treated water) stream. In this study, flowrate of the reject stream was varied. Starting with permeate flowrate to reject flowrate ratio of 80:20 in the reverse osmosis unit, samples of permeate and reject were collected when the system reached a steady state. No changes were made in the ultrafiltration system. Similarly, samples were collected after reaching a steady-state when permeate flowrate to reject flowrate ratio of 60:40 and 50:50 were maintained at start-up. Reject sample from the ultrafiltration unit was also collected.

To further comprehend the performance of the advanced filtration system, a denim washing factory was selected for case study. For this study, permeate flowrate to reject flowrate ratio of 60:40 was maintained at start-up and the filtration unit was continuously run for 2 hours. After that samples of permeate and reject from the reverse osmosis unit and reject from the ultrafiltration unit were collected. TDS, TSS, Colour, BOD₅, COD and pH of the samples were measured in the laboratory.

Chapter 4: Results and Discussions

4.1 Characterization of wastewater and ETP treated water

In addition to extensive quantities of water and dyes, wet textile manufacturing requires a variety of inorganic and organic chemicals, detergents, soaps and finishing chemicals to assist in the dyeing process to impart the desired properties to textile items that are dyed. From these methods, residual chemicals mostly remain in the effluent. In addition, during the desizing, scouring and bleaching operations, natural impurities such as waxes, proteins and pigments and other impurities used in processing, such as spinning oils, sizing chemicals and oil stains found in cotton textiles, are excluded. This results in low quality effluent, which is rich in BOD₅ and COD amounts [28].

4.1.1 Characterization of wastewater

In this study, wastewater samples were collected from the ETP inlet section of the selected textile industries for characterization. They were stored in separate 1-liter sample bottles at 4°C. Water quality parameter pH, TDS, TSS, colour, BOD₅ and COD values of wastewater that were collected from selected six textile industries are presented in table 4.1.

It was observed that in most cases, water quality parameters pH, TDS, TSS, colour, BOD⁵ and COD values were way out of the DOE guidelines. So, the wastewater needed to be treated to bring the values within the acceptable range. The selected textile industries had effluent treatment plants to facilitate this.

It can be seen that pH of the wastewater samples was always greater than 7. This means they are mildly or strongly basic.

Out of the six industries, 4 industries (two denim washing factories, one woven dyeing factory and one knit dyeing factory) had TDS value within DOE standards. Other industries had TDS value greater than 2100 mg/l.

Except for one knit dyeing and one woven dyeing industry, all industries had TSS value within DOE standards (<150 mg/l).

While color is not included in the Environment Conservation Rules (1997), it is a problem in dye house management since it is so noticeable unlike other pollutants. And as such, reducing color is critical for a factory's public perception.

BOD⁵ and COD are very critical in evaluating the quality of any kind of water. It was observed that in all cases, these values were way outside the range of the DOE standards. So extensive treatment of the wastewater is required to conserve the environment.

4.1.2 Correlation between BOD⁵ and COD of wastewater of the textile industries

If a correlation can be established between BOD₅ and COD for a specific type of wastewater, from the BOD⁵ value of that water, COD value can be obtained and vice versa. Instead of using the BOD⁵ test which takes five days to get result, COD analysis can estimate the amount of organic matter in wastewater in just $(3-4)$ hours and can be used as an alternative. The ratio between BOD₅ and COD values can differ depending on the characteristics of the raw wastewater. This ratio has been widely used as a biodegradation capability predictor. It is named as Biodegradability Index (B.I.). It is commonly regarded as the cut-off point between biodegradable and non-biodegradable waste [54]. The B.I. values of the wastewater of the selected industries are presented in table 4.1. The B.I. values ranged from 0.29 to 0.44. It is important to know the biodegradability index of the

raw influent wastewater inside the treatment plant for choosing the appropriate wastewater treatment technology. If B.I. is greater than 0.6, then the wastewater should be treated biologically because it will be fairly biodegradable [54]. For the selected industries, B.I. was always below 0.6, so the corresponding wastewater of the factories were not readily biodegradable.

Figure 4.1: COD and BOD⁵ of wastewater of textile industries

The BOD₅ and COD values of the selected industries in Dhaka during the wastewater characterization are shown in the figure 4.1.

4.1.3 Characterization of ETP treated water

The effluent treatment plant or ETP is primarily designed to purify industrial wastewater. It aims to release cleaner water to the environment so that the harmful effects of the effluent are mitigated. The effluent must comply with the national quality requirements for effluent discharge. Pollutants

are eliminated as water passes through the ETP and the water quality is improved, enabling final discharge to the environment without substantial risk. All selected industries for this study had ETP installed in their factories. The ETP treated water had significantly lower values of TDS, TSS, colour, $BOD₅$ and COD. This makes the effluent safer to discharge into the environment. Water quality parameter pH, TDS, TSS, colour, BOD₅ and COD values of ETP treated water from selected six textile industries are presented in table 4.2.

	pH	TDS	TSS	Colour (Pt-	BOD ₅	COD
Factory number		(mg/l)	(mg/l)	Co)	(mg/l)	(mg/l)
KD#1	7.8	1700	12	464	32	80
KD#2	8.1	518	13	98	259	465
WD#1	7.5	2240	17	240	153	305
WD#2	8.4	1920	5	357	120	357
DW#1	7.4	766	5	88	39	91
DW#2	8	1120	6	177	25	85
DOE standard for inland surface water	$6-9$	< 2100	<150		$<$ 50	$<$ 200

Table 4.2: pH, TDS, TSS, colour, BOD⁵ and COD values of ETP treated water from selected textile industries

Figure 4.2: pH values of wastewater and ETP treated water of the selected textile industries

(i) pH: from the results, it was observed that wastewater from all selected industries except for denim washing industries had pH values outside the standard range (6-9). After exiting the ETP, pH values of the treated water for all industries came within the range. The pH values of the treated water varied from 7.4 to 8.4. An interesting observation was that in the case of denim washing industries, pH values increased while for other industries, corresponding pH values decreased. The variation of pH values of wastewater and ETP treated water from the selected textile industries are shown in figure 4.2.

Figure 4.3: TDS values of wastewater and ETP treated water of the selected textile industries

(ii) TDS: Total dissolved solids (TDS) is a measure of the dissolved combined content of all suspended molecular, ionized, or micro-granular (colloidal sol) inorganic and organic substances contained in a liquid. For our selected industries, TDS value of the ETP treated water came within the range of DOE standards (<2100 mg/l) except for woven dyeing 1 factory. After treatment in the ETP, TDS value varied from 518 mg/l to 2240 mg/l. For the denim washing and woven dyeing 2 factories, TDS values increased significantly. In other cases, TDS values either decreased or did not vary much after treatment. The variation of TDS values of wastewater and ETP treated water from the selected textile industries are shown in figure 4.3.

Figure 4.4: TSS values of wastewater and ETP treated water of the selected textile industries

(iii) TSS: Substantial amount of reduction of Total suspended solids were observed for all textile factories. After treatment in the ETP, TSS value varied from 5 mg/l to 17 mg/l. These values are well within the range of DOE standards (<150 mg/l). The variation of TSS values of wastewater and ETP treated water from the selected textile industries are shown in figure 4.4.

Figure 4.5: Colour values of wastewater and ETP treated water of the selected textile industries

(iv) Colour: Colour is an important parameter for characterizing water. It is a highly critical parameter in terms of public perception. Color had been significantly removed from the wastewater after being treated in the ETP for all textile industries. After treatment in the ETP, colour varied from 88 Pt-Co to 464 Pt-Co. The variation of colour values of wastewater and ETP treated water from the selected textile industries are shown in figure 4.5.

Figure 4.6: COD values of wastewater and ETP treated water of the selected textile industries

(v) COD: COD is the amount of oxygen equivalents consumed in the chemical oxidation of organic matter by strong oxidant (e.g., potassium dichromate). The treatment of wastewater by effluent treatment plant led to a huge reduction of COD in wastewater for all factories. While the COD of wastewater varied from 1208 mg/l to 11383 mg/l, COD of the ETP treated water varied from 80 to 465 mg/l. Even after treatment, COD of water from the selected woven dyeing factories and knit dyeing factory 2 did not meet the criterion for discharge in inland surface water set by the DOE (<200 mg/l). The variation of COD values of wastewater and ETP treated water from the selected textile industries are shown in figure 4.6.

Figure 4.7: BOD⁵ values of wastewater and ETP treated water of the selected textile industries

(vi) BOD5: BOD⁵ shows the quantity of oxygen absorbed by bacteria and other micro-organisms in a water sample over a span of 5 days at a temperature of 20°C to aerobically degrade the water contents. The treatment of wastewater by effluent treatment plant showed almost similar trend in removing BOD₅ like it was observed for COD removal. While the BOD₅ of wastewater varied from 376 mg/l to 4104 mg/l, BOD_5 of the ETP treated water varied from 25 to 259 mg/l. After treatment, BOD₅ of water from the selected washing factories and knit dyeing factory 1 met the criterion for discharge in inland surface water set by the DOE (<50 mg/l). But others did not meet the criterion. The variation of BOD₅ values of wastewater and ETP treated water from the selected textile industries are shown in figure 4.7.

4.2 Advanced treatment of ETP treated water by Ultrafiltration and Reverse osmosis

Reverse osmosis and ultrafiltration, commonly referred to as RO and UF, use membrane technology. Ultrafiltration membranes can remove dissolved compounds like colloids, carbohydrates, proteins, etc. that have high molecular weight. The UF membranes used for this study had hollow fine-fiber modules. Reverse osmosis is a technology that uses a semipermeable membrane to remove contaminants from water. It can remove dissolved solids, organics, pyrogens, submicron colloidal matter, color, nitrate and bacteria from water. The RO membrane used for this study had a spiral wound module.

The used equipment for the study was a mobile filtration unit which consisted of a pre-treatment filter, ultrafiltration system and reverse osmosis system. The details of the equipment can be found in table 3.1. The setup was used at factory premises to further treat ETP treated water. For this treatment, the selected industries were the same as previous. The advanced treatment was conducted for three different permeate to reject ratio in order to observe changes in the permeate and reject water quality.

4.2.1 Advanced treatment of ETP treated water of Knit dyeing industry

Knit dyeing is a technique of dyeing knitted fabrics. The dyeing of knitted fabrics occurs in the exhaust method or in batch-wise process. Two knit dyeing factories were selected for this study. Knit dyeing factory 1 and 2 had installed biological ETP and combination of physicochemical and biological ETP respectively in their factories. The ETP treated water of the selected knit dyeing factories were stored in a tank. This water was treated at three different permeate to reject flowrate ratios, 80:20, 60:40 and 50:50 in the RO unit. For each ratio, data was taken after the system became stable. Tables 4.3 and 4.4 represent amounts of contaminants removal for the knit dyeing 1 and knit dyeing 2 factory respectively for permeate to reject flowrate ratio of 80:20, 60:40 and 50:50 in the RO unit.

Table 4.3: Contaminants removal for knit dyeing industry 1 for permeate to reject flowrate ratio of 80:20, 60:40 and 50:50 in the RO unit

BDL: below detection limit

Figure 4.8: (a) Wastewater (b) ETP treated water (c) UF reject water (d) RO permeate and (e) RO reject water for the knit dyeing factory 1

High performance was observed in TDS removal for all situations. The removal efficiency was found to be 98.6%, 99% and 99.3% respectively for permeate to reject flowrate ratios of 80:20, 60:40 and 50:50. In all cases, the permeate did not contain any suspended solid. For all cases, colour was almost removed from the ETP treated water (feed). Removal efficiency varied from 99% to 99.6% suggesting nearly the same efficiency in removing colour from ETP treated water containing colour of 464 Pt-Co.

In all cases, a large amount of removal of BOD₅ and COD was obtained. The removal efficiency for BOD₅ varied from 72% to 75% for the three different permeate to reject flow rate ratio. In case of COD removal, the efficiency varied from 75% to 78% for the three different permeate to reject flow rate ratio. For the 80:20, 60:40 and 50:50 permeate to reject flow rate ratio, the pH of the

permeate was 6.5 in all cases. The variation of parameters along with the increase in initial permeate flowrate percentage from the RO unit is presented in figure 4.9.

Figure 4.9: Change in removal efficiency of TDS, TSS and colour with the change in initial permeate flowrate (%) in the RO unit for knit dyeing industry 1

Table 4.4: Contaminants removal for knit dyeing industry 2 for permeate to reject flowrate ratio of 80:20, 60:40 and 50:50 in the RO unit

Permeate	Parameter	Feed	Reject from	Permeate	Process	Reject	contaminant
reject to			ultrafiltration	from	water	from	removal $(\%)$
flowrate				reverse	(Ground	reverse	
ratio				osmosis	water)	osmosis	
	TDS (mg/l)	518	609	35.7	$<$ 180	1045	93.1
	TSS(mg/l)	13	24	$\mathbf{1}$	BDL	$\overline{2}$	92.3
	Colour (Pt-	98.1	197	$\mathbf{1}$	BDL	73	$\overline{99}$
80:20	Co)						
	BOD ₅ (mg/l)	259	83	6	$0.5 - 10$	6	97.9
	COD (mg/l)	465	163	9	$<$ 30	19	98.1
	pH	8.1	8	6.5	$6.5 - 7.5$	8	$\overline{}$
	$\overline{\text{TDS}}$ (mg/l)	518	609	8.4	< 180	968	98.4
	TSS (mg/l)	13	24	$\mathbf{1}$	BDL	$\overline{2}$	92.3
	Colour (Pt-	98.1	197	$\overline{2}$	BDL	64	98
60:40	Co)						
	BOD ₅ (mg/l)	259	83	5	$0.5 - 10$	26	97.9
	COD (mg/l)	465	163	11	$<$ 30	39	97.6
	pH	8.1	8	6.5	$6.5 - 7.5$	7.9	\equiv
50:50	TDS(mg/l)	518	609	7.1	$<$ 180	925	98.6
	TSS(mg/l)	13	24	$\mathbf{1}$	BDL	$\overline{2}$	92.3
	Colour $(Pt-$	98.1	197	$\overline{2}$	BDL	144	98
	Co)						
	BOD ₅ (mg/l)	259	83	$\overline{7}$	$0.5 - 10$	20	97.5
	COD (mg/l)	465	163	16	$<$ 30	46	96.5
	pH	8.1	8	6.5	$6.5 - 7.5$	7.6	\blacksquare

BDL: below detection limit

Figure 4.10: (a) Wastewater (b) ETP treated water (c) UF reject water (d) RO permeate and (e) RO reject water for the knit dyeing factory 2

As previously shown for knit dyeing factory 1, very good efficiency was also noted for knit dyeing factory 2 in the removal of TDS. The removal efficiency was found to be 93.1%, 98.4% and 98.6% for permeate to reject flowrate ratios of 80:20, 60:40 and 50:50 respectively. For all ratios, only 1 mg/l of suspended solid was found in the permeate which corresponded to 92.3% removal. For all cases, colour was almost completely removed from the ETP treated water (feed). The ETP treated water contained colour of 98.1 Pt-Co.

For all permeate to reject flowrate ratios, highly efficient removal of BOD₅ and COD was obtained. he removal efficiency for BOD₅ varied from 97.5% to 97.9% for the three different permeate to reject flow rate ratio. In case of COD removal, the efficiency varied from 96.5% to 98.1% for the three different permeate to reject flow rate ratio. For the 80:20, 60:40 and 50:50 permeate to reject flow rate ratio, the pH of the permeate was 6.5 in all cases. The variation of parameters along with the increase in initial permeate flowrate percentage from the RO unit is presented in figure 4.11.

4.2.2 Advanced treatment of ETP treated water of woven dyeing industry

Woven dyeing is a technique of dyeing woven fabrics. Two woven dyeing factories were selected for this study. These woven dyeing factories 1 and 2 had installed combination of physicochemical and biological ETP and biological ETP respectively in their factories. As previous, advanced treatment of the ETP treated water of these factories was done using the mobile filtration unit. Table 4.5 and 4.6 represent amounts of contaminants removal for the woven dyeing 1 and woven dyeing 2 factory respectively for permeate to reject flowrate ratio of 80:20, 60:40 and 50:50 in the RO unit.

BDL: below detection limit

Figure 4.12: (a) Wastewater (b) ETP treated water (c) UF reject water (d) RO permeate and (e) RO reject water for the woven dyeing factory 1

For the woven fabric dyeing factory 1, high performance was observed in TDS removal. The removal efficiency was found to be 98.3%, 97.5% and 97.6% respectively for permeate to reject flowrate ratios of 80:20, 60:40 and 50:50. For all ratios, only 1 mg/l of suspended solid was found in the permeate which corresponded to 94.1% removal. For all cases, colour was completely removed from the ETP treated water (feed). The ETP treated water contained colour of 240 Pt-Co.

In all cases, a large amount of removal of BOD₅ and COD was obtained. The removal efficiency for BOD₅ varied from 94.1% to 98% for the three different permeate to reject flow rate ratio. In case of COD removal, the efficiency varied from 90.9% to 97.6% for the three different permeate to reject flow rate ratio. For the 80:20, 60:40 and 50:50 permeate to reject flow rate ratio, the pH of the permeate was 6.5 in all cases. The variation of parameters along with the increase in initial permeate flowrate percentage from the RO unit is presented in figure 4.13.

Figure 4.13: Change in removal efficiency of TDS, TSS and colour with the change in initial permeate flowrate (%) in the RO unit for woven dyeing industry 1

Permeate	Parameter	Feed	Reject from	Permeate	Process	Reject	contaminant
to reject			ultrafiltration	from	water	from	removal
flowrate				reverse	(Ground	reverse	(%)
ratio				osmosis	water)	osmosis	
	TDS (mg/l)	1920	1073	28.8	< 180	3150	98.5
	TSS(mg/l)	5	$\overline{4}$	$\mathbf{1}$	BDL	$\overline{4}$	80
	Colour $(Pt-$	357	161	BDL	BDL	156	100
80:20	Co)						
	BOD ₅ (mg/l)	51	88	9	$0.5 - 10$	24	82.2
	COD (mg/l)	120	188	25	$<$ 30	55	79.2
	pH	8.4	7.5	6.5	$6.5 - 7.5$	7.9	$\mathbb{L}^{\mathbb{N}}$
	$\overline{\text{TDS}}$ (mg/l)	1920	1073	27	< 180	3820	98.6
	TSS (mg/l)	5	$\overline{4}$	$\mathbf{1}$	BDL	$\overline{4}$	80
	Colour $(Pt-$	357	161	$\mathbf{1}$	BDL	289	99.7
60:40	Co)						
	BOD ₅ (mg/l)	51	88	10	$0.5 - 10$	22	80.3
	COD (mg/l)	120	188	23	$<$ 30	102	80.6
	pH	8.4	7.5	6.5	$6.5 - 7.5$	7.6	$\mathbb{L}^{\mathbb{N}}$
50:50	TDS (mg/l)	1920	1073	28.3	< 180	3540	98.5
	TSS(mg/l)	5	$\overline{4}$	$\mathbf{1}$	BDL	$\overline{3}$	80
	Colour $(Pt-$	357	161	$\mathbf{1}$	BDL	331	99.7
	Co)						
	BOD ₅ (mg/l)	51	88	9	$0.5 - 10$	$\overline{7}$	82.2
	COD (mg/l)	120	188	28	$<$ 30	58	76.7
	pH	8.4	7.5	6.5	$6.5 - 7.5$	8	\blacksquare

Table 4.6: Contaminants removal for woven dyeing industry 2 for permeate to reject flowrate ratio of 80:20, 60:40 and 50:50 in the RO unit

BDL: below detection limit

As previously observed for woven dyeing factory 1, high performance was also observed in TDS removal for all cases for woven dyeing factory 2. The removal efficiency was found to be 98.5%, 98.6% and 98.5% respectively for permeate to reject flowrate ratios of 80:20, 60:40 and 50:50. In all cases, only 1 mg/l of suspended solid was found in the permeate which corresponded to 80% removal. 99.7% colour was eradicated from the ETP treated water (feed) for permeate to reject flowrate ratios of 60:40. For other ratios, colour was completely removed.

In all cases, a large amount of removal of $BOD₅$ and COD was obtained. But removal efficiency was lower compared to woven dyeing factory 1. The removal efficiency for BOD₅ varied from 80.3% to 82.2% for the three different permeate to reject flow rate ratio. In case of COD removal, the efficiency varied from 76.7% to 80.6% for the three different permeate to reject flow rate ratio. For the 80:20, 60:40 and 50:50 permeate to reject flow rate ratio, the pH of the permeate was 6.5 in all cases. The variation of parameters along with the increase in initial permeate flowrate percentage from the RO unit is presented in figure 4.14.

Figure 4.14: Change in removal efficiency of TDS, TSS and colour with the change in initial permeate flowrate (%) in the RO unit for woven dyeing industry 2

4.2.3 Advanced treatment of ETP treated water of denim washing industry

Denim washing is the aesthetic finish, which is given to the denim fabric to enhance the appeal and to provide strength. Several washing effects can be created in the case of denim washing, such as colour fading with or without patchiness, seam puckering, de-pilling, crinkles, hairiness, etc. Two denim washing factories were selected for this study. These denim washing factories 1 and 2 had installed combination of physicochemical and biological ETP and biological ETP respectively in their factories. As previous, advanced treatment consisting of Ultrafiltration and reverse osmosis of the ETP treated water of these factories was done using the mobile filtration unit. Table 4.7 and 4.8 represent amounts of contaminants removal for the denim washing 1 and denim washing 2 factories respectively for permeate to reject flowrate ratio of 80:20, 60:40 and 50:50 in the RO unit.

Table 4.7: Contaminants removal for denim washing industry 1 for permeate to reject flowrate ratio of 80:20, 60:40 and 50:50 in the RO unit

BDL: below detection limit

Figure 4.15: (a) Wastewater (b) ETP treated water (c) UF reject water (d) RO permeate and (e) RO reject water for the washing factory 1

For the denim washing factory 1, high performance was observed in TDS removal. The removal efficiency was found to be 95%, 95.7% and 97.5% respectively for permeate to reject flowrate ratios of 80:20, 60:40 and 50:50. In all cases, the permeate did not contain any suspended solid. For all cases, colour was almost completely removed from the ETP treated water (feed). The ETP treated water contained colour of 88 Pt-Co.

In all cases, fairly good removal of BOD₅ and COD was obtained. The removal efficiency for BOD⁵ varied from 79.6% to 89.3% for the three different permeate to reject flow rate ratio. In case of COD removal, the efficiency varied from 72.4% to 74.4% for the three different permeate to reject flow rate ratio. For the 80:20, 60:40 and 50:50 permeate to reject flow rate ratio, the pH of the permeate was 6.5, 6.7 and 6.5 respectively. The variation of parameters along with the increase in initial permeate flowrate percentage from the RO unit is presented in figure 4.16.

Figure 4.16: Change in removal efficiency of TDS, TSS and colour with the change in initial permeate flowrate (%) in the RO unit for denim washing industry 1

Table 4.8: Contaminants removal for denim washing industry 2 for permeate to reject flowrate ratio of 80:20, 60:40 and 50:50 in the RO unit

BDL: below detection limit

Figure 4.17: (a) Wastewater (b) ETP treated water (c) UF reject water (d) RO permeate and (e) RO reject water for the denim washing factory 2

For denim washing factory 2, in TDS removal, good performance was observed for all cases. For permeate to reject flowrate ratio of 60:40, 80:20 and 50:50, the removal efficiency was found to be 93.4%, 95.3% and 97.9% respectively. In all cases, no suspended solid was found in the permeate. Colour was completely removed from the ETP treated water (feed) for permeate to reject flowrate ratio of 60:40 and 80:20. In the case of permeate to reject flowrate ratio of 50:50, 99.4% colour removal was observed which indicates practically the same efficiency in removing colour from the ETP treated water which contained colour of 177 Pt-Co. A significant amount of BOD₅ and COD removal was obtained for all cases. In case of permeate to reject flowrate ratio of 60:40, The removal efficiency for BOD_5 and COD were found to be 90% and 90.6% respectively. For other ratios, efficiency was found to be almost 92% for both BOD⁵ and COD. For permeate to

reject flowrate ratio of 80:20, 60:40 and 50:50, pH was 7.8, 7.5 and 7.6 respectively. A decreasing pattern of TDS removal was observed with an increase in the initial permeate flowrate percentage from the RO unit. TSS and colour removal were found to be almost constant. BOD₅ removal varied from 90% to 92% whereas COD removal varied from 90.6% to 91.8%. With the increase in initial permeate flowrate percentage, BOD⁵ and COD removal remained mostly consistent. The variation of parameters along with the increase in initial permeate flowrate percentage from the RO unit is presented in figure 4.19.

4.2.4 Reusing permeate as process water

Textile sector consumes large amount of water for various wet processing operations, and hence, has very high water footprint. Currently 98% of the water used by local textile factories is groundwater, which is causing depletion of ground water levels at a high rate [3]. Instead of following any specific guidelines, these local factories use groundwater for their wet processes after softening. The groundwater quality parameters vary slightly in different areas of Dhaka. The range of values of these parameters were collected from the factories. The range for groundwater quality parameter values at Narayanganj and Gazipur for wet processing operations are listed in tables 4.3-4.8.

The quality parameters of groundwater were compared with the permeate water for the selected industries at three permeate flowrate to reject flowrate ratios. For the knit dyeing industry 1, it was observed that TDS, TSS, $BOD₅$ and COD values were within range for all ratios. Slight colour was observed ranging from 2 Pt-Co to 5 Pt-Co. pH value was 6.5. For the knit dyeing factory 2, TDS, BOD₅ and COD values were within range for all ratios. Again, very slight TSS (1 mg/l for all ratios) and colour (1 Pt-Co for permeate to reject flowrate ratio of 80:20 and 2 Pt-Co for other ratios) were observed. pH value was found to be 6.5 here also.

For the woven dyeing industry 1, it was observed that TDS, colour, $BOD₅$ and COD values were within range for all ratios. Very low concentration of TSS was observed corresponding to 1 mg/l. pH value was 6.5. For the woven dyeing industry 2, TDS, BOD₅ and COD values were within range. Very slight colour (1 Pt-Co) and TSS (1 mg/l) were observed for all ratios which are negligible. pH value was found to be 6.5 here also.

For the denim washing industry 1, it was observed that TDS, TSS, BOD₅ and COD values were within range for all ratios. Negligible colour was observed (1 Pt-Co) for permeate to reject flowrate ratio of 80:20 and 50:50. pH values varied from 6.3 to 6.7. For the denim washing industry 2, TDS, TSS, BOD₅ and COD values were within range. Very slight colour (1 Pt-Co) was observed for permeate to reject flowrate ratio of 50:50 which is negligible. pH values varied from 7.5-7.8.

For all industries and for all permeate to reject flowrate ratios, TDS , $BOD₅$ and COD values were well within the range of groundwater quality. Slight TSS and colour were observed in a few cases which are negligible. In the case of denim washing factory 2, pH values were slightly higher than 7.5 for permeate to reject flowrate ratios of 80:20 and 50:50. So, acid and alkali solutions might be required before to reuse the permeate water in wet processing. Overall, it can be said that the permeate water can be successfully reused in all cases for all selected industries.

4.3 Hydraulic performance and economic analysis of a denim washing factory

To further understand the performance of ultrafiltration and reverse osmosis for treating ETPtreated-water, a denim washing factory (which was previously mentioned as denim washing factory 2) was selected for a case study. This factory is located at Gazipur, Dhaka. For fabric processing and other sanitary purposes, the factory requires fresh water at a rate of $800 \text{ m}^3/\text{day}$. The source of this freshwater is groundwater. For treating this wastewater, the industry has a fullscale effluent treatment plant (ETP). This ETP has a design capacity of $100 \text{ m}^3/\text{hour}$. It consists of a conventional biological treatment plant. Figure 4.18 represents the block diagram of the ETP.

The treated effluent coming out of the sedimentation unit of the ETP is normally discharged in inland surface water. For this study, a portion of this water was being stored in a drum and flowed through the mobile UF and RO advanced treatment unit using a submersible pump.

Figure 4.18: Process block diagram of the biological effluent treatment plant located at denim washing factory
4.3.1 Contaminants removal and permeate water reuse

The untreated wastewater sample and ETP treated water sample from the denim washing factory were collected for characterization. Based on the samples, TDS, TSS, colour, BOD₅, COD and pH values were measured. ETP treated water satisfied the national DOE standard for discharge in inland surface water presented in ECR 1997. However, quality of ETP treated water was inferior to that of process water, surface water or groundwater, and cannot be reused as process water. TDS, TSS, colour, BOD5, COD and pH values of untreated wastewater, ETP treated water, DOE standards and process water are listed in table 4.9.

Table 4.9: TDS, TSS, colour, BOD5, COD and pH values of untreated wastewater, ETP treated water, DOE standards and process water for case study

	Untreated	ETP treated	DOE standard for	Process water
Parameter	wastewater	water	inland surface water (Groundwater)	
TDS (mg/l)	587	1120	< 2100	< 180
TSS(mg/l)	38	6	< 150	Nil
$Colour (Pt-Co)$	3350	177		N _{il}
BOD_5 (mg/l)	603	25	< 50	$0.5 - 10$
COD (mg/l)	1440	85	200	$<$ 30
pH	7.2	8	$6-9$	$6.5 - 7.5$

The ETP treated water was continuously fed to the filtration unit. The performance of the mobile filtration unit was evaluated for three different permeate to reject ratio in the reverse osmosis unit. It was assumed that the quality of ETP treated water was consistent during the pilot experiment.

By controlling the reject flowrate from the reverse osmosis unit, the permeate and reject flowrate from the RO unit was maintained at three different ratios in the beginning. After the system became stable, water samples from RO permeate, RO reject and UF reject were collected. Selected water quality parameters were measured and the values of those are listed in table 4.8. The test results given in table 4.8 are average of three readings.

Due to variation in upstream processes, ETP treated water quality might change with time. Since feed water property might have changed slightly during the operation, the actual removal percentage of contaminants may vary from the measured values. This might be a reason for observing no specific trends for all parameters. The variation of parameters along with the increase in initial permeate flowrate percentage from the RO unit is presented in figure 4.19.

Figure 4.19: Change in removal efficiency of TDS, TSS and colour with the change in initial permeate flowrate (%) in the RO unit

It is necessary to know whether the water quality of the permeate changes with time. If the water quality does not meet the requirement for wet processing operations, it cannot be used. So, to understand this phenomenon, it is necessary to operate the advanced filtration system for long periods with the same feed. For conducting this study, a specific permeate flowrate to reject flowrate ratio needs to be maintained to observe the difference. The permeate flowrate to reject flowrate ratio that was selected for this study was 60:40.

By controlling the reject flowrate from the reverse osmosis unit, the permeate and reject flowrate from the RO unit was maintained at a ratio of 60:40 in the beginning. After the system became stable, water samples from RO permeate stream, RO reject stream and UF reject stream were collected. The water quality parameter values of this treatment are presented in table 4.10. After the continuous operation of 2 hours, samples from the same streams were collected again. Based on the samples, TDS, TSS, colour, BOD₅, COD and pH were measured and the values of those are also listed in table 4.10.

BDL: below detection limit

Ultrafiltration does not remove dissolved salts from water. So, TDS in the UF permeate sample was not reduced significantly and found to be 1750 mg/l. After 2 hours, TDS was found to be 1760 mg/l. The readings were directly taken from the digital TDS meter placed in the UF permeate stream.

Adequate TDS removal from the ETP treated water was obtained which was almost 95%. After 2 hours of operation, the TDS removal reached almost 97% which indicates very good performance by the filtration unit.

Total suspended solids were significantly removed from the ETP treated sample. The total removal percentage by the filtration unit remained constant at 100 % after 2 hours.

Colour is a very important parameter in determining the suitability of reusing the UF and RO treated water. 100% removal of colour was obtained by the advanced treatment process (UF and RO) throughout the operation time.

COD and $BOD₅$ are very important parameters for any water. $BOD₅$ is a measure of the amount of oxygen needed for bacteria to break down the organic molecules present in the water whereas COD is the amount of total oxygen required to chemically oxidize all organic matter in the water. In the beginning, 90% BOD₅ removal was obtained which decreased to 84% after 2 hours of operation. Similarly, COD removal varied from 90.6% to 86% after 2 hours.

The pH of the permeate sample was found to vary between 7.5 and 7.7. These values are in line with national and international standards. Since all the parameter values remain well within range even after 2 hours of operation, they can be successfully reused in the wet processing operations. But with time, their flowrate will decrease due to build-up of resistance across the membranes.

4.3.2 Membrane hydraulic performance and longevity

The hydraulic performance of the membranes was observed for the initial permeate flowrate to reject flowrate ratio of 60:40 in the reverse osmosis unit. Before the operation, the entire filtration system was backwashed including the membranes using freshwater. During the whole operation, no backwashing or chemical cleaning was done. Permeate from the ultrafiltration system directly passed to the reverse osmosis unit. No water sample was taken from the ultrafiltration permeate. The initial feedwater flowrate to the ultrafiltration unit was observed to be around 1770 l/h. The

initial permeate flowrate was observed to be around 95%. This corresponded to a specific flow of 24 (l/h)/m².

The whole operation was conducted for 2 hours. During this time, permeate flowrate from the reverse osmosis unit decreased gradually since the permeate flow from the ultrafiltration unit also decreased with time. Initially permeate flowrate from the reverse osmosis filtration unit was observed to be 900 l/h which corresponded to a specific flow of 24.3 $(l/h)/m^2$. After 2 hours, the permeate flow rate decreased to 780 l/h which corresponded to a specific flow of 14.6 (l/h)/m². At start-up, permeate to reject flowrate ratio was 62.5:37.5 and after 2 hours, the ratio became 57.1:42.9. Almost 40% of the feed to the reverse osmosis unit was rejected as concentrate into the environment. At this rate and considering backwashing of membranes every 8 hours, $16 \text{ m}^3/\text{day}$ of permeate water can be obtained from the advanced filtration system.

Many factors affect the separation efficiency of reverse osmosis (RO) desalination (e.g. salt rejection and permeate flux) and membrane longevity. These factors include, but not limited to, membrane fouling and scaling, consistency of the quality of feed water and the efficacy of treatment, and stability and reliability of plant process equipment [55]. Several studies have endeavoured to extend the lifetime of the RO membranes. However, even with the required feedwater pre-treatment, the lifetime of the membranes is normally limited to 3-5 years, or 5-7 years [56].

As suggested by the supplier, the feedwater quality for the UF unit is given as the effluent water quality from ETP primary treatment. The feedwater quality for the RO unit is given as water having TDS<3000 ppm, pH: 6.5-7.5 and chlorine concentration<0.1 ppm. The ETP treated water in this study already had good enough quality to be discharged into the environment. This water was further flowed through a 100-micron pre-filter before entering the UF membranes to remove most contaminants and suspended solids. So, considering the quality of feed water, the lifespan of the UF and RO membranes was taken as 5 years for this study.

4.3.3 Operating and Investment costs and water requirement of the factory

Based on the results obtained, the following economic considerations can be drawn. Considering permeate quality and backwash for RO unit, permeate and reject flowrate ration 60:40 was considered the optimum ratio out of three set ratios used in this study. Considering the membrane surface areas of UF and RO units used in this study, costs to treat full volume of ETP treated water of that specific factory were calculated. For the above assumptions, the following economic data can be found considering operating life of 5 years that is shown in table 4.11. The capital cost and operating cost were calculated after consulting with KOCH Separation Solutions Ltd. and SUEZ Water Technologies and Solutions Ltd. that are leading separation membranes supplier around the world.

Table 4.11: Operating and investment costs for ultrafiltration and reverse osmosis membrane units to treat ETP treated textile wastewater (permeate to reject flowrate ratio of 60:40)

a assuming electricity cost \$0.091 per kWh as per Dhaka Power Distribution Company Limited (DPDC) and 330 working days in a year

Calculation for Operating and Investment costs of Denim Washing industry 2 is as follows.

Investment cost:

Water requirement of the factory= $800 \text{ m}^3/\text{day} = 33.33 \text{ m}^3/\text{hr}$

Price of ultrafiltration membrane = ϵ 35 (euro)/m²

For permeate to reject flowrate ratio of 60:40, reusable water from the filtration plant

 $=800\times0.95\times0.60 \text{ m}^3/\text{day} = 456 \text{ m}^3/\text{day} = 19 \text{ m}^3/\text{hr}$

For 24 LMH flow from ultrafiltration membrane, membrane area for ultrafiltration

$$
=\frac{33.33\times1000}{24} \text{ m}^2 = 1388.89 \text{ m}^2
$$

Price of ultrafiltration membrane= ϵ 35 (euro)/m² × 1388.89 m² × 1.19 USD/1 ϵ (euro)= \$57847

For 24 LMH flow from reverse osmosis membrane, Price of reverse osmosis membrane= \$17136 (collected from SUEZ Corporation)

Investment cost= $$57847 + $17136 = 74983

After rounding up, investment cost= \$75000

Operating cost:

Pumping energy= 0.75 kWh/m³ water

Per day electricity consumption= 600 kWh

Electricity consumption cost and cleaning chemicals cost= 8.89 BDT/ $m³$ water

 $=8.89\times800\times330 = 2346960$ BDT/year

After rounding up, the yearly cost is \$28000 (*assuming electricity cost \$0.091 per kWh as per Dhaka Power Distribution Company Limited (DPDC) and 330 working days in a year)*

Total cost in 5 years (Investment cost + Operating cost) = $$215,000$

Water to be collected in 5 years = $456 \times 330 \times 5 = 752,400$ m³

Water production cost= $\frac{$215,000}{$752,400 \text{ m}^3}$ = \$0.286/m³ = 24.29 BDT/m³

From the above calculation, cost for advanced water treatment was found to be **24.29 BDT/m³** over the period of 5 years.

The ETP treatment cost for wastewater varies based on the type of the ETP. The operating costs of ETPs can also vary greatly depending on quality and quantity of inputs such as chemicals, the efficiency and size of motors and therefore the energy required, the method of treatment and the efficiency of ETP management. The treatment cost usually varies from $15{\text -}150$ BDT/m³ of water treated [57]. The denim washing factory 2 has a biological ETP installed in their facility. Since biological ETP treatment cost is usually lower than other type of effluent treatment plants. It is assumed as 20 BDT $/m³$ for this analysis. The cost for pumping groundwater is on average 6.5

BDT/m³. Considering the reusable water volume from the advanced treatment (UF and RO) and the groundwater volume required for wet processing operations, the overall cost for using water in the industry can be calculated.

Water reused in the industry= $456 \text{ m}^3/\text{day}$

Water pumped from ground $=(800-456)$ m³/day = 344 m³/day

ETP treatment cost = 20 BDT/m³ of treated water

and advanced treatment cost= 24.29 BDT/ $m³$ of water produced

Water pumping cost= 6.5×344=2236 BDT/day

Total cost (Groundwater pumping cost $+$ ETP treatment cost $+$ Advanced treatment cost) $=$ 6.5×344+20×800+24.29×456= 29312.24 BDT/day.

Overall cost= $\frac{29312.24 \text{ BDT/day}}{800 \text{ m}^3/\text{day}}$ = 36.64 BDT/m³ of water used in the industry

If high permeate flowrate to reject flowrate ratio is used, freshwater production rate will increase. But at the same time, more frequent backwash will be required, and depreciation of the membranes will be faster. This will in turn increase the water production cost since membranes will have less longevity and will be needed to change more frequently.

Considering the same assumptions made for the case study, similar economic conclusions can be drawn for the other selected industries for the permeate to reject flowrate ratio of 60:40. Considering operating life of 5 years, the results are summarized in table 4.12.

Industry	Water	Capital	Operating	Advanced	Advanced Water
	requirement	cost()	cost/year	Water	treatment cost
	(m^3/day)		$(\$\)$	treatment cost	(BDT/m^3)
				$(\frac{\text{S}}{\text{m}^3})$	
Knit dyeing factory 1	3250	305,000	113,000	0.285	24.19
Knit dyeing factory 2	2880	270,000	100,000	0.28	24.16
Woven dyeing 1	4200	393,000	145,000	0.283	24.06
Woven dyeing 2	6500	608,000	225,000	0.283	24.10
Denim washing 1	1100	103,000	38,000	0.283	24.07
Denim washing 2	800	75,000	28,000	0.286	24.29

Table 4.12: Water treatment cost for ultrafiltration and reverse osmosis membrane units to treat ETP treated textile wastewater for the selected industries

Large number of advanced filtration units may result in land area crisis. For these cases, filtration units with higher filtration capacity might be used. This will reduce the capital cost to some extent and also save land area. It will also reduce water required to backwash the membranes. In turn, water treatment cost will also decrease.

4.3.4 Water footprint of the factory

The water footprint (WF) was first proposed in 2002 as a measure of the quantity of freshwater used for productive activities, both in terms of the amount of water consumed (green and blue WF) and the quantity of water contaminated (grey WF). The volume of rainwater collected in the root zone of the soil and evaporated, transpired, or absorbed by plants is referred to as the green water footprint. The volume of surface and groundwater required (evaporated or consumed directly) for the creation of a good or service is referred to as the blue water footprint. The amount of water required to assimilate a pollution load in order to achieve specified water quality requirements that reaches a water body is referred to as the grey water footprint [58]. In terms of agricultural water consumption for cotton farming, high water uses in textile production, and water contamination, the textile sector as a whole has a massive water footprint.

For denim washing factory 2, the groundwater that is being used in the industry is considered as blue water and this corresponds to blue water footprint of 800 $\mathrm{m}^3/\mathrm{day}$. For the permeate to reject flowrate ratio of 60:40, 60% of the water is being reused in the process. This gives the opportunity to reduce blue water footprint up to 60%. The rest requirement of the factory is to be fulfilled by withdrawing groundwater.

After treating in the effluent treatment plant, the treated effluent is released in the environment. Usually, 1 or 2% volume of water is removed from the process and the rest is released in the environment. So, this volume can be considered as constant. For this volume and considering the pollution load of the effluent, the corresponding grey water footprint was found to be 391.11 m³/day. After being treated by the advanced filtration unit (UF and RO), reject streams from UF and RO unit are released in the environment. For these two separate streams, grey water footprint was found to be 270.75 m^3 /day combinedly. This indicated that grey water footprint was reduced by 120.36 m³/day (30.8%).

Calculation for grey water footprint of Denim Washing industry 2 is as follows.

Grey water footprint (GWF)=
$$
\frac{L}{C_{max} - C_{nat}}
$$
 [volume/time]

where, L=the pollutant load entering a water body (mass/time)

 C_{max} = the maximum acceptable concentration in mass/volume

 $C_{nat} =$ its natural background concentration in the receiving water body in mass/volume

For a point source, the pollutant load, L can be calculated as the effluent volume (Effl, in volume/time) multiplied by the concentration of the pollutant in the effluent (C_{eff} , in mass/volume) minus the water volume of the abstraction (Abstr, in volume/time) multiplied by the actual concentration of the intake water $(C_{\text{act}},$ in mass/volume).

In this study, pollution load is calculated for $BOD₅$ since grey water footprint for $BOD₅$ was found to be the largest when comparing with other contaminants. The ambient water quality standard (C_{max}) was assumed to be 50 ppm [29], natural concentration (C_{nat}) in the receiving water body (surface water) and actual concentration (C_{act}) were assumed as 5 ppm and 3 ppm respectively [59].

For the ETP treated water, in terms of pollutant $BOD₅$,

$$
GWF = \frac{800 \times (25-3)}{50-5} = 391.11 \text{ m}^3/\text{day}
$$

For the UF reject stream, flowrate = $800 \times .05 = 40 \text{ m}^3/\text{day}$

RO reject stream flowrate = $800 \times .95 \times .40 = 304 \text{ m}^3/\text{day}$

GWF of UF reject stream = $\frac{40 \times (110 - 3)}{50 - 5}$ =95.11 m³/day

GWF of RO reject stream = $\frac{304 \times (29-3)}{50-5}$ = 175.64 m³/day

Total GWF of reject streams = $270.75 \text{ m}^3/\text{day}$

Reduced GWF= $120.36 \text{ m}^3/\text{day}$.

In similar way, the reduced GWF of the other selected industries were calculated. The values are tabulated in table 4.13.

Factory	GWF of ETP treated water (m^3/day)	GWF of Reject streams (m^3/day) (After advanced treatment)	Reduced GWF (m^3/day)
KD#1	2094.44	1939.9	$154.6(7.4\%)$
KD#2	16384	815.4	15568.6 (95%)
WD#1	14000	2181.6	11818.6 (84.4%)
WD#2	6933.33	1822.6	5110.7 (73.7%)
DW#1	880	359.8	520.2 (59.1%)
DW#2	391.11	270.8	120.4 (30.8%)

Table 4.13: Grey water footprint of the selected textile factories

The ETP treated water of knit dyeing factory 1 had $BOD₅$ of 32 mg/l as shown in table 4.2. After advanced treatment, reject streams from UF and RO contained BOD⁵ of 31 and 70 mg/l respectively. This corresponded to reduction of Grey Water Footprint by 7.4%. For knit dyeing factory 2, ETP treated water had BOD₅ of 259 mg/l. After advanced treatment by UF and RO, reject streams from UF and RO contained BOD₅ of 83 and 26 mg/l respectively. Since the pollutant load in the reject streams was greatly reduced, the Grey Water Footprint was also significantly reduced. This corresponded to reduction of Grey Water Footprint by 95%.

As indicated in table 4.2, the ETP treated water of woven dyeing factory 1 had a $BOD₅$ of 153 mg/l. BOD₅ levels in UF and RO reject streams were 83 and 50 mg/l, respectively, after advanced treatment. This equated to 84.4% reduction in Grey Water Footprint. ETP treated water from Woven Dyeing Factory 2 had a BOD₅ of 51 mg/l. UF and RO reject streams had BOD₅ levels of 88 and 22 mg/l, respectively, after advanced treatment. Since the pollutant load in the reject streams was greatly reduced, the Grey Water Footprint was also significantly reduced. This corresponded to reduction of Grey Water Footprint by 73.7%.

The ETP treated water from denim washing factory 1 had a $BOD₅$ of 39 mg/l, as shown in table 4.2. After advanced treatment, BOD₅ values in UF and RO reject streams were 15 and 40 mg/l, respectively. This amounted to an 59.1% decrease in Grey Water Footprint. The BOD₅ level in ETP treated water from denim washing factory 2 was 25 mg/l. After advanced treatment, $BOD₅$ values in UF and RO reject streams were 110 and 29 mg/l, respectively. This equated to 33% reduction in Grey Water Footprint.

If a relationship between water quality parameters and grey water footprint can be established for a certain type of feedwater to the advanced filtration system (UF and RO), the possible reduction in grey water footprint can be calculated from that water quality parameter value. This correlation will be crucial in taking decision about using advanced treatment for the initially treated water. It will help to evaluate the potential of advanced treatment in terms of increasing pollution load to the environment. The GWF in this study was calculated based on the pollutant load of BOD5. Change in grey water footprint reduction $\frac{6}{9}$ with BOD₅ of the feed (ETP treated water) for the advanced treatment UF and ROis presented in figure 4.20. The figure shows values for the selected six textile factories. It can be observed that with increase in the BOD⁵ values for the feed water, the GWF reduction percent increased for all factories except for knit dyeing factory 1. The rising trend indicates that GWF reduction percent (%) is independent of the type of textile factories and mostly depends on the water quality parameter, BOD₅. However, further experimental results are required to verify this observation.

Figure 4.20: Change in grey water footprint reduction (%) with BOD⁵ of ETP treated water for the selected textile factories

The reject streams from the UF and RO unit contain less BOD⁵ than the feedwater (ETP treated water) to the advanced treatment unit (UF and RO) in most cases. Without any treatment, this feedwater with high pollution load would be discharged into the environment. Thus, advanced treatment results in the reduction of grey water footprint to a good extent.

4.4 General Discussion

Ultrafiltration and reverse osmosis are used as membrane separation processes for advanced treatment of effluent treatment. In this study, these two processes were combinedly used for removal of contaminants from the ETP treated water. Ultrafiltration removes molecules or colloids with high molecular weight. It does not remove small ions or molecules. However, using the advanced filtration unit (UF and RO) for the two woven dyeing industries, a fairly neutral and a declining trend of TDS values were observed respectively with increase in initial permeate flowrate to reject flowrate ratio in the reverse osmosis unit. But for the selected denim washing industries and the knit dyeing industries, an increasing trend of TDS value was observed.

TSS and colour were fairly removed from all selected industries for all permeate flowrate to reject flowrate ratios. For BOD₅ and COD removal, no consistency was found in removal efficiency for different permeate flowrate to reject flowrate ratios.

There was decrease in pH value after treatment by ultrafiltration and reverse osmosis. The pH values of ETP treated water that was used as feedwater for advanced filtration system were above 7.5 in most cases which means it was mostly basic. After treatment, the pH values fairly decreased to around 6.5 in most cases which made the treated water slightly acidic. But no trend was observed for changes in permeate flowrate to reject flowrate ratios.

Physicochemical and biological processes handle the polluting effluents of textile and dying industries that are of major environmental concerns. Membrane technologies including Reverse Osmosis / Nano filtration and ultra-filtration are used in the physicochemical process. Effluent treatments are mainly successful, with RO generating 50-80% of the permeate that can be used. But the remaining 30-50% of reject water from reverse osmosis contains a high concentration of total dissolved solids and organics [60]. This might be an opportunity to recover resources like salts after proper separation procedure. This also gives an opportunity to reuse the water separated from salts and organics. But extensive energy consumption will be required for this. The scope of the study did not include the management of reject water. However, different methods like multiple effect evaporation (MEE), electrodialysis (ED), etc. can be used to recover resources like salt and fresh water from the reject water streams.

Multiple Effect Evaporators (MEE) can be used to remove salts and other solids from the reject water streams. The combined reject from ultrafiltration and reverse osmosis membranes can be sent to the multiple effect evaporators. The condensate from MEE and permeate from RO can be reused as process water. The main drawback for this process is the large energy requirement for steam production.

Electrodialysis (ED) can be used for concentrating rejects collected from reverse osmosis (RO) plants to decrease the volume load on evaporators. The efficacy of the Electrodialysis membrane method in achieving the required concentration of the textile discharge treated with RO is well established [61]. Therefore, ED should be able to concentrate the RO reject obtained from textile industrial effluent successfully. In the energy-efficient treatment of RO rejects with low COD concentration, ED was found to be particularly applicable; the dilute stream obtained could then be returned to the RO plant to further recover reusable process water.

The reject streams from UF and RO need to be discharged into the environment unless methods like Multiple effect evaporation, electrodialysis, etc. are used. The UF reject streams from most of the selected factories, except for woven dyeing 1 and denim washing 2 factories, can be directly discharged into the environment since they satisfy the discharge standard into the environment. Woven dyeing 1 factory does not satisfy discharge standard for TDS, BOD₅ and COD whereas denim washing factory does not satisfy discharge standard for BOD₅ and COD [29].

In most cases, the RO reject streams cannot be discharged directly into the environment as per given guidelines by DOE. For the woven dyeing factories and denim washing 2 factories, TDS value did not satisfy the guideline given in ECR 1997. For the knit dyeing 1 factory, TDS and BOD⁵ value did not satisfy the ECR 1997 rules. However, for knit dyeing 2 and denim washing 1 factory, the reject streams satisfied the discharge standard. So they can be directly discharged into the environment [29].

Another issue that can rise concern is the management of used membranes after their lifespan expires. Their management was not in the scope of this study. However, because they're often made of polymers, they can be converted to other useful polymer forms. They can be disposed of by controlled burning in incinerators if they become utterly unusable.

An important contribution of this study is the reduction of blue and grey water footprint. The blue water footprint could be reduced from 50% up to 80% based on the operation procedure of the reverse osmosis unit. Although it was assumed that the grey water footprint would remain the same after advanced treatment, it was observed that it was reduced by a significant amount, ranging from 7.4% to 95%.

Knit dyeing, woven dyeing and washing processes have significantly different operations. Even the same operations can be done using different chemicals. These chemicals might have different pollution loads and different water footprints. So, it is difficult to scrutinize the performance of advanced treatment ultrafiltration and reverse osmosis based on the type of textile industry. More number of industries and the pollution load of the used chemicals need to be scrutinized for this study.

Chapter 5: Conclusion and Future work

5.1 Conclusion

Advanced membrane processes ultrafiltration and reverse osmosis have proven to be promising purification methods for textile wastewater reuse, with direct environmental benefits. It is necessary to know how impactful these advanced processes can be to successfully reuse textile effluents in Bangladesh. The study conducted in this thesis, investigated the possibility to reuse the huge volume of textile effluents generated. This study also analyzed the economic considerations for establishing these advanced processes in the industries.

The results of this study show that for the selected industries (knit dyeing, woven dyeing and denim washing), the ETP treated water satisfies the local guidelines for disposal into the environment. After further treating this water with ultrafiltration and reverse osmosis, the water achieves the recyclable water quality. This quality satisfies the groundwater quality used by the local textile industries. Thus, it is quite possible for the industries to reuse it in different wet processes. This will help to reduce demand for groundwater.

The study also considered the blue and grey water footprint associated with the factory. It was found that blue water footprint can be reduced up to 80% by maintaining permeate to reject flowrate ratio in the RO unit. Grey water footprint was also measured, and it was found that after UF and RO treatment, the GWF can be reduced from 154.6 m^3/day (7.4%) to 15568.6 m^3/day (95%). This reduction in footprint will help conserve the groundwater and surface water.

The study also determines the investment and operating costs for using ultrafiltration and reverse osmosis in the selected industries. Considering 5 years for operating life of membranes, the added treatment cost would vary between 24.06 BDT/ $m³$ to 24.29 BDT/ $m³$ for the selected industries for permeate to reject flowrate ratio of 60:40. Although this cost is a lot higher than the pumping cost of groundwater, it might become a necessity within the next few decades. However, it will also give other direct and indirect benefits, such as: reduction of ground water consumption, reduction of ground water pumping cost, reduction of ground water treatment cost (when applicable), and reduction of grey and blue water footprint.

5.2 Recommendations for Future Work

There is a lot of scope to improve the current wastewater management system of the textile industry. This study conducted here may just be the beginning of it. This study can help to further investigate many crucial issues associated with the textile wastewater management. Some recommendations for research in future are suggested below.

- 1. The reject stream from the filtration unit was allowed to discharge into the environment. Instead of discharging it, the reject samples can be further analyzed to recover the resources. This stream contains dissolved solids and salts like NaCl and Na2SO4. This stream can be further treated by Nanofiltration or other processes to recover materials for reuse which can be investigated.
- 2. The study of hydraulic performance of the membranes and economic analysis was done for permeate to reject ratio of 60:40. For other ratios, the study can be done.
- 3. Zero liquid discharge (ZLD) is a new concept for Bangladesh. But many countries are already trying to implement ZLD in their country. The possibility of achieving ZLD for this process can be further investigated.
- 4. Effects of temperature on the lifetime and performance of the used membranes can be investigated. The material selection for the membranes can also be studied for the selected textile industries.
- 5. The used Reverse osmosis membrane was operated at low pressure. The efficiency of highpressure RO along with ultrafiltration as pre-treatment can be analyzed at a different study.

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Appendix

Appendix A: Spectrophotometer, HACH DR 6000

The DR6000™ is an advanced laboratory spectrophotometer for water testing. It offers high-speed wavelength scanning across the UV and Visible Spectrum and comes with over 250 preprogrammed methods including the most common water and environmental testing methods used today. With optional accessories allowing for high-volume testing via a carousel sample changer, and increased accuracy with a sample delivery system that eliminates optical difference errors, this instrument ensures to handle wide-ranging water testing needs [62].

The DR 6000 is a UV-VIS-spectrophotometer with a wavelength range of 190 to 1100 nm. The visible spectrum (320 to 1100 nm) is covered by a halogen lamp and a deuterium lamp produces the light in the ultraviolet spectrum (190 to 360 nm). The instrument is supplied with a complete range of application programs and supports several languages.

Figure A1: HACH DR 6000 Spectrophotometer

The DR 6000 spectrophotometer contains the following programs and operating modes:

• Stored programs (pre-installed tests)

- Barcode Programs
- User Programs
- Favorites
- Single Wavelength
- Multi Wavelength
- Wavelength Scan
- Time course

The DR 6000 spectrophotometer provides digital readouts of concentration, absorbance and percent transmittance [63].

Appendix B: COD Measurement

Standard Method 5220 D (Closed Reflux, Colorimetric Method)

Used Reagents

a. Digestion solution, high range: Add to about 500 mL distilled water 10.216 g $K_2Cr_2O_7$, primary standard grade, previously dried at 150° C for 2 h, 167 mL conc H₂SO4, and 33.3 g HgSO4. Dissolve, cool to room temperature, and dilute to 1000 ml.

b. Digestion solution, low range: Prepare as in 'a section' but use only 1.022 g potassium dichromate.

c. Sulfuric acid reagent: Add Ag2SO4, reagent or technical grade, crystals, or powder, to conc. H2SO⁴ at the rate of 5.5 g Ag2SO4/kg H2SO4. Let stand 1 to 2 d to dissolve. Mix.

d. Sulfamic acid: Required only if the interference of nitrites is to be eliminated.

e. Potassium hydrogen phthalate standard: Lightly crush and then dry KHP to constant weight at 110°C. Dissolve 425 mg in distilled water and dilute to 1000 ml. KHP has a theoretical COD of 1.176 mg O₂/mg and this solution has a theoretical COD of 500 μ g O₂/ml. This solution is stable when refrigerated, but not indefinitely. Be alert to development of visible biological growth. If practical, prepare and transfer solution under sterile conditions. Weekly preparation usually is satisfactory.

Procedure

a. Treatment of samples: Measure suitable volume of sample and reagents into tube or ampule. Prepare, digest, and cool samples, blank, and one or more standards. Note the safety precautions. It is critical that the volume of each component be known and that the total volume be the same for each reaction vessel. If volumetric control is difficult, transfer digested sample, dilute to a known volume, and read. Premixed reagents in digestion tubes are available commercially.

b. Measurement of dichromate reduction: Cool sample to room temperature slowly to avoid precipitate formation. Once samples are cooled, vent, if necessary, to relieve any pressure generated during digestion. Mix contents of reaction vessels to combine condensed water and dislodge insoluble matter. Let suspended matter settle and ensure that optical path is clear.

Measure absorption of each sample blank and standard at selected wavelength (420 nm or 600 nm). At 600 nm, use an undigested blank as reference solution. Analyze a digested blank to confirm good analytical reagents and to determine the blank COD; subtract blank COD from sample COD. Alternately, use digested blank as the reference solution once it is established that the blank has a low COD. At 420 nm, use reagent water as a reference solution. Measure all samples, blanks, and standards against this solution. The absorption measurement of an undigested blank containing dichromate, with reagent water replacing sample, will give initial dichromate absorption. Any digested sample, blank, or standard that has a COD value will give lower absorbance because of the decrease in dichromate ion. Analyze a digested blank with reagent water replacing sample to ensure reagent quality and to determine the reagents' contribution to the decrease in absorbance during a given digestion. The difference between absorbances of a given digested sample and the digested blank is a measure of the sample COD. When standards are run, plot differences of digested blank absorbance and digested standard absorbance versus COD values for each standard.

c. Preparation of calibration curve: Prepare at least five standards from potassium hydrogen phthalate solution with COD equivalents to cover each concentration range. Make up to volume with reagent water; use same reagent volumes, tube, or ampule size, and digestion procedure as for samples. Prepare calibration curve for each new lot of tubes or ampules or when standards prepared differ by $\geq 5\%$ from calibration curve. Curves should be linear. However, some nonlinearity may occur, depending on instrument used and overall accuracy needed.

Calculation

If samples, standards, and blanks are run under same conditions of volume and optical path length, calculate COD as follows:

$$
COD \text{ as mg O}_2/L = \frac{mg O_2 \text{ in final volume} \times 1000}{ml \text{ sample}}
$$

Preferably analyze samples in duplicate because of small sample size. Samples that are inhomogeneous may require multiple determinations for accurate analysis. These should not differ

from their average by more than 5% for the high-level COD test unless the condition of the sample dictates otherwise [64]**.** For this study, the prepared calibration curve is showed in figure B1.

Figure B1: Calibration curve for spectrophotometer reading and COD values in mg/l

Using this curve, the COD values were measured for separate readings in the spectrophotometer.

Appendix C: BOD⁵ Measurement

Standard Method 5210 B (5-day BOD test)

Apparatus

a. Incubation bottles: Use glass bottles having 60 mL or greater capacity (300-mL bottles having a ground-glass stopper and a flared mouth are preferred). Clean bottles with a detergent, rinse thoroughly, and drain before use. As a precaution against drawing air into the dilution bottle during incubation, use a water seal. Obtain satisfactory water seals by inverting bottles in a water bath or by adding water to the flared mouth of special BOD bottles. Place a paper or plastic cup or foil cap over flared mouth of bottle to reduce evaporation of the water seal during incubation.

b. Air incubator or water bath, thermostatically controlled at 20 ± 1 °C. Exclude all light to prevent possibility of photosynthetic production of DO.

Reagents

Prepare reagents in advance but discard if there is any sign of precipitation or biological growth in the stock bottles. Commercial equivalents of these reagents are acceptable and different stock concentrations may be used if doses are adjusted proportionally.

a. Phosphate buffer solution: Dissolve 8.5 g KH2PO4, 21.75 g K2HPO4, 33.4 g Na 2HPO4⋅7H2O, and 1.7 g NH4Cl in about 500 mL distilled water and dilute to 1 L. The pH should be 7.2 without further adjustment. Alternatively, dissolve $42.5 \text{ g } KH_2PO_4$ or $54.3 \text{ g } K_2HPO_4$ in about 700 mL distilled water. Adjust pH to 7.2 with 30% NaOH and dilute to 1 L.

b. Magnesium sulfate solution: Dissolve 22.5 g MgSO4⋅7H2O in distilled water and dilute to 1 L.

c. Calcium chloride solution: Dissolve 27.5 g CaCl₂ in distilled water and dilute to 1 L.

d. Ferric chloride solution: Dissolve 0.25 g FeCl3⋅6H2O in distilled water and dilute to 1 L.

e. Acid and alkali solutions, 1N, for neutralization of caustic or acidic waste samples.

1) Acid—Slowly and while stirring, add 28 mL conc sulfuric acid to distilled water. Dilute to 1 L.

2) Alkali—Dissolve 40 g sodium hydroxide in distilled water. Dilute to 1 L.

f. Sodium sulfite solution: Dissolve 1.575 g Na₂SO₃ in 1000 mL distilled water. This solution is not stable; prepare daily.

g. Nitrification inhibitor, 2-chloro-6-(trichloromethyl) pyridine.

h. Glucose-glutamic acid solution: Dry reagent-grade glucose and reagent-grade glutamic acid at 103°C for 1 h. Add 150 mg glucose and 150 mg glutamic acid to distilled water and dilute to 1 L. Prepare fresh immediately before use.

i. Ammonium chloride solution: Dissolve 1.15 g NH4Cl in about 500 mL distilled water, adjust pH to 7.2 with NaOH solution, and dilute to 1 L. Solution contains 0.3 mg N/ml.

j. Dilution water: Use demineralized, distilled, tap, or natural water for making sample dilutions.

Procedure

a. Preparation of dilution water: Place desired volume of water in a suitable bottle and add 1 mL each of phosphate buffer, MgSO₄, CaCl₂, and FeCl₃ solutions/L of water.

Seed dilution water, if desired. Test dilution water so that water of assured quality always is on hand. Before use bring dilution water temperature to 20 ± 3 °C. Saturate with DO by shaking in a partially filled bottle or by aerating with organic-free filtered air. Alternatively, store in cottonplugged bottles long enough for water to become saturated with DO. Protect water quality by using clean glassware, tubing, and bottles.

b. Dilution water storage: Source water may be stored before use as long as the prepared dilution water meets quality control criteria in the dilution water blank. Such storage may improve the quality of some source waters but may allow biological growth to cause deterioration in others. Preferably do not store prepared dilution water for more than 24 h after adding nutrients, minerals, and buffer unless dilution water blanks consistently meet quality control limits. Discard stored source water if dilution water blank shows more than 0.2 mg/L DO depletion in 5 d.

c. Glucose-glutamic acid check: Because the BOD test is a bioassay its results can be influenced greatly by the presence of toxicants or by use of a poor seeding material. Distilled waters frequently are contaminated with copper; some sewage seeds are relatively inactive. Low results always are obtained with such seeds and waters. Periodically check dilution water quality, seed effectiveness, and analytical technique by making BOD measurements on a mixture of 150 mg glucose/L and

150 mg glutamic acid/L as a ''standard'' check solution. Glucose has an exceptionally high and variable oxidation rate but when it is used with glutamic acid, the oxidation rate is stabilized and is similar to that obtained with many municipal wastes. Alternatively, if a particular wastewater contains an identifiable major constituent that contributes to the BOD, use this compound in place of the glucose-glutamic acid.

Determine the 5-d 20°C BOD of a 2% dilution of the glucose-glutamic acid standard check solution. Adjust concentrations of commercial mixtures to give 3 mg/L glucose and 3 mg/L glutamic acid in each GGA test bottle. Evaluate data, Precision and Bias.

d. Seeding: 1) Seed source—It is necessary to have present a population of microorganisms capable of oxidizing the biodegradable organic matter in the sample. Domestic wastewater, unchlorinated or otherwise-undisinfected effluents from biological waste treatment plants, and surface waters receiving wastewater discharges contain satisfactory microbial populations. Some samples do not contain a sufficient microbial population (for example, some untreated industrial wastes, disinfected wastes, high-temperature wastes, or wastes with extreme pH values). For such wastes seed the dilution water or sample by adding a population of microorganisms. The preferred seed is effluent or mixed liquor from a biological treatment system processing the waste. Where such seed is not available, use supernatant from domestic wastewater after settling at room temperature for at least 1 h but no longer than 36 h. When effluent or mixed liquor from a biological treatment process is used, inhibition of nitrification is recommended. Some samples may contain materials not degraded at normal rates by the microorganisms in settled domestic wastewater. Seed such samples with an adapted microbial population obtained from the undisinfected effluent or mixed liquor of a biological process treating the waste. In the absence of such a facility, obtain seed from the receiving water below (preferably 3 to 8 km) the point of discharge. When such seed sources also are not available, develop an adapted seed in the laboratory by continuously aerating a sample of settled domestic wastewater and adding small daily increments of waste. Optionally use a soil suspension or activated sludge, or a commercial seed preparation to obtain the initial microbial population. Determine the existence of a satisfactory population by testing the performance of the seed in BOD tests on the sample. BOD values that increase with time of adaptation to a steady high value indicate successful seed adaptation.

2) Seed control—Determine BOD of the seeding material as for any other sample. This is the seed control. From the value of the seed control and a knowledge of the seeding material dilution (in the dilution water) determine seed DO uptake. Ideally, make dilutions of seed such that the largest quantity results in at least 50% DO depletion. A plot of DO depletion, in milligrams per liter, versus milliliters of seed for all bottles having a 2-mg/L depletion and a 1.0-mg/L minimum residual DO should present a straight line for which the slope indicates DO depletion per milliliter of seed. The DO-axis intercept is oxygen depletion caused by the dilution water and should be less than 0.1 mg/L. Alternatively, divide DO depletion by volume of seed in milliliters for each seed control bottle having a 2-mg/L depletion and a 1.0-mg/L residual DO. Average the results for all bottles meeting minimum depletion and residual DO criteria. The DO uptake attributable to the seed added to each bottle should be between 0.6 and 1.0 mg/L, but the amount of seed added should be adjusted from this range to that required to provide glucose-glutamic acid check results in the range of 198 ± 30.5 mg/L. To determine DO uptake for a test bottle, subtract DO uptake attributable to the seed from total DO uptake. Techniques for adding seeding material to dilution water are described for two sample dilution methods.

e. Sample pretreatment: Check pH of all samples before testing unless previous experience indicates that pH is within the acceptable range.

1) Samples containing caustic alkalinity (pH >8.5) or acidity (pH <6.0)—Neutralize samples to pH 6.5 to 7.5 with a solution of sulfuric acid (H_2SO_4) or sodium hydroxide (NaOH) of such strength that the quantity of reagent does not dilute the sample by more than 0.5%. The pH of dilution water should not be affected by the lowest sample dilution. Always seed samples that have been pH-adjusted.

2) Samples containing residual chlorine compounds—If possible, avoid samples containing residual chlorine by sampling ahead of chlorination processes. If the sample has been chlorinated but no detectable chlorine residual is present, seed the dilution water. If residual chlorine is present, dechlorinate sample and seed the dilution water. Do not test chlorinated/dechlorinated samples without seeding the dilution water. In some samples, chlorine will dissipate within 1 to 2 h of standing in the light. This often occurs during sample transport and handling. For samples in which chlorine residual does not dissipate in a reasonably short time, destroy chlorine residual by adding Na₂SO₃ solution. Determine required volume of Na₂SO₃ solution on a 100- to 1000-mL portion of neutralized sample by adding 10 mL of $1 + 1$ acetic acid or $1 + 50$ H₂SO₄, 10 mL potassium iodide (KI) solution (10 g/100 mL) per 1000 mL portion and titrating with $Na₂SO₃$ solution to the starchiodine end point for residual. Add to neutralized sample the relative volume of $Na₂SO₃$ solution determined by the above test, mix, and after 10 to 20 min check sample for residual chlorine.

3) Samples containing other toxic substances—Certain industrial wastes, for example, plating wastes, contain toxic metals. Such samples often require special study and treatment.

4) Samples supersaturated with DO—Samples containing more than 9 mg DO/L at 20°C may be encountered in cold waters or in water where photosynthesis occurs. To prevent loss of oxygen during incubation of such samples, reduce DO to saturation at 20°C by bringing sample to about 20°C in partially filled bottle while agitating by vigorous shaking or by aerating with clean, filtered compressed air.

5) Sample temperature adjustment—Bring samples to 20 ± 1 °C before making dilutions. 6) Nitrification inhibition—If nitrification inhibition is desired add 3 mg 2-chloro-6-(trichloro methyl) pyridine (TCMP) to each 300-mL bottle before capping or add sufficient amounts to the dilution water to make a final concentration of 10 mg/L.

Samples that may require nitrification inhibition include, but are not limited to, biologically treated effluents, samples seeded with biologically treated effluents, and river waters. Note the use of nitrogen inhibition in reporting results.

f. Dilution technique: Make several dilutions of sample that will result in a residual DO of at least 1 mg/L and a DO uptake of at least 2 mg/L after a 5-d incubation. Five dilutions are recommended unless experience with a particular sample shows that use of a smaller number of dilutions produces at least two bottles giving acceptable minimum DO depletion and residual limits. A more rapid analysis, such as COD, may be correlated approximately with BOD and serve as a guide in selecting dilutions. In the absence of prior knowledge, use the following dilutions: 0.0 to 1.0% for strong industrial wastes, 1 to 5% for raw and settled wastewater, 5 to 25% for biologically treated effluent, and 25 to 100% for polluted river waters.

Prepare dilutions either in graduated cylinders or volumetric glassware, and then transfer to BOD bottles or prepare directly in BOD bottles. Either dilution method can be combined with any DO

measurement technique. The number of bottles to be prepared for each dilution depends on the DO technique and the number of replicates desired.

When using graduated cylinders or volumetric flasks to prepare dilutions, and when seeding is necessary, add seed either directly to dilution water or to individual cylinders or flasks before dilution. Seeding of individual cylinders or flasks avoids a declining ratio of seed to sample as increasing dilutions are made. When dilutions are prepared directly in BOD bottles and when seeding is necessary, add seed directly to dilution water or directly to the BOD bottles. When a bottle contains more than 67% of the sample after dilution, nutrients may be limited in the diluted sample and subsequently reduce biological activity. In such samples, add the nutrient, mineral, and buffer solutions directly to individual BOD bottles at a rate of 1 mL/L (0.33 mL/300-mL bottle) or use commercially prepared solutions designed to dose the appropriate bottle size.

1) Dilutions prepared in graduated cylinders or volumetric flasks—If the azide modification of the titrimetric iodometric method (Section 4500-O.C) is used, carefully siphon dilution water, seeded, if necessary, into a 1- to 2-L-capacity flask or cylinder. Fill half full without entraining air. Add desired quantity of carefully mixed sample and dilute to appropriate level with dilution water. Mix well with a plunger-type mixing rod; avoid entraining air. Siphon mixed dilution into two BOD bottles. Determine initial DO on one of these bottles. Stopper the second bottle tightly, water-seal, and incubate for 5 d at 20°C. If the membrane electrode method is used for DO measurement, siphon dilution mixture into one BOD bottle. Determine initial DO on this bottle and replace any displaced contents with sample dilution to fill the bottle. Stopper tightly, water-seal, and incubate for 5 d at 20° C.

2) Dilutions prepared directly in BOD bottles—Using a wide-tip volumetric pipet, add the desired sample volume to individual BOD bottles of known capacity. Add appropriate amounts of seed material either to the individual BOD bottles or to the dilution water. Fill bottles with enough dilution water, seeded if necessary, so that insertion of stopper will displace all air, leaving no bubbles. For dilutions greater than 1:100 make a primary dilution in a graduated cylinder before making final dilution in the bottle. When using titrimetric iodometric methods for DO measurement, prepare two bottles at each dilution. Determine initial DO on one bottle. Stopper second bottle tightly, water-seal, and incubate for 5 d at 20°C. If the membrane electrode method is used for DO measurement, prepare only one BOD bottle for each dilution. Determine initial DO

on this bottle and replace any displaced contents with dilution water to fill the bottle. Stopper tightly, water-seal, and incubate for 5 d at 20°C. Rinse DO electrode between determinations to prevent cross-contamination of samples.

Use the azide modification of the iodometric method or the membrane electrode method to determine initial DO on all sample dilutions, dilution water blanks, and where appropriate, seed controls. If the membrane electrode method is used, the azide modification of the iodometric method is recommended for calibrating the DO probe.

g. Determination of initial DO: If the sample contains materials that react rapidly with DO, determine initial DO immediately after filling BOD bottle with diluted sample. If rapid initial DO uptake is insignificant, the time period between preparing dilution and measuring initial DO is not critical but should not exceed 30 min.

h. Dilution water blank: Use a dilution water blank as a rough check on quality of unseeded dilution water and cleanliness of incubation bottles. Together with each batch of samples incubate a bottle of unseeded dilution water. Determine initial and final DO. The DO uptake should not be more than 0.2 mg/L and preferably not more than 0.1 mg/L Discard all dilution water having a DO uptake greater than 0.2 mg/L and either eliminate source of contamination or select an alternate dilution water source.

i. Incubation: Incubate at $20^{\circ}\text{C} \pm 1^{\circ}\text{C}$ BOD bottles containing desired dilutions, seed controls, dilution water blanks, and glucose-glutamic acid checks. Water-seal bottles.

j. Determination of final DO: After 5 d incubation determine DO in sample dilutions, blanks.

5. Calculation

1) For each test bottle meeting the 2.0-mg/L minimum DO depletion and the 1.0-mg/L residual DO, calculate BOD₅ as follows:

$$
BOD_5, mg/L = \frac{D_1 - D_2 - S \times V_S}{P}
$$

where:

 D_1 = DO of diluted sample immediately after preparation, mg/L,
$D_2 = DO$ of diluted sample after 5 d incubation at 20 \degree C, mg/L,

S = oxygen uptake of seed, \triangle DO/mL seed suspension added per bottle (S = 0 if samples are not seeded),

Vs= volume of seed in the respective test bottle, mL, and

 $P =$ decimal volumetric fraction of sample used; $1/P =$ dilution factor.

2) If DO depletion is less than 2.0 mg/L and sample concentration is 100% (no dilution except for seed, nutrient, mineral, and buffer solutions), actual seed-corrected, DO depletion may be reported as the BOD even if it is less than 2.0 mg/L.

3) When all dilutions result in a residual $DO \leq 1.0$, select the bottle having the lowest DO concentration (greatest dilution) and report:

$$
BOD, mg/L > \frac{D_1 - D_2 - S \times V_S}{P}
$$

In the above calculations, do not make corrections for DO uptake by the dilution water blank during incubation. This correction is unnecessary if dilution water meets the blank criteria. If the dilution water does not meet these criteria, proper corrections are difficult; do not record results or, as a minimum, mark them as not meeting quality control criteria [65]**.**