# Performance Evaluation of Ultra-filtration (UF) as a Clean Technology for Reclamation and Reuse of Textile Wastewater



Submitted by

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# **APPROVAL SHEET**

It is certified that the contents and form of thesis entitled

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# Abbreviations

BOD <sub>5</sub>	Biochemical Oxygen Demand
CAS	Conventional Activated Sludge
CER	Certified Emission Reduction
COD	Chemical Oxygen Demand
СТР	Conventional Treatment Plant
GDP	Gross Domestic Product
LiP	Lignin Peroxidase
LME	Lignin Modifying Enzymes
MAF	Million Acre Foot
MBR	Membrane Bioreactor
MMF	Multimedia Filtration
MnP	Manganese Peroxisade
MWCO	Molecular Weight Cut-off
NF	Nanofiltration
PVA	Poly Vinyl Alcohol
RO	Reverse Osmosis
SVI	Sludge Volume Index
TMP	Trans membrane Pressure
TN	Total Nitrogen
TP	Total Phosphorous
TS	Total Solids
TSS	Total Suspended Solids
UASB	Upflow Anaerobic Sludge Blanket
UF	Ultrafiltration
UNDP	United Nations Development Programme
UNICEF	United Nations Children's Fund
UV	Ultra Violet
VSS	Volatile Suspended Solids
WAS	Waste Activated Sludge
WHO	World Health Organization
WRF	White-Rot Fungi
WWTP	Wastewater Treatment Plant
ZDHC	Zero Discharge of Hazardous Chemicals

# ABSTRACT

The textile industry is one of the most important industries in the world, providing jobs for people contributing significantly to the economies of agrarian countries like Pakistan. During the manufacturing process, the textile industry uses a variety of chemicals as well as a huge amount of water. To make one kilogramme of textile, around 200 litres of water are utilised. Water is mostly utilised to apply chemicals to the fibres and to rinse the finished products. The wastewater generated during this process contains a high concentration of colors and pollutants that might affect the environment and human health. The textile industry's effluent water goes through a number of physio-chemical treatment processes. To keep the environmental balance, wastewater must be properly treated before being discharged. This study looked into and examined the levels of pollution in real textile wastewater and investigated the performance evaluation of Ultra-filtration (UF) for reclamation and reuse of textile wastewater. pH, Total Suspended Solid (TSS), Total Dissolved Solid (TDS), Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD<sub>5</sub>), and Color were used to characterise the samples. Results showed that desizing was the most polluting wet process of the textile industry having high values of COD, BOD, TSS and TDS. However, Printing processes were the major contributors for the color in the wastewater. Experiments showed that Ultrafiltration eliminated more than 95% of TSS, as well as 23% TDS, 46% COD, and just 19% color.

# **Chapter 1**

# INTRODUCTION

# 1.1 Background

In Pakistan, the per capita water availability has been reduced to less than 1000 m<sup>3</sup> per capita per day, making it a water scarce country (UNDP, 2018). Excessive water consumption due to modern lifestyles has led to an imbalance between the demand and supply of fresh water globally. The situation in a country like Pakistan is worsening by the fact that the sources of fresh water available to the population are contaminated. One of the major reasons is the toxic chemicals present in industrial effluents, and the illegal dumping of hazardous solid waste into water streams. According to WHO report, 16 million people in Pakistan suffer from water borne diseases and around 3 million are consuming water from unsafe water sources (WHO, 2019). With the annual population increase of 3.2% and inadequate reservoirs to store water, Pakistan is expected to face a water shortage of 33 Million Acre Foot (MAF) by the year 2025 according to a UN report (UNICEF, 2013).

In Pakistan, the textile sector has an overwhelming impact on the economy, being the second largest employment sector and contributing 60% to the country's exports, responsible for 8.5% to the Gross Domestic Product (GDP) of Pakistan (Textile Industry Division, Pakistan). The textile industry is known for its high-water consumption. As a result, the reuse of textile effluent has become a significant challenge. Wet finishing processes can consume up to 200 litres of water per kilogramme of fibre, making wastewater the largest waste in this sector by volume (Chidambaram et al., 2015). Taking these factors into account, this research is aimed at the treatment and recycling of textile effluent.

As per latest environmental monitoring standards including Zero Discharge of Hazardous Chemicals (ZDHC) discharging untreated wastewater may lead to several basic health and environmental risks, therefore to reduce the impacts, affordable treatment technologies shall be established for such waste streams (Metcalf & Eddy, 2003). In developing countries, the centralized treatment option is expensive and not feasible in large urban areas due to complexity of sewerage network, whereas many houses, cluster of houses, and small communities even lack sewer systems (Crites & Tchobanoglous, 1998). In this context, it is

preferred to adopt on-site treatment options based upon the situation, locality and environment (Luostarinen & Rintala, 2005).

Several authors have conducted research on the use of membrane technology to treat textile effluents. These experiments suggest the suitability of permeate to be reused after detailed evaluations of the treated effluents. However, as per our knowledge, no one has conducted a comprehensive analysis of permeate reuse in fabric dyeing, at laboratory as well as the industrial scale.

# **1.2 Problem statement**

For a variety of factors, the textile industry is being compelled to consider water management. The primary reasons for this are increased competition for clean water due to falling water tables, less reserves of clean water, and increased demands from both industrial and residential development, both of which result in higher prices for this natural resource. In most situations, water and effluent costs will account for up to 5% of total production cost.

The most often used wastewater treatment processes are biological or physico-chemical in nature. These techniques can satisfy regulatory criteria, but they do not meet the criteria to reuse treated water in the textile wet processes. Membrane technologies like Ultrafiltration (UF) and Nano filtration (NF) can be used as a post treatment of biologically or physico-chemically treated effluent for the reclamation of textile wastewater. UF process will be our choice in this study as it has many advantages over NF process. Not only UF has high pure water flux but also does not require a high operation pressure.

Ultrafiltration (UF) is a membrane separation procedure that is widely used to isolate macromolecule and colloid from solution and is usually retained in thousands of Daltons by molecular weight. This research aims to propose the optimum operating conditions of UF process on reclamation of real textile wastewater. In brief, the thesis fills some significant gaps in the research on textile wastewater reclamation and provides profound evidence that will serve as a basis for future studies in this field.

## **1.3** Objectives of the study

The study is conducted to assess the Ultrafiltration technology as an option for water reclamation from real textile wastewater. There have been relatively few findings on the reclamation of textile wastewater, the bulk of which were performed on a lab sized reactor using synthetic wastewater. In accordance with this, following objectives were formulated for this research:

- To install and operate an Ultrafiltration plant at a private textile industry
- To analyse the characteristics of the wet processing effluents and the industry's wastewater
- To study the performance evaluation of Ultrafiltration setup for polishing the wastewater treatment plant's effluent

# **1.4** Scope of the study

The study is based on a full-scale operational Wastewater Treatment Plant (WWTP) coupled with pilot-scale Ultrafiltration setup in a textile industry in Rawalpindi, Pakistan. Following considerations make the project significant and novel:

- The study is based on pilot scale study of Ultrafiltration plant for wastewater recovery.
- In this study, all the analysis was done using real textile wastewater.

## Zero-discharge system

The system promotes a zero-discharge system. It encourages the reclamation of the wastewater from industrial applications to be reused for processing of the fabric. The reject of the UF setup may be utilized for the non-potable applications in the industry.

# Easy up-scaling potential

The system can be easily upgraded to enhance the reclamation and reuse capacity of the industry by installing multistage filters.

## **Experimentation with Textile wastewater**

A new aspect of the study is the real textile wastewater recovery using Ultrafiltration setup. Limited research has been conducted on this aspect.

# **Chapter 2**

# **Literature Review**

Water is valuable and principle need in every aspect of life. Approx. 71% of the world is covered with water and out of it, 96.5% is held by the sea, which is saline and cannot be used for drinking purposes. There are approximately 0.9 billion people who lack the access to safe drinking water which is expected to increase to more than 1.3 billion by 2025 (Gikas & Tchobanoglous, 2009).

Every third person on earth is affected by water scarcity. According to World Health Organization (WHO) 60% people of earth will be affected by water scarcity under these conditions by 2025. Gap between demand and supply is also increasing due to urbanization and industrialization (Hulton & Organization, 2012).

# 2.1 Industrial wastewater treatment technologies

The term "conventional wastewater treatment" refers to biological, chemical, and physical processes that separate solids, toxins, and organic matter from wastewater. The methods for eliminating pollutants from drainage are biodegradation, sorption of excess sludge, and volatilization. The composition of the molecules, their physicochemical properties, and the ability of microorganisms to degrade certain molecules all play a role in biodegradability. Sorption is a method of removing hydrophobic materials; accumulate onto primary and secondary sludge (Rogers, 1996). In terms of income generation, biological treatment is certainly the right option for wastewater treatment from carbon credits otherwise known as certified emission reduction credits. The anaerobic treatment also produces methane gas, which can be used as renewable green energy. Almost all wastewaters with BOD to COD ratio  $\geq 0.5$  and having biodegradable components can be effectively treated biologically (Metcalf & Eddy, 2003). It also has a lower treatment price than other approaches of wastewater treatment and produces no secondary contamination. (Sponza & Uluköy, 2006).

Both the techniques, aerobic and anaerobic, will be used. Aerobic treatment includes microorganisms that use oxygen (dissolved or free) to biodegrade carbon-based wastes into new cells and CO2 while in anaerobic treatment a three-step process (hydrolysis, acidogenesis, and methanogenesis) takes place in the absence of oxygen to degrade organic matter into

methane, carbon dioxide and water. Aerobic biological processes are used to achieve high level of treatment effectiveness for treating the organic wastewaters, while in anaerobic treatment, much progress has been made on the principle of nutrient recovery while accomplishing the primary aim of controlling the pollution, as stated by (Seghezzo et al., 1998).

## 2.2 Industrial wastewater treatment in developing and developed countries

Globally, 2.6 billion people have no access to proper sanitation facilities from which 2 billion people belong to developing countries. In other words, approximately 70% of population in developing countries lack the facilities of water, sanitation, and personal hygiene (Tebbutt, 2013). More than 2.4 million people could be saved by proper sanitation and hygiene (Pruss-Ustun & Organization, 2008). In developing countries, rural schemes are not as feasible due to limited resources.

Developing countries do not have proper facilities for the wastewater storage and treatment facilities, semi-urban and agricultural regions in particular. The centralized systems are complex and costly to build and manage (Al-Shayah & Mahmoud, 2008). International organizations are taking interest as a global issue to improve sanitation and hygiene schemes. Most of the developing countries lie in the regions having warm climate. Even in Pakistan, high temperatures persist 8 to 9 months of a year. Therefore, anaerobic technologies are less expensive and highly effective in warm climate (Foresti, 2002).

Pakistan is a developing country and ranked 80<sup>th</sup> out of 122<sup>th</sup> regarding drinking water quality. Surface and groundwater are badly contaminated with toxic metals, coliform and pesticide due to improper sanitation and management. More than 70% people are forced to live without proper sanitation (Ahmed, 2013)

# 2.3 Textile Wastewater treatment in Pakistan

Currently, Pakistan is among the water stressed countries, with water capacity of only 1200 m3/capita/year, and is on the verge of water scarcity (1000 m<sup>3</sup>/capita/year). With a better way of life and a growing population, the demand for fresh water has also increased significantly. Politically, the construction of new water reservoirs is unfavourable. As a result, the only viable option is wastewater treatment and reusing it for domestic, agricultural, commercial and non-potable purposes. Furthermore, as land prices are increasing in Pakistan's heavily populated cities, using traditional wastewater treatment technology is not feasible, such as the

conventional activated sludge process, which needs more space and creates non-reusable effluent.

The membrane-based technologies are the new advancements in the field of wastewater treatment and they give a plausible solution to the wastewater reuse challenge. These membrane-based technologies have been used to generate clean water over the last two decades, including the treatment and recycling of wastewater by membrane bioreactors (MBR) (Williams & Pirbazari, 2007). MBR has many advantages, including a small area footprint and higher-level decomposition of organics in the wastewater, resulting in good quality effluent. As a result, reusing the effluent treated by MBR is technically as well as commercially viable.

MBR is a proven technology to recover wastewater, but the high energy demand for operating the membranes costs a high operating expense which limits its application. The high suction pressure also causes the deposition of contaminants on the membrane surface as well as inside the pores, which causes a decline in the continuous flux and deterioration of the membrane due to repeated chemical cleaning of the membrane. As a result, extensive commercialization of membrane-based technologies has been stifled. Prospective wastewater treatment technologies must overcome these constraints in order for membranes to operate sustainably.

## 2.4 General properties of textile industry

Textile sector include operations like sizing, scouring, desizing, bleaching, weight production, mercerizing, rinsing and dyeing, printing and finishing. Textile dyeing, for example, uses water more than 100 L/kg of fabric produced, making it as one of the most water-intensive manufacturing industries. The above-mentioned methods are described comprehensively:

**Sizing** is the process of adding large amount of Poly Vinyl Alcohol (PVA) to cotton or plastic threads to improve tensile strength and smoothness. The thickening agent is reusable.

**Scouring** impurities in synthetic fibres (catalysts etc.) or in natural fibres (grease, wax etc.) are removed using caustic, detergent etc.

**Desizing** In this process Poly Vinyl Alcohol is extracted using boiling water, a mild oxidising agent or detergent, to allow for further wet processing.

**Bleaching** improves the paleness of cotton by removing its natural color and residual impurities; the amount of bleaching needed is defined by the required whiteness and absorbency. Cotton can be bleached with an oxidising agent such as diluted hydrogen peroxide

or diluted sodium hypochlorite. Lower degrees of bleaching are appropriate if the cloth is to be dyed a darker shade. White bed sheets and medical applications, on the other hand, require the highest levels of whiteness and absorbency. Sodium hypochlorite, hydrogen peroxide, and sodium perborate are examples of bleaching agents, and so are optical brighteners. Batch bleaching is achieved in dyers where the cloth is tacked for a fixed amount of time to allow the chemical to work until the goods are removed from the bottom of the box.

Weight production In this process polyester fibre is processed with caustic after being removed by washing using cold and hot water, and 10-20% of the weight of the fibre is removed.

**Mercerizing** Cotton is processed with condensed caustic to improve curling, minimise shrinkage, and improve dye attachment, in which the additional chemical is extracted with a warm water/detergent wash.

**Rinsing and Dyeing** to extract unfixed pigment and auxiliaries, a hot water/detergent rinse is used in which various dyes and its auxiliaries, for example, electrolytes, dispersing agents, smoothing agents, surfactants, and so on, are used.

**Printing** Direct printing entails printing dyes/pigments directly onto the fabric's surface. Discharge printing is the process of extracting pigment from individual areas of an already colored cloth using suitable chemicals.

**Finishing** In finishing, specific chemicals are used to provide additional attributes to the fabric including antibacterial properties, water and stain resistance, sheen. This also reduces the volume of water used and the resulting runoff. In Figure 1, typical flow sheet can be seen:



Figure 1. Typical flow sheet of dyeing and finishing processes (Lu et al., 2010).

Since water is used as the primary medium for applying dyes and finishing chemicals, as well as for removing impurities, the textile manufacturing industry is considered a water-intensive industry. Therefore, the amount of water released and the hazardous load it carries is the most serious environmental problem. To illustrate,  $20 - 350 \text{ m}^3$  of water is consumed per tonne of fabric produced, with the wide range reflecting the number of processes and process sequences involved (Arslan-Alaton et al., 2012). A large amount of chemicals (dyes, auxiliaries) is consumed during cloth dyeing processes.

### 2.5 Characterization of textile wastewater

Wet manufacturing processes are the key sources of water contamination caused by textiles. The following processes are included: sizing/desizing, scouring, bleaching, mercerizing, dyeing, printing, and finishing. In wet processing, 1kg of fabric typically requires 120L of water to complete the process. So, a vast volume of wastewater is generated daily by various textile mills. The effluent from the textile industry has a high concentration of dissolved organic matter and inorganic compounds, as well as a high pH and a low BOD/COD ratio. Heavy metals, sulphide elements, fats, oils, and fibres are also present. When compared to food industries, the biodegradability ratios for textile and tannery industries are low due to the high COD content and various other contaminants. As a result the traces of non-biodegradable dyes usually remain in the processing effluent. When dyed with reactive dyes, the average rate of dye-fixation ranges within 60 to 80%. Color particles that have not been fixed are washed out of the textile, polluting the wastewater. (Lu et al., 2010). The biodegradability of dyes and color removal are also extensively studied and explored in the literature (Pearce et al., 2003). Organic matter accounts for the majority of the emission load in textile wastewater, implying that

biological processes should be used to treat it. However, recalcitrant organic compounds and residual color often obstruct the implementation of efficient and long-term water recycling strategies in this industry. Advanced treatment technologies are needed, particularly if treated wastewater is reused. The major objectives are effective organic material reduction and complete decolorization. The water quality criteria for all processes in the textiles are tabulated in Table 1.

Parameters	Scouring	Manufacturing	Finishing (Wool)	Finishing (Woven)	Finishing (Knitted Fabric)	Finishing (Carpet)	Dyeing
BOD	6,000	300	350	650	350	300	250
TSS	8,000	130	200	300	300	120	75
COD	30,000	1,040	1,000	1,200	1,000	1,000	800
Color	2,000	1,000	-	325	400	600	600
Water Usage	12		284	113	83	47	100

 Table 1. Process wise textile wastewater parameters.

Textile effluent is hard to process due to its color and recalcitrant COD. Textile industry discharge standards are getting strict in order to minimise environmental effects. Managers are being forced to update out-dated waste management systems or add new systems where none were previously needed due to stricter regulations. Furthermore, due to rising water prices and the need to preserve natural water resources, reuse of treated effluent will become increasingly important in the future. Advanced treatment solutions for textile wastewaters are usually needed due to poor biodegradability, toxicity, and color issues. The textile industry is motivated to reclaim treated wastewaters by stricter disposal standards while increasing the cost for wastewater processing.

In general, textile wastewater treatment is accomplished through a combination of technologies. Because of the high cost and sludge production, single chemical treatment is not used. On the contrary, biological treatment is unable to treat non-biodegradable organic compounds and color in textile wastewater and thus cannot be used as a stand-alone option.

Combining different technologies is also preferred because it not only treats wastewater but also allows it to be reused for other purposes. The following sections explain both traditional and novel methods for treating textile wastewater.

## 2.6 **Physico-chemical treatment**

#### 2.6.1 Coagulation-flocculation

It is a basic physicochemical treatment technique that is used to decolorize wastewater and minimise overall particulate matter in textile wastewater treatment plants. This process is capable of completely decolorizing wastewater (Papić et al., 2004). The key benefit of this approach is that it decolorizes wastewater by separating dye molecules from effluents rather than incomplete degradation of dyes, which can result in potentially hazardous and unsafe aromatic residues (Golob et al., 2005). The efficacy of this process is determined by the raw wastewater properties including the solution's pH and temperature, type, strength and quantity of coagulants, and mixing time.

#### 2.6.2 Adsorption

It is a widely used physico-chemical wastewater treatment process, in which the wastewater is combined with porous substance, like activated carbon or clay, or the wastewater is allowed to flow through a granular filter medium. This system adsorbs and removes pollutant from wastewater on the surface of a porous material or filter. Activated charcoal, silicon polymers, and kaolin are widely known adsorbents. Dyes are selectively absorbed by various adsorbents. So far, activated carbon has shown to be the most effective dye adsorbent in wastewater. In series adsorption reactors, 91.15% of COD can be reduced, allowing the wastewater to meet textile effluent requirements and can be used again as processing water. Since activated carbon can adsorb colorants, it can easily extract water-soluble dyes including reactive, basic, and azo dyes from the wastewater, however it cannot adsorb suspended particles or insoluble dyes. Furthermore, because the cost of regenerating activated charcoal is extremely high, activated carbon is typically used to treat wastewater streams with low pollution loads.

#### 2.7 **Biological treatment**

## 2.7.1 Aerobic treatment

Aerobic biological treatment is one of the most traditional methods of treatment used for dyeing effluent which uses activated sludge (Joshi et al., 2004). Aerobic treatment usually uses stabilisation basins, aerated ponds, or filters. Microorganisms in aerobic treatment use soluble oxygen to create more biomass when turning waste into carbon dioxide and water. Organic matter is oxidised, and the energy generated is utilised to generate new living cells. After settling to the bottom of the reactor, flocs are separated. (Naresh et al., 2013). According to the findings of different researchers, dyestuffs do not biodegrade significantly under aerobic conditions. However, research into aerobic microorganisms having the ability of destroying dyes and similar compounds is ongoing. Bacteria and fungi are the two the most used microorganism species (Kumar & Rai, 2005).

#### 2.7.2 Treatment by Fungi

White-rot fungi (WRF) are the most powerful microorganisms for destroying artificial dyes commonly available. WRF is a type of microorganism that produces enzymes that can degrade dyes in aerobic environments. (Chakraborty et al., 2013). WRF lignin modifying enzymes (LME) are specifically degrade not only lignin, but also a variety of dyes. (Chaturvedi, 2019). The white rot fungus Phanerochaete chrysosporium has been extensively researched in recent years due to its capacity to degrade multiple recalcitrant contaminants. It has also been shown that it can decolorize dyes. Under aerobic conditions, Phanerochaete chrysosporium has been shown to decolorize a variety of azo dyes. It has been hypothesized that various extracellular peroxidases or laccases are involved in the dye decolorization process. (Forgacs et al., 2004).

#### 2.7.3 Treatment by bacteria

Decolorization by bacteria is usually quicker than fungal decolorization. (Pandey et al., 2007). Bacterial strains can mineralize different dyes under aerobic conditions (Kumar & Rai, 2005). A few researchers have also used sulphate-reducing bacteria to dissolve dyes, as well as Rhizobium radiobacter for triphenylmethane dye biodegradation (Parshetti & Doong, 2009). Many bacterial strains that can decolorize azo dyes in aerobic conditions have been isolated in recent years. Many bacterial strains have been documented to be dye decolorizers or dye degraders, such as: *Bacillus megaterium, Alcaligenes faecalis etc.* 

#### 2.7.4 Anaerobic treatment

According to the literature, the ability of anaerobic microorganisms to decolorize dyes has been well known and identified (Kumar & Rai, 2005). Anaerobic removal of azo dyes using sludge can be an efficient and cost-effective treatment method for removing color from clothing wastewater. The research on anaerobic decolorization of azo dyes began in the early 1970s. Several laboratories have reported using intestinal anaerobic bacteria to decolorize azo dyes. Anaerobic bioremediation can decolorize azo and other water-soluble dyes. (Anjaneyulu et al., 2005). Anaerobic bioremediation of azo dyes by breaking them down into corresponding amines for decolorization has received a lot of attention. The removal of the azo bond can be used to accomplish primary oxidation. The azo bond can also be reduced under the reducing conditions found in anaerobic bioreactors. The amines formed by azo dye reduction are colorless, but under anaerobic conditions they resist further degradation. Many anaerobic bacteria (e.g., Clostridium sp.) and facultative anaerobic bacteria (e.g., Streptococcus faecalis) can decolorize different azo dyes under anaerobic conditions by reducing the azo connection. Azo dyes are converted into aromatic amines under anaerobic conditions, which can be toxic and potentially carcinogenic to mammals. As a result, another stage involving aerobic biodegradation of the generated aromatic amines is needed to achieve complete degradation of azo dyes (Li et al., 2012). Aromatic amines are not usually degraded further in anaerobic conditions. Anaerobic treatment must thus be regarded as just the first stage of the full degradation of azo dyes, while the second stage entails aerobic transformation of the produced aromatic amines.

#### 2.7.5 Combined anaerobic and aerobic treatment

Most dyes are usually resistant to aerobic degradation but can be decolorized under anaerobic conditions, according to a common finding that has evolved over the years. Since azo dyes are the precursors of aromatic amines, which are carcinogenic and mutagenic, they are poisonous and hazardous to the environment. Azo dyes are usually reduced by bacteria under anaerobic conditions to colorless poisonous aromatic amines, some of which are readily metabolised under aerobic conditions. The most plausible concept for extracting azo dyes from wastewater is a procedure that incorporates anaerobic and aerobic environments.

Azo dye mineralization necessitates an incorporated or concurrent anaerobic and aerobic phase. Staphylococcus arlettae was also used in parallel micro-aerophilic aerobic reactors for dye biodegradation (Franciscon et al., 2009).

Over time, it has been apparent that dyes are difficult to degrade. There are several biological technologies used to degrade the pigment. However, owing to the complexity of textile effluent, these technologies have not been applied. A single widely applicable end-of-pipe approach for purifying textile wastewater appears to be impractical, while a mixture of suitable techniques is regarded as a theoretically and economically feasible alternative.

#### 2.8 Membrane separation processes

Membrane separation is a filtering technique that separates specific substances in wastewater by using the membrane's micro pores membrane's selective permeability. Membrane separation methods are reliant on membrane pressure and are currently used to treat dyeing effluent. Membrane separation is a relatively modern separation technique that offers high separation performance, ease of operation, and ultra-pure effluent. Nevertheless, due to limitations such as membrane fouling, the need for specialised equipment, high investment, and a variety of other factors, this technology has yet to be widely adopted (Ranganathan et al., 2007).

#### 2.8.1 Reverse osmosis

Reverse Osmosis, yields high-quality permeate, having retention rate higher than 90%. In a homogeneous system, reverse osmosis can decolorize and remove chemical components from dyeing wastewater. Reverse osmosis could extract all minerals, water - soluble dyes, and chemical auxiliaries. It's important to note that the higher the dissolved salt level, the greater the osmotic pressure, which means the separation process, would take more energy. (Pensupa et al., 2017).

#### 2.8.2 Nano-filtration

The colored textile effluents have been treated using Nano-filtration. For the dye effluent treatment, a combination of adsorption and Nano-filtration can be used. The adsorption step comes before the Nano-filtration step because it reduces concentration polarisation during the filtration procedure, increasing the yield of the process. Nano filtration membranes retain high concentrations of divalent ions, significant monovalent ions, water - soluble dyes, and

auxiliaries. High dye and salt levels in dyeing wastewater have been linked to a variety of adverse effects. The pollutants are formed with a specific dye in most cases, and the quantity investigated is negligible. Among the few uses for the treatment of highly condensed and complicated solutions, using Nano-filtration is the treatment for dyeing wastewater. (Pensupa et al., 2017). The accumulation of dissolved solids is a major issue that makes the discharge of the treated wastewater into water bodies complicated. Various academic researchers have worked to create inventions for effective dye effluent treatment that are commercially feasible. In terms of fulfilling environmental regulations for discharging wastewater into water bodies, Nano-filtration treatment has proven itself very effective.

#### 2.8.3 Ultrafiltration

Ultrafiltration, with the pore size ranging between 0.002 to 0.1 microns, allows for the removal of macromolecules and particles, but complete removal of polluting dyes cannot be achieved. As well as in the best-case situation, the treated wastewater's quality prevents it from being used in sensitive applications like garment dyeing. (Rott & Minke, 1999) emphasise that 40% water processed by UF can be reused in the textiles processes defined as "minor" (like rinsing, washing). Ultrafiltration should be used as a pre-treatment for reverse osmosis. (Ghaly et al., 2014).

#### 2.8.4 Micro-filtration

Microfiltration with an aperture between ranges of 0.1 - 1 m is appropriate for the treatment of pigment dye effluent and successive rinsing baths. The dye bath chemicals remain in the bath that is not filtered by micro-filtration. Micro-filtration can also be utilised as a precursor to Nano-filtration or reverse osmosis (Ghaly et al., 2014). Textile effluent contains a high concentration of recalcitrant carbon-based matter as well as inorganic substances. Many factories are now using physicochemical treatment processes.

Advanced treatment technologies are recommended for favourable wastewater treatment and reuse outcomes due to the recalcitrant quality of textile wastewater. In the literature review, hetero processes, also known as hybrid/combined processes were carried out. Few examples are given in the context of textile effluent, where the goal was to research multiple membrane-based methods to analyse their performance for the reuse and treatment of textile wastewater (Marcucci et al., 2001). Badani et al. (2005) demonstrated that UF process removed 97% of COD after the biological treatment side stream. Similarly, when Brik et al. (2006) used the UF

process, 60 - 95% of COD removal was observed. For color removal, they suggested the Nanofiltration process. Lu et al. (2010) reported a removal rate of 93, 94, and 93% for COD, color, and turbidity, respectively. Srivastava et al. (2011) used PVDF membrane for the pre-treatment because of its significant features such as good pH performance, hydrophobic adsorption ability for dyestuff, and perfect fouling resistance. The COD rate is 97% and the color removal rate is 85%. The RO and NF were compared by (Hai et al., 2011). Kurt et al. (2012) used a pilot scale system to treat dye wash effluent by decolorization and COD removal. NF and RO tests were also carried out. Lin et al. (2017) developed a joint reactor, which was a blend of coagulation and ultrafiltration, allowing for complete color removal and 88% COD removal efficiency. In this case, fouled membranes were more hydrophobic than new membranes. These studies are primarily concerned with membrane filtration rather than membrane bio-reactors.

## 2.9 Membrane treatment

Membrane is a selective barrier, allowing certain components to pass through it, demonstrating its selective permeable nature. A typical anatomy of a membrane can be seen in the Figure 2.



Figure 2. Membrane Structure

The level of selectivity is determined by the membrane's openings and material. Membranes were first commercialised in the early 1990s and have since been used in specialised applications in water and wastewater treatment. The use of membranes allows for the best

wastewater recycling and reuse solutions. In the wastewater treatment industry, annual growth rates of up to 15% have been recorded (Smith et al., 2007). With time-sensitive effluent discharges and water-quality legislation and effective wastewater treatment are primary factors for the improvement of this technology. Figure 3 shows a general schematic of the membrane.



Figure 3. Schematic of Membrane

#### 2.9.1 Membrane processes

Domestic wastewater reclamation is recognised as a capable method of meeting rising demands for water possessions. Domestic wastewater contaminated by pollutants, on the other hand, may pose health risks, be a source of pathogenic infection, and have poisonous effects. As a result, municipal wastewater recovery necessitates advanced treatment techniques in order to obtain higher-quality effluents, as well as financial viability considerations (Ivanovic & Leiknes, 2011).

To meet environmental regulations, the use of membrane has increased in a variety of wastewater treatment processes. Environmental awareness, laws, efficiency, and a wide range of treatment technologies have made water reuse more feasible (Hoinkis et al., 2012).

The membrane process separates the majority of the suspended and colloidal material. It also improves and concentrates it as a product. This product is recyclable and reusable. As a result, the pollutant emitted into the environment is reduced, resulting in a new and cleaner technology (Eliceche et al., 2002).

#### 2.9.2 Membrane operational modes

Depending on the degree of contamination and the nature of use, the membrane can be operated in a variety of configurations and filtration modes. Dead-end filtration, commonly known as direct filtration, is characterised by perpendicular flow direction to the membrane. The membrane filters the entire feed stream. The particles retained cause cake layer formation on the membrane's surface. The deposited layer of these particles may damage and clog the membrane's pores. Fouling develops over time due to solid accumulation, which can be reduced by backwashing. This flow is preferred when the feed contains few foulants. Figure 4 demonstrates dead-end filtration below:



Figure 4. Dead-end filtration mode

The flow is tangential across the surface of the membrane in cross-flow filtration. The permeate is a fraction of the influent that passes through the membrane while the respite is rejected. It prevents cake formation by scouring the membrane surface with the flow until the adhesive forces that hold the cake layer to the membrane are balanced. When this equilibrium is reached, a steady state is achieved, resulting in increased permeate flow. Figure 5 shows cross flow filtration below:



Figure 5. Cross flow filtration mode

### 2.9.3 Membrane modules

Configuration of a membrane unit is very critical as it influences the unit's ability to pass all of the flow coming to the membrane. Membrane fouling is connected to the configuration of the membrane module, and a variety of membrane geometries regulate micro or ultra-filtration. There are currently six MBR configurations in use:

- Plate and frame/ flat sheet (FS)
- Hollow fibre (HF)
- Multi tubular (MT)
- Capillary tube (CT)
- Pleated filter cartridge (FC)
- Spiral wound (SW)

The most suitable are FS, HF, and MT because they allow for turbulence and regular cleaning (Santos et al., 2011).

#### Hollow fibre membrane (HFM)

A hollow fibre scheme consists of several thin fibres arranged in a sealed unit known as a cartridge. This system's influent wastewater can come from either inside or outside the fibre. At the cartridge's opening end, effluent is captured from the inside or outside of the fibre. Hollow fibre membranes have a vast surface area and allow for more filtering in a small amount. (Bassyouni et al., 2019).

The category of membranes under investigation was hollow fibre membranes, which have been successfully used in industries. The hollow fibre geometry allows for a large membrane surface area to be constrained in a small unit. This way, a large volume can be filtered while taking up little space and using less energy.

There are two distinct modes of filtration in hollow fibre membrane.

(A) Outside-in filtration

The ability to handle extremely high concentrations of suspended solids is the main advantage of outside-in filtration. Outside-in modes are used by all commercially available hollow fibre modules. These modules are submerged directly in mixed liquor where influent water strikes the shell side of the membrane and effluent is collected by applying a vacuum. Air scouring is

provided in the shell side either continuously or intermittently to prevent solids from accumulating on the membrane's surface.

A number of outside-in modules used to treat water with low suspended solids (50 mg/L). Huge amounts of solids are stored in the empty spaces between the fibres, reducing the flux through the membrane at the maximum allowable pressure. Back flushing is done on an as-needed basis to remove accumulated solids when the transmembrane pressure (TMP) exceeds the limit. The treated effluent is obtained through vacuum, in which hollow fibres are horizontally mounted without the use of a pressure vessel. The hollow fibres in most other modules for low suspended solids are enclosed in a pressure vessel and run under positive pressure in direct filtration.

#### (B) Inside-out filtration

These hollow fibres are adequate for maintaining consistent hydrodynamics in the lumen, but the turbulence required to alleviate cake layer development is nearly impossible to achieve due to the small inside diameter and low water velocity. Furthermore, because the fibre lumen can be clogged by particles in the influent wastewater, this type of membrane can only filter water with low suspended solids, such as surface water.

Both modes of operation are depicted in Figure 6.



Figure 6. Modes of filtration in Hollow-Fibre membranes

#### 2.9.4 Pore size and pore size distribution

The size of pore is another important factor to consider when analysing a fouling pattern because it has a direct impact on filtration. By reducing the pore size, the filtration value increases significantly (Koo et al., 2012).

## 2.9.5 Membrane filtration

MBR filters can be filtered at several levels, including microfiltration, ultrafiltration, nanofiltration, and reverse osmosis. Micro filtration pores range in size from 0.1 to 1 micron. The pore size of ultrafiltration is reduced to 0.001 to 0.1 micron. Nano-filtration removes salts and sugars from water, making it suitable for water softening. Reverse osmosis can remove almost everything from water, resulting in ultrapure water. The operating pressure for micro-filtration and ultrafiltration membranes rises, as does the operational cost (Naveed et al., 2006). Table 2 shows the details of membranes:

Membrane Filtration	Size (µm)	Removal
Туре		
Microfiltration	0.1	Bacteria and suspended particles.
Ultrafiltration	0.01	Macro molecules and viruses.
Nano Filtration	0.001	Dissolved contaminants and water hardness.
Reverse Osmosis	0.0001	All dissolved contaminants

 Table 2. Membrane Filtration and Pathogen Removal.

# 2.9.6 Membrane operation

The membrane separation process divides a feed stream into two permeate streams known as permeate and retentate. A membrane is a fence that separates two stages and binds the transport of a variety of chemical materials. The flow separated by the membrane is known as the permeate stream, whereas the flow retained by the membrane is known as the retentate stream. Depending on the desire, any of the above streams can be used for membrane separation (Mulder & Mulder, 1996).

Colloids are tiny particles with a size range of 1–1000 nm. These tiny collides have a high propensity to clog membranes in pressure-driven membrane systems, resulting in significant loss of water permeability and, in some cases, deterioration of product water quality (Tang et al., 2011). The adherence of particulate, colloidal, or soluble substances inside the openings or on the surface of the membrane causes fouling (Böhm et al., 2012).

Membranes must be cleaned on a regular basis, either physically or chemically, in order to keep the process running smoothly. According to Metzger et al. (2007), frequent chemical cleaning is required to maintain long-term operations, which may increase operational costs and ultimately reduce membrane life. Membrane separation is an important technology in desalination and wastewater reclamation. For large-scale wastewater treatment, these technologies have largely replaced traditional separation processes. They are more environmentally friendly and are referred to as a clean technology (Soni et al., 2009).

Because of their versatility, extraction processes using hollow fibre membranes are of particular interest in terms of membrane filtration performance. Membranes are becoming increasingly popular for use in treatment processes due to their strength and pliability.

This chapter discusses about the study location, description of the UF plant, sampling strategy and various physico-chemical tests that were performed to fulfil the objectives of this research.

# **Chapter 3**

# Methodology

# **3.1 Study Location**

A preassembled UF pilot-scale plant was installed at a local textile industry situated in Rawalpindi, Pakistan.

# **3.2 Process Description**

The UF treatment plant was designed, manufactured, and shipped as part of the InoCotton GROW project (Innovative impulses reducing the global cotton-textile industry's water footprint towards the UN Sustainable Development Goals), which was funded by the German Ministry of Education and Research (BMBF). The plant was built by A3 Water Solutions GmbH in Germany, and Figure 7 depicts a pictorial view of the ultrafiltration plant.



Figure 7. UF pilot plant

The UF pilot plant was installed to polish the effluent of the wastewater treatment plant. The UF plant operates on the batch mode. Since the pilot plant could not be connected directly with multimedia filters (MMF) of the treatment plant, a collection tank of capacity 300 L, was provided to store the feed water of the UF plant. From the storage tank, the water is driven

through a coarse filter, installed at the upstream of UF module to avoid frequent backwashing. A feed water pump was installed to inject the water into UF membranes. Because UF membranes operate from the inside out, suspended particles are retained inside the membrane while permeate is collected from the outside. Once treated, the effluent of UF membranes is passed to the RO plant to remove the dissolved solids. The concentrate of the UF is collected through a ball valve and sent back to the wastewater treatment stream. The process flow diagram is depicted in Figure 8.



Figure 8. Process flow diagram

# **3.3**Components of UF plant

## 3.3.1 UF membrane

For this research, a ceramic multi-channel membrane module was used. The membrane is made up of  $Al_2O_3$  material having the area of 0.2 m<sup>2</sup>, length 1000 mm and channel diameter of 3.3 mm. These have proven to be dependable due to their unique material properties, which include high flow capacities and excellent chemical, thermal, and mechanical resistance. The water to be filtered flows through the channels of the membrane carrier during cross-flow filtration with ceramic membranes. All molecules larger than pore diameter are held back. Figure 9 depicts the UF membrane below:



Figure 9. Membrane filtration module

The centrifugal pump transports the water to be filtered from the storage tank, made of stainless steel, through the coarse filter into the UF module. The pump has a maximum capacity of 4.4  $m^3/h$  flow and 2.9 bar pressure. The feed pump is shown in Figure 10.



**Figure 10. Feed Pump** 

## 3.3.2 Sensors

Multiple sensors were installed to monitor and control the flow of water including one level sensor (Liquiphant FTL31) and two flow rate sensors (SM 8000 & SM 6000). The liquid level in the storage tank was controlled by a point level switch. It shuts down the feed pump in case the liquid level falls below the limit value. The feed flow was measured by a magnetic-

inductive flow sensor SM 8000 having a range of  $0.01 - 6 \text{ m}^3/\text{h}$  and the filtrate output was measured by flow sensor SM 6000 having a range of  $-1.8 - 1.8 \text{ m}^3/\text{h}$ . The sensors are shown in the Figure 11.



Figure 11. A) Level sensor; B) Flow sensor (feed); C) Flow sensor (permeate)

# **3.4Operational Cycle**

To establish the baseline, controlled 3-hour runs were performed by using distilled water as a feed stream. The feed water from the MMF was treated in the batch modes at different pressures. Each reading was taken after 30 minutes of operation to obtain the reading from a stabilized system. The pressure of the system was controlled through manually operated valve.

## 3.4.1 Maintenance of UF Plant

The filtration membrane must be cleaned at regular intervals to maintain its performance (backwashing). When the membrane clogs frequently, it is thoroughly cleaned with chemicals in addition to backwashing. Organic and non-organic deposits are removed from the filtration membrane during the cleaning process. The cleaning process varies depending on the characteristics of the liquid to be filtrated and the filtration pressure difference.

#### **3.4.2** Sampling Strategy

For the first objective, seven sampling points were selected to analyze the composition of waste streams of wet processing department of the textile industry. The samples were taken from the flowing points: mercerizing, bleaching, dyeing, desizing, finishing, and reactive and pigment

printing. One sample was collected each month for the duration of five months to analyse the trend of different parameters in the wastewater streams. To fulfil the second objective, four sampling points were chosen. Three were located on the downstream of wastewater treatment plant (WWTP), multimedia filters, UF plant and one was located at the upstream of WWTP. For a period of five months, the sampling frequency was five samples per month from each point. In order to meet the third objective, samples were collected and analysed from the textile industry's full scale UF plant so that the performance of the full scale and pilot scale UF plants could be compared. Each time, a 1L sampling volume was collected.

## **3.5Physico-chemical Analysis**

Following the collection of the samples, the following analytical techniques were used:

#### 3.5.1 Total Suspended Solids

The EPA Method 160.2 (gravimetric method, dried at 105 °C) was used to measure TSS. A total of 20–30 mL of sample was filtered. The filter paper was then dried in a 105 °C oven for 2 hours. The TSS concentration is determined by the difference in the weight of filter paper before and after drying.

#### 3.5.2 TDS

Total Dissolved Solids (TDS) were measured with a TDS metre (Hone Forest) with a measuring range of up to 9990 ppm (parts per million) and a 2% accuracy. Total Solids (TS) can also be calculated by evaporating the sample water at a predetermined temperature. TDS is computed by subtracting TSS from TS.

#### 3.5.3 BOD<sub>5</sub>

The waste sample, or an appropriate dilution, is incubated at 20°C in the dark for 5 days. The decrease in dissolved oxygen concentration during the incubation period is used to calculate the biochemical oxygen demand. Standard Methods for the Examination of Water and Wastewater, 15th Edition, p.83, Method 507.

#### 3.5.4 COD

COD was determined using the closed reflux titrimetric method. In COD vials, 2.5 mL of sample, 1.5 mL of digestion reagent ( $K_2Cr_2O7$ ), and 3.5 mL of sulfuric acid ( $H_2SO_4$ ) reagent were added, capped, and placed in a preheated (150°C) COD digester for 2 hours.

The vials were then allowed to cool to room temperature before being titrated with ferrous ammonium sulphate (FAS) using ferroin as an indicator.

#### 3.5.5 pH

pH was measured by pH meter (Cybesrscan 500)

## 3.5.6 Color

The Platinum-Cobalt Scale (Pt/Co scale or Alpha-Hazen Scale) ASTM D1209-00, Standard Test Method for Color of Clear Liquids (Platinum-Cobalt Scale), ASTM International was used to measure the color. Allen Hazen, a chemist, created the index to assess the level of pollution in wastewater.

#### **3.6Data Analysis**

The results obtained from the chemical analysis were used to determine the removal efficiencies of TSS, TDS, COD, and BOD<sub>5</sub>. The efficiency was calculated using Equation 1.

Removal efficiency (%) = 
$$\frac{Ci-Ce}{Ci} * 100$$
 Eq.1

Where:

Ci = Concentration in the influent (mg/L)

Ce = Concentration in the effluent (mg/L)

Further, following equation was used for the COD calculation:

$$COD \left(\frac{mg}{L}\right) = \frac{\left[(A-B)*1000*8\right]}{Sample \ Volume \ (mL)}$$
Eq.2

Where:

A= Volume of FAS used to titrate the sample (mL)

B= Volume of FAS used to titrate the blank (mL)

# **Chapter 4**

# **Results and Discussion**

## 4.1 Characterization of textile wastewater

The characterization of all the wet processes of the textile industry was done at the immediate downstream of the process including Desizing, Bleaching, Mercerizing, Dyeing, Pigment printing, Reactive printing and finishing to estimate the pollution load of the individual processes at the source. The process streams were tested for the selected six parameters including BOD, COD, TDS, TSS, pH and color over a period of five months to accommodate the high and low production of the textile industry and get the average value of each pollutant in the waste streams. The detailed results are shown in the paragraphs below:

#### 4.1.1 Biochemical Oxygen Demand (BOD)

The presence of unoxidized organic matter causes the wastewater's biochemical oxygen demand (BOD). It denotes the amount of oxygen consumed by microorganisms such as bacteria while decomposing organic matter under aerobic conditions at a specific temperature. The highest BOD was found in the Desizing waste stream with the average BOD value of 20,013 mg/L. The contribution of all other processes was small in comparison to the Desizing process. The average BOD value of the streams of Bleaching, Mercerizing, Dyeing, Pigment printing, Reactive printing and finishing were 458.6, 63, 246.4, 616.4, 560.8, and 437 mg/L respectively. The minimum BOD was of Mercerizing waste stream with the average value of 63 mg/L only. Cloth is treated with starch, gum, and enzymes during the sizing and desizing processes, which eventually end up in wastewater. This is the cause of the high biochemical oxygen demand. The BOD level was found to be 1193.4 mg/L at the Conventional treatment plant (CTP) inlet and 51.4 mg/L at the outlet.



Figure 12. BOD in wastewater samples of different processes

#### 4.1.2 Chemical Oxygen Demand (COD)

Chemical Oxygen Demand (COD) is a second method of estimating how much oxygen would be depleted from a receiving body of water as a result of bacterial action. For the COD test, a strong chemical oxidising agent, potassium dichromate (K2Cr2O7), was used to chemically oxidise the organic material in the wastewater samples. Similarly, to BOD, the highest COD was found in the Desizing waste stream with the average value of 34156 mg/L. The average values of COD in the streams of Bleaching, Mercerizing, Dyeing, Pigment printing, Reactive printing and finishing were 1245.6, 346.2, 583.6, 1418.4, 1541.2 and 996.8 mg/L respectively. The minimum COD was of Mercerizing waste stream with the average value of 346.2 mg/L only. The COD level was found to be 3034.2 mg/L at the Conventional treatment plant (CTP) inlet and 111.8 mg/L at the outlet. In the case of biodegradable organics, the COD is typically 1.3 to 1.5 times the BOD. COD levels are greater than 1.5 times those of BOD, indicating that a significant portion of the organic material in the wastewater is not biodegradable by common microorganisms.



Figure 13. COD in wastewater samples of different processes

#### 4.1.3 Total Suspended Solids (TSS)

Total Suspended Solids are solids in water that can be trapped by a filter. The water sample is filtered through a pre-weighed filter to determine the TSS. The TSS was calculated at the downstream of Desizing, Bleaching, Mercerizing, Dyeing, Pigment printing, Reactive printing and finishing processes and the values were 1540.8, 993.4, 28.8, 87.2, 283.8, 140.6 and 207.4 mg/L respectively. Desizing and Bleaching were found to be the top contributors of TSS in the waste stream. Surfactants, enzymes, acids, alkalis, and additives used in size recipes contribute to the TSS load of desizing effluent. Bleaching removes the natural yellowish colorings of cotton and other fibres, making them whiter. Because of the chemicals used, such as sodium hypochlorite and hydrogen peroxide, its wastewater typically has a high solid content. TSS levels in the generated wastewater are high at the inlet, with an average of 528.6 mg/L and 133.2 mg/L at the outlet. The TSS should be further removed by tertiary methods for making this water fit for reusing in the wet processing.



Figure 14. TSS in wastewater samples of different processes

#### 4.1.4 Total Dissolved Solids (TDS)

Total Dissolved Solids (TDS) are inorganic salts, organic matter, and other dissolved materials in water that are measured. Total solids (TS) are determined by evaporating water at a predetermined temperature. TDS is computed by subtracting TSS from TS. The TDS values were tested at the downstream of Desizing, Bleaching, Mercerizing, Dyeing, Pigment printing, Reactive printing and finishing processes and the values were 12511.3, 4386.4, 802, 3595.2, 13127.6, 11313.6 and 383 respectively. The high TDS value in Pigment and Reactive printing indicates that there is a high content of dissolved solids in the dyeing effluent because the dyeing process requires a significantly high concentration of salts (up to 80 g L-1 NaCl) and sufficient alkali to raise the pH to between 12 and 12.5. Furthermore, the dyes used in the industry are soluble in water, making them difficult to treat using traditional methods. Dye effluents, in general, have a high color content, a high TDS, and a moderate BOD value. The average TDS value at the CTP inlet was 2897 mg/L, and the value at the outlet was 2494 mg/L.



Figure 15. TDS in wastewater samples of different processes

#### 4.1.5 pH

The pH of the solution has been shown to affect both particle and membrane surface electrochemical properties, varying the performance of ultra-filtration membranes (Zhang et al., 2008). Further, the effect of feed pH on process performance is dependent on other operating conditions such as Molecular weight cut-off (MWCO) and Trans membrane pressure (TMP). As a result, below an applied pressure of 2 bars, the influence of feed pH may be considered insignificant. This pattern is related to particle diffusion back to the bulk solution or shear-induced inertial lift (Ogunbiyi & Nick J. Miles, 2008). The lowest pH was observed for the desizing process at the value of 5.6. The pH for Bleaching, Mercerizing, Dyeing, Pigment printing, Reactive printing and finishing processes were also measured and the values were 10.6, 11.9, 8.2, 7.7, 9.7 and 7.7, respectively. All of these processes contributed to the pH level of the combined wastewater which was 11.5 at the inlet and was balanced during the treatment to the average of 8.2 at the CTP outlet. According to Aimar and Meireles (2010), flocculation or particle aggregation may occur at pH 8 due to lower electronic repulsion forces between particles.



Figure 16. pH in wastewater samples of different processes

#### 4.1.6 Color

The color was measured using the Platinum Cobalt Scale. The color values were tested of Desizing, Bleaching, Mercerizing, Dyeing, Pigment printing, Reactive printing and finishing processes and the values were 199.6, 56.6, 8.6, 2151.2, 779, 15182 and 1142.5, respectively. The color value at the CTP inlet was 1078.4 and at the outlet were 181.4. It can be seen that color pollution in the textile industry is caused by the dyeing and printing processes where reactive printing is the major contributor with an average value of 15182. Although ultra-filtration (UF) removes particles and macromolecules, it does not completely remove color. However, UF permeate meet the requirements for allowing wastewater reuse in minor textile industry processes (rinsing, washing) (Allègre et al., 2006).



Figure 17. Color in wastewater samples of different processes

Parameters	Desizing	Bleaching	Mercerizi ng	Dyeing	Printing (Pigment)	Printing (Reactive)	Finishing	CTP Influent	CTP Effluent
BOD <sub>5</sub> (mg/L)	20013.5	458.6	63.0	246.4	616.4	560.6	437.0	1193.2	51.4
	S.D;4371.8	S.D; 200.3	S.D; 35.6	S.D; 141.2	S.D; 489.05	S.D; 234.3	S.D; 194.2	S.D;487.2	S.D; 25.16
COD (mg/L)	34156.0	1245.6	346.2	583.6	1418.4	1541.2	996.8	3034.2	111.8
	S.D; 7220.2	S.D; 610.2	S.D; 196.7	S.D; 244.6	S.D; 877.3	S.D; 791.9	S.D; 314.8	S.D; 911.5	S.D; 15.2
Color (Pt/Co)	199.6	56.6	8.6	2151.2	779.0	15182.1	1142.2	1078.4	181.4
	S.D; 144.0	S.D; 16.2	S.D; 7.0	S.D; 397.5	S.D; 212.3	S.D; 414.2	S.D; 119.9	S.D; 243.6	S.D; 45.2
TSS (mg/L)	1540.8	993.4	28.8	87.6	283.8	140.6	207.4	528.6	133.2
	S.D; 303	S.D; 513	S.D; 8.9	S.D; 46.8	S.D; 156.3	S.D; 45.6	S.D; 50.0	S.D; 178.3	S.D; 47.8
TDS (mg/L)	12511.3	4386.4	802	3595.2	13127.6	11313.6	383.0	2494.0	2897.1
	S.D; 3003.2	S.D; 1362.4	S.D; 138.6	S.D; 317.4	S.D; 3993.4	S.D; 2324.4	S.D; 21.9	S.D; 1290.6	S.D; 168.6
pH	5.6	10.6	11.9	8.2	7.7	9.7	7.7	11.5	8.2
	S.D; 1.0	S.D; 0.8	S.D; 0.5	S.D; 0.6	S.D; 1.0	S.D; 0.6	S.D; 0.5	S.D; 0.7	S.D; 0.4

Table 3.	Characteristics	of textile	wastewater	(Averages	of 5 (	different	sample	es).
				(				~

## 4.2 Characteristics of Ultrafiltration membrane

Initially, the characteristic of membrane was studied; pure water was passed through membrane to know the flux and resistance properties of the UF membrane before operating with wastewater.

The permeate flux was determined to evaluate the membrane fouling. It was determined by measuring the permeate volume collected in a certain period and using the following equation 3:

$$J(L/m2 - h) = V/A * \Delta t$$
 Eq.3

Where J is the permeate flux (L m<sup>-2</sup> h<sup>-1</sup>), A is the effective area of the membrane (m<sup>2</sup>) and V (L) is the collected volume in a time interval  $\Delta t$  (h).

The linear relationship between permeate flux,  $J_w$  and transmembrane pressure is shown in Figure 18. The membrane resistance,  $R_m$  was obtained as functions of  $J_w$ ,  $\Delta P$ ,  $\mu$  (viscosity) by equation 4

$$R_m = \Delta P / (J_w * \mu)$$
 Eq.4

The results showed that permeate flux increases with the increase in transmembrane pressure. The membrane resistance,  $R_m$  obtained here is 2 \* 10<sup>13</sup> m<sup>-1</sup>. And the permeate flux,  $J_w$  of this UF membrane is in the range of 20 – 100 L/m<sup>2</sup>/hr at the operating transmembrane pressure of 0.5 – 2.5 bars.



Figure 18. The relationship between permeate flux and trans membrane pressure for pure water

# 4.3 Polishing of textile wastewater by UF pilot plant

Each parameter was tested against variable pressure to study the effect of pressure on the performance of UF pilot plant. The results are presented in the following sections:

## 4.3.1 COD removal

The COD after MMF reduced from 103 mg/L to 91 achieving an average removal efficiency of 11.65% while the coarse filter decreased the COD by 2.19% only from 91 mg/L to 89.20 mg/L. After this COD was measured at different pressures ranging from 0.5 to 2.5 bar. The COD was reduced from 89.20 mg/L to 65.75, 55.44, 46.03, 49.03, 75.62 mg/L at 0.5, 1, 1.5, 2 and 2.5 bar, respectively. The graph shows that COD removal increased with pressure increase up to 1.5 bar and then decreased with pressure increase because the dissolved particles are pushed into the permeate as pressure increases. 1.5 bar is the ideal pressure for COD removal. All of the above values have a standard deviation of  $\pm 15.2$  mg/L. Arnal et al. (2008) reported COD removal efficiency between 53 and 59% when working at 1.5 bar and 4.5 bar pressure.



Figure 19. Average COD removal

### 4.3.2 TSS removal

The analysis of textile wastewater shows that dyeing and bleaching are the major contributors to Total Suspended Solids (TSS) in the wastewater. Overall, the various processes had low TSS and moderate-to-high TDS levels. TSS was found to be 133.2 mg/L on average in wastewater effluent samples, with a standard deviation of 47.8 mg/L. The removal efficiency of the Multi Media filters (MMF) and the coarse filter for TSS was quite low having removal efficiency of 28.7 and 29.9% respectively. The TSS removal was also measured at different pressures ranging from 0.5 to 2.5 bar. The TSS removal efficiency achieved was 94.7, 95.9, 96.7, 93.4, 90.2% at 0.5, 1, 1.5, 2 and 2.5 bar, respectively. It can be seen in the graph that TSS removal increased with the increase in pressure up to 1.5 bar and afterwards it decreased with the increase in pressure. This happens because with the increase in pressure some suspended particles are pushed into permeate. The optimal pressure for TSS removal is 1.5 bar with a removal efficiency of 96.7%. Alves and de Pinho (2000) studied the efficiency of two stage ultrafiltration on the dyeing effluent and the results showed 99% removal of SS.



Figure 20. Average TSS removal

#### 4.3.3 TDS removal

Textile wastewater from various processes had low TSS and moderate-to-high TDS levels. The total solids include electrolytes, acids, and alkalis used in dyeing. Wastewaters from batch dyeing cotton with reactive dyes are typically high in dissolved solids because this dyeing process necessitates a significant concentration of salts and sufficient alkali to raise the pH to between 12 and 12.5.

Effluent Wastewater of textile industry was found to contain TDS level of 2400 mg/L on average. The results show that UF membranes are not very effective in removing TDS as the dissolved particles are smaller in size than the pores of UF. The removal efficiency of TDS remained between 20 to 25% for the applied pressure from 0.5 to 2.5 bars which shows that removal efficiency of UF membranes for TDS is independent of applied pressure. However, the highest removal efficiency of 23.7% was achieved at 2 bar pressure. Fersi and Dhahbi (2008) proved that UF membranes cannot retain monovalent ions.



Figure 21. Average TDS removal

#### 4.3.4 Turbidity removal

The turbidity was reduced from 5.08 to 2.03 NTU with the average removal efficiency of 60% while the coarse filter decreased the turbidity by 68.7% only from 2.03 to 1.59 NTU. The turbidity removal was also measured at different pressures ranging from 0.5 to 2.5 bar. The turbidity was reduced from 1.59 NTU to 0.47, 0.24, 0.31, 0.32, 0.48 NTU at 0.5, 1, 1.5, 2 and 2.5 bar, with a removal efficiency of 90.7, 95.3, 93.9, 93.7, and 90.6% respectively. The graphs show that turbidity removal increased with pressure increase up to 1 bar and then decreased slightly because as pressure increased, the dissolved particles were pushed into the permeate. The optimal pressure for turbidity removal is 1 bar. The results also indicate that more than 90% turbidity removal was achieved through UF membranes at all pressures. The duration of operation of the UF membrane is the most statistically significant factor along with the technical efficiency measure. Chemical cleaning can probably improve the efficiency performance of the UF membranes.



Figure 22. Average turbidity removal

#### 4.3.5 Color removal

Color is usually visible at dye concentrations above 1 mg/L and has been reported in textile manufacturing effluent at higher concentrations, owing to the fact that 10-15% of the dye is lost into wastewater during the dyeing process (Johnston et al., 1981). Although color is not specifically mentioned in the Pakistan Environmental Protection Act (PEPA), it is a concern in dye house effluent because, unlike other pollutants, it is easily visible. As a result, removing color is critical for improving the public's perception of a factory. Color, on the other hand, is less of a concern as a health and environmental issue than many of the other parameters.

It can be seen in the graph that Multi Media filters (MMF) achieved 26.3% color removal and the coarse filter only managed to remove 15.7% of the color. The color removal efficiency was also studied at pressure from 0.5 to 2.5 bar. It was noted that color removal efficiency was highest of 40.7% at lowest pressure of 0.5 bar. The increase in pressure resulted in the decrease of color removal as the removal efficiency achieved was 24.6, 18.6, 8.5 and 5.1% at 1, 1.5, 2 and 2.5 bar respectively. The removal efficiency decreases with increase in pressure because the color is mainly due to soluble organic particles which pass easily through the UF membranes with increased pressure. Although ultra-filtration (UF) removes particles and macromolecules, it does not remove color completely. However, UF permeate meets the requirements for allowing wastewater reuse in minor textile industry processes (rinsing,

washing) (Allègre et al., 2006). Buscio et al. (2015) treated real textile effluent and reported color removal of 30%.





## 4.3.6 pH

The basic principle of electronic pH measurement is to determine the activity of hydrogen ions using a potentiometer and a standard sensing electrode (glass electrode) and a reference electrode. The pH of a solution is a measurement of the acid-base equilibrium achieved by various dissolved compounds. The rate of microbial growth is greatly influenced by the pH of the environment. The function of metabolic enzymes is influenced by PH. Most microorganisms thrive in the pH range of 6.5-8.5. This parameter is significant because aquatic life, such as most fish, can only survive in a narrow pH range of 6-9.

The pH of the dyes in textile processing units is a critical factor in the dyeing step because it affects their solubility. The pH also varies depending on the type of cloth used. The pH of the textile composite wastewater was found to be pressure independent, remaining in the range of 8.39 to 8.46. (Within NEQS range). Thus, the wastewater of this textile industry is alkaline in nature because caustic and other alkali detergents are used in large quantities in most of the steps.



Figure 24. pH in permeate

Description	MMF	Coarse filter	UF @ 0.5	UF @ 1	UF @ 1.5	UF @ 2	UF @ 2.5
Parameters	Sample	UF	Bar	Bar	Bar	Bar	Bar
Feed COD (mg/L)	103	91	89	89	89	89	89
Permeate COD (mg/L)	91	89.20	65.75	55.44	46.03	49.03	75.62
COD Removal (%)	11.65	2.19	26.12	37.70	48.28	44.91	15.03
Feed TDS (mg/L)	2384	1953	1907	1907	1907	1907	1907
Permeate TDS (mg/L)	1953	1907	1474	1492	1482	1455	1473
TDS Removal (%)	18.07	2.35	22.70	21.76	22.28	23.70	22.75
Feed TSS (mg/L)	122	87	61	61	61	61	61
Permeate TSS (mg/L)	87	61	2	2.5	2	4	6
TSS Removal (%)	28.68	29.88	94.72	95.90	96.72	93.44	90.16
Feed Color (Pt-Co)	190	140	118	118	118	118	118
Permeate Color (Pt-Co)	140	118	70	89	96	108	112
Color Removal (%)	26.31	15.71	40.67	24.57	18.64	8.47	5.08
Feed pH	8.7	8.39	8.46	8.46	8.46	8.46	8.46
Permeate pH	8.39	8.46	8.43	8.44	8.43	8.45	8.45
Feed Turbidity (NTU)	5.08	2.03	1.59	1.59	1.59	1.59	1.59
Permeate Turbidity (NTU)	2.03	1.59	0.47	0.24	0.31	0.32	0.48
Turbidity Removal%	60.0	68.7	90.7	95.3	93.9	93.7	90.6

Table 4. Summary of the results during test I to V (average).

# Chapter 5

# **Conclusion and Recommendations**

## 5.1 Conclusion

The high-water consumption in the textile industry has caused water scarcity and the increase of water cost in certain regions. In addition, the new environmental policies are focused on water recycling and reuse. Wastewater reuse involves both environmental and economic benefits. On the one hand it decreases the discharged of pollutant into the environment and on the other hand it allows to reduce water consumption and the associated costs.

According to results the Desizing is the most polluting wet process of the textile industry having high values of COD, BOD, TSS and TDS. However, Color mainly comes from the Printing Processes. The UF plant contributed to COD removal of 46% and color reduction of about 19%. The highest efficiency was obtained with the suspended solids (95%). The product water, still needs to be processed through RO before it can be used for recycling in wet processes of the textile industry because of high TDS and Color. The combination of ultrafiltration with reverse osmosis is better suited to treat textile wastewater as it reduces all the studied parameters, including color and TDS.

### **5.2 Recommendations**

The desizing wastewater should be separated from the rest of the wastewater and treated separately to minimize the treatment costs. Secondly, different UF membrane types should also be studied for the textile wastewater reclamation. The pilot scale UF plant should also be studied for the recovery of Indigo dyes used in the denim industry (Indigo dyes have larger molecular weight than UF pore size).

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