

# **Hybridization of Biological Systems to Treat Real Textile Wastewater**



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**MASTER OF SCIENCE**

**in**

**ENVIRONMENTAL ENGINEERING**

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**H-12, Islamabad 44000, Pakistan**

**2017**

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Thesis submitted to the Institute of Environmental Sciences and  
Engineering in partial fulfillment of the requirements for the  
degree of

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*This thesis work is dedicated to the profound love and strength of my beloved parents who brought me to the destination and taught me that it is never too late to pursue your true passion and that context is everything.*

# Acknowledgements

Utmost sense of obligation to Allah Almighty for giving me an insight and strength to undertake this research objectively and efficaciously.

***“He [Allah] grants wisdom to whom He pleases; and he to whom wisdom is granted indeed receives a benefit overflowing. But none will grasp the Message except men of understanding.” [2:269]***

I pay my sincerest gratitude to my worthy supervisor, Dr. Sher Jamal Khan, who has supported me throughout my research with his patience and knowledge whilst allowing me the room to work in my own way. His invaluable guidance and encouragement during this strenuous work has been a constant source of learning during my stay at Institute of Environmental Science & Engineering, IESE-SCEE, NUST. One essentially could not wish for a friendlier mentor & supervisor.

I am thankful to all members of the Guidance and Examination Committee (GEC) Dr. Imran Hashmi, Dr. Zeshan Sheikh and Dr. Yousuf Jamal who were extremely reliable source of practical scientific knowledge. I attribute the level of my Master’s degree to their consolation and without their guidance this thesis, as well; would not have been completed or composed. I would also like to thank IESE staff who contributed a lot to the research work during my tenure. I am grateful for the finding sources that allowed me to pursue my research work: IESE Digital library and IESE laboratories for providing the numerous tools and equipments I needed to carry out my research. Special thanks to lab assistant Muhammad Basharat, who graciously supported my efforts with his time, interest and his extensive knowledge on experimental laboratory analytical techniques.

I take pride in acknowledging the insightful guidance and support of my Mom and Dad for their selfless love and showing faith in me. I would never be able to pay back the love and affection showered upon by my parents. Heartfelt gratitude to my siblings Dr. Ayesha Shaukat, Dr. Asma Shaukat and Hira Shaukat for their cherished prayers and their backing which made me confident throughout my journey towards this degree. Above all a special thank you to a very extraordinary man in my life, my dearest Husband Hasan; who has been my rock this whole time through his meaningful words, moral support and faith in me especially during the challenging final stages of my MS degree.

I owe profound thanks to my best friend Orooj Surriya, who was always around at hard times and helped me to keep things in perspective as well as her motivation aided me to reach my desired achievements beyond my own expectations through this arduous journey. I feel great pleasure to express my gratitude to friends especially Ayesha Asif, Shabilla Parveen and Alia Aslam for invigorating me during this research. Last but not the least, I owe a debt of gratitude to MBR group fellows Khadija Noor, Ammara Haider, Tabish Khan, Usman Saleem, Adnan Rao, Muhammad Hanan Masood and Aamir Khan for their kind support.

***RABIA SHOUKAT***

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## **LIST OF ABBREVIATIONS**

<b>Abs</b>	Absorbance
<b>AeSBR</b>	Aerobic Sequencing Batch Reactor
<b>AnCSTR</b>	Anaerobic Continuously Stirred Tank Reactor
<b>APHA</b>	American Public Health Association
<b>BOD</b>	Biological Oxygen Demand
<b>CAS</b>	Conventional Activated Sludge
<b>CH<sub>4</sub></b>	Methane
<b>CO<sub>2</sub></b>	Carbon Dioxide
<b>COD</b>	Chemical Oxygen Demand
<b>CSTR</b>	Continuously Stirred Tank Reactor
<b>DO</b>	Dissolved Oxygen
<b>EC</b>	Electrical Conductivity
<b>EPA</b>	Environmental Protection Agency
<b>F/M</b>	Food to Microorganism Ratio
<b>HRT</b>	Hydraulic Retention Time
<b>IESE</b>	Institute of Environmental Sciences & Engineering
<b>MBR</b>	Membrane Bioreactor
<b>mg/l</b>	Milligram per Liter
<b>MLSS</b>	Mixed Liquor Suspended Solids
<b>MLVSS</b>	Mixed Liquor Volatile Suspended Solids
<b>NEQs</b>	National Environmental Quality Standards
<b>OLR</b>	Organic Loading Rate
<b>POPs</b>	Persistent Organic Pollutants
<b>SBR</b>	Sequencing Batch Reactor

<b>SRT</b>	Solid Retention Time
<b>TDS</b>	Total Dissolved Solids
<b>Temp</b>	Temperature
<b>TKN</b>	Total Kjeldahl Nitrogen
<b>TOC</b>	Total Organic Carbon
<b>TSS</b>	Total Suspended Solids
<b>VFA</b>	Volatile Fatty Acids
<b>μS/cm</b>	Micro Siemens per Centimeter

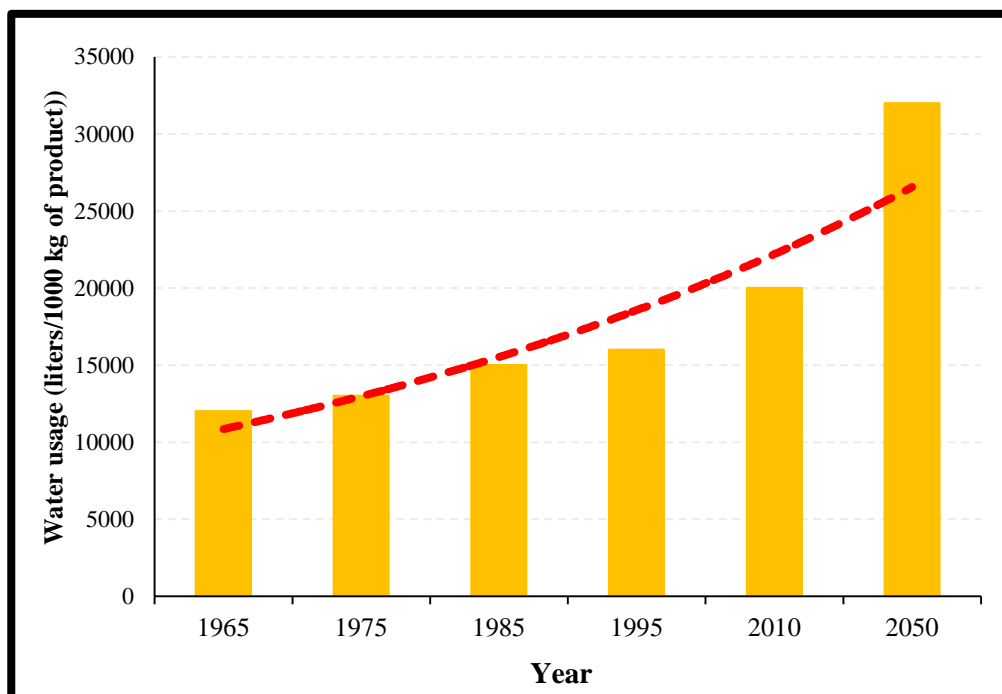
## **ABSTRACT**

Wastewater reclamation from textile industrial sector using hybrid biological technology of anaerobic CSTR - aerobic SBR is considered to be a feasible option to adopt in developing countries. Microbial consortium in anaerobic process has the ability to degrade persistent and complexly degradable compounds resulting into the production of biogas energy in addition to the removal of biodegradable organics and decolorisation from textile wastewater. This study evaluates the performance of lab scale hybrid anaerobic-aerobic biological treatment technology for real textile wastewater on the basis of system evaluation and optimization by running system with synthetic industrial wastewater. In Phase I, system was optimized at laboratory scale on synthetic wastewater and later real textile wastewater was treated in Phase II. The study was conducted for 242 days for studying real textile wastewater treatment. Activated sludge used in the process was obtained from NUST Membrane Bioreactor (MBR) Plant and acclimatized anaerobically to synthetic and real textile wastewater over a period of 120 days. The experimental results showed that for textile wastewater of variable composition, an individual application of conventional aerobic treatment technologies is not sufficient and effective. By application of hybrid anaerobic-aerobic system, removal efficiencies of 95.6% dyes, 99.5% COD and 78.4% TKN were achieved. The anaerobic treatment technology plays the key role in treatment efficiency in addition to the generation of sustainable energy source, biogas. Study inferred that aerobic treatment is required to enhance treatment level by providing final polishing to the anaerobically treated effluent. Implication of this work to the industrial scale would be a sustainable solution for reclaiming the treated wastewater for agricultural sector and textile processing operations.

## **INTRODUCTION**

### **1.1 Background**

Textile industry is deliberated as one of the uppermost Pakistan’s industry which significantly contributes to the country’s economy, involving a variety of intricate technological operations. The production of fundamentals covers both the natural fibers i.e. cotton, yarn, wool and artificial fibers i.e. polyester, acrylic, nylon, ending up adding an excessive value to the economy of developing countries. Textile wet processing operations involve the utilization of large amount of water which results into the large quantity of wastewater generation (Holkar et al., 2016). Global water consumption in wet processing operations for cotton textile industrial sector is shown in Figure 1.1.



**Figure 1.1: Global water usage for textile industry (Adopted from Aksu et al. 2015).**

The wastewater composition predominantly includes chemicals like acids, alkalis, dyes and other toxic compounds (Paul et al., 2012). In last few years, a prompt involvement of textile manufacturing industries handles an extensive range of products in which the use of process chemicals and dye products have been increased manifold. Therefore, wastewater generation is a chief conservational impediment for the progression of textile industry in addition to the release of large amount of colored wastewater which is well-thought-out to be a major concern referring to environmental problems and public health. The direct discharge of industrial wastewater into water bodies results in the non-aesthetic and lethal pollution and harmful risks to the ecosystem. Unlike domestic wastewater treatment, traditional wastewater treatment technologies are not sufficient to treat industrial wastewater to remove COD and degrade dye compounds. Developing countries like Pakistan lack the vision of textile wastewater treatment relative to the developed countries which are profound to meet industrial wastewater discharge standards to oversee environmental and health issues. In developed countries, an approach to meet stringent requirements of textile discharge is in practice by acquainting with numerous types of wastewater treatment technologies. Previously, it was reported that an average of 20 % of industrial wastewater is contributed by merely textile dyeing and finishing treatment (Kant, 2012). It is also reported in literature that ordinarily dye compounds used in dyeing process are made from carcinogens i.e. benzidine, aromatic compounds (Sarayu & Sandhya, 2012).

### **1.1.1 Environmental Impacts of Textile Discharge**

Among the distinct categories of technological industries in an industrial hub, textile industry is considered as one of the largest source of environmental pollution to the environment. Textile wastewater discharge contains high organic loads, synthetic dyes and chemicals that come under the category of Persistent Organic Pollutants (POPs)

which sustain in the environment and takes long term degradation rate which makes the treatment of textile wastewater a major environmental obstacle (Utami et al., 2016). Characteristics of textile wastewater like turbidity due to colloidal matter interferes the oxygen transfer and results in the eutrophication effecting aquatic life along with aesthetic deterioration (Kant, 2012). Due to large environmental impact of direct disposal of textile wastewater, anaerobic wastewater treatment technology provided with aerobic MBR as a post treatment endows with the best approach of environmental safety. The proportional increase in textile wastewater discharge is due the demand of textile products resulting from exponential growth of global population. The presence of nondegradable dyes and additives necessitates the increase of economical and efficient treatment technologies that satisfy the demand of industrial ecology.

## **1.2 Eminent Technologies for Textile Wastewater Treatment**

Industrial wastewater discharge problems allied to deteriorating conditions of environment familiarized the concept of biological treatment technologies. Several approaches of wastewater treatment technologies have been adopted to treat synthetic textile wastewater. But inadequate research have been done in treating real textile wastewater due to its distinct complex nature. Today's most encouraging wastewater treatment technologies like advanced Membrane Bioreactors provide sustainable advancements for the treatment of low strength wastewater i.e. municipal wastewater, efficiently. Research studies has shown that a lot of effort has been performed on aerobic MBR subjected to the treatment of municipal wastewater, indeed is experiencing an unprecedented development nowadays. Contrarily a certain number of limitations of MBR technology usually came across in case of industrial wastewater treatment which is characterized as high strength wastewater. Anaerobic biological treatment is highly predictable for the treatment of wastewaters that are not considered



for the aerobic treatment due to inauspicious constraints (Hai et al., 2013). Certain parameters of industrial wastewater are considered to be dominantly distinguishable unlike municipal wastewater i.e. organic strength (COD >1000 mg/L), variable range of pH, temperature, salinity and color (Lin et al., 2012). Therefore, treatment of industrial wastewater by aerobic MBR requires high operational cost of aeration in addition to the production of greenhouse gases i.e. carbon dioxide and bio-solids. On the other hand, anaerobic treatment is an exclusive treatment that can be used for the treatment of high strength industrial wastewater by producing methane gas at lower operational costs.

Biological treatment consists of innumerable categories depending upon the target pollutants to be removed in treatment performance. Furthermost common technologies which have been in practice since early years for low strength wastewater treatment are conventional activated sludge process and trickling filters. Trickling filters have been in practice but it involves the media to promote bacterial growth which is accountable for treatment. Due to certain limitations like high frequency of media clogging and limited flexibility, the proficiency of trickling filter is relatively less than activated sludge process. Comparatively, in activated sludge type treatment, bacterial microbes in the sludge are involved which are responsible for an effective treatment without flocculating wastewater.

### **1.2.1 Conventional Activated Sludge (CAS)**

Conventional treatment technologies principally adopted to treat municipal wastewater are ordinarily slow in operation because of their lack of potential to remove pathogens from the effluent. Amongst conventional biological treatment technologies, activated sludge technology is not effective in degradation of disperse type azo compounds. CAS is not acclaimed for treating textile wastewater as it involves high organic loading and

dyes i.e. aerobic bacteria are accountable for treatment which are inefficient and incapable for degrading complex dye compounds (Oh et al., 2004).

### **1.2.2 Membrane Bioreactors (MBR)**

The treatment performance varies depending upon the nature of treatment technologies subjected to treat high strength textile wastewater. The most advanced and efficient treatment technology of treating wastewater is membrane bioreactors in which the combination of biological processes is a key concern to achieve acceptable level of treatment. Membrane bioreactors are composed of two primary processes i.e. a combination of biological treatment and filtration. Based on these principles, membrane bioreactors can be of anaerobic or aerobic type reactors depending upon the treatment type. The effluent quality of aerobic MBR with respect to COD is better than any conventional technologies making effluent particle free due to the absorbance and enhanced degradation of colloidal compounds. The intensified potential of treatment and operational flexibility is perhaps the chief advantage that drags its importance of installation especially for the treatment of high strength wastewaters (Judd, 2010). MBR technology professed novelty supports the investment decisions for the treatment of wastewater as it provides enhanced beneficiaries of energy efficiency, improved operational procedures and longer warranty.

### **1.2.3 Hybrid Anaerobic-Aerobic Treatment Systems**

The ignorance of industries in the direct discharge of wastewater which is highly toxic, resulting in detrimental environmental effects. Therefore, treatment of high strength industrial wastewater demands highly proficient treatment technology. For the key concern of better quality effluent, anaerobically treated wastewater can be further subjected to aerobic treatment. Anaerobically treated wastewater usually comes up with the problem of biomass separation which may be overcome with the aerobic post

treatment to meet the high quality effluent criteria (Lerner et al., 2007). Therefore, industrial wastewater with high organic loads can be treated with hybrid anaerobic-aerobic treatment technology in a cost effective and energy efficient manner by varying the parameters of HRT and SRT.

The theory of wastewater treatment by anaerobic and aerobic processes have been introduced since early but the practical applications were not executed and established due to lack of interest in regard to the treatment of industrial wastewater. This combined technology encompasses on the anaerobic type reactor where the retention of biomass takes place so that the concentration of sludge is usually kept high in the anaerobic reactor. The production of methane gas makes this concept of hybrid technology more demanding & challenging prior to solitary aerobic membrane bioreactor system. Therefore, this experimental study evaluates the treatment performance of textile industrial wastewater using the technology of hybrid anaerobic CSTR – aerobic SBR at laboratory scale providing highly effective treatment efficiencies. The combination of various treatment technologies can provide enhanced treatment efficiency in treating high strength wastewaters (Popli & Patel, 2015).

### **1.3 Problem Statement**

Large amount of wastewater generation from textile industrial hub results in environment pollution. Toxic nature of textile wastewater along with a custom of large magnitude of water for industrial operations demands a regulatory control and reliable treatment. This problem has been accomplished by providing a solution of various wastewater treatment technologies. Aerobic MBR has a limitation of membrane fouling and its consequences in terms of plant maintenance and operating costs confine the applications of MBRs to treat high strength textile wastewater. The most concerned problem in textile wastewater is the engrossment of chemicals and nondegradable dyes

that result in environmental and public health problems. Presently, aerobic conventional and advanced wastewater treatment technologies are applicable for municipal wastewater treatment but considered to be inadequate for treating high strength textile industrial wastewaters, effectively.

## **1.4 Objectives**

In this study, the well-established hybrid system for textile wastewater treatment has been endorsed to treat high strength textile wastewater effectively along with energy resource recovery. The research activities were conducted in the Water and Wastewater Laboratory of Institute of Environmental Science and Engineering (IESE), National University of Sciences & Technology, Islamabad. The research objectives were deliberated as follows:

1. Performance evaluation and optimization of hybrid anaerobic - aerobic system by varying HRTs, using synthetic textile wastewater.
2. Treatment of real textile wastewater at optimized conditions.

## **1.5 Thesis Layout**

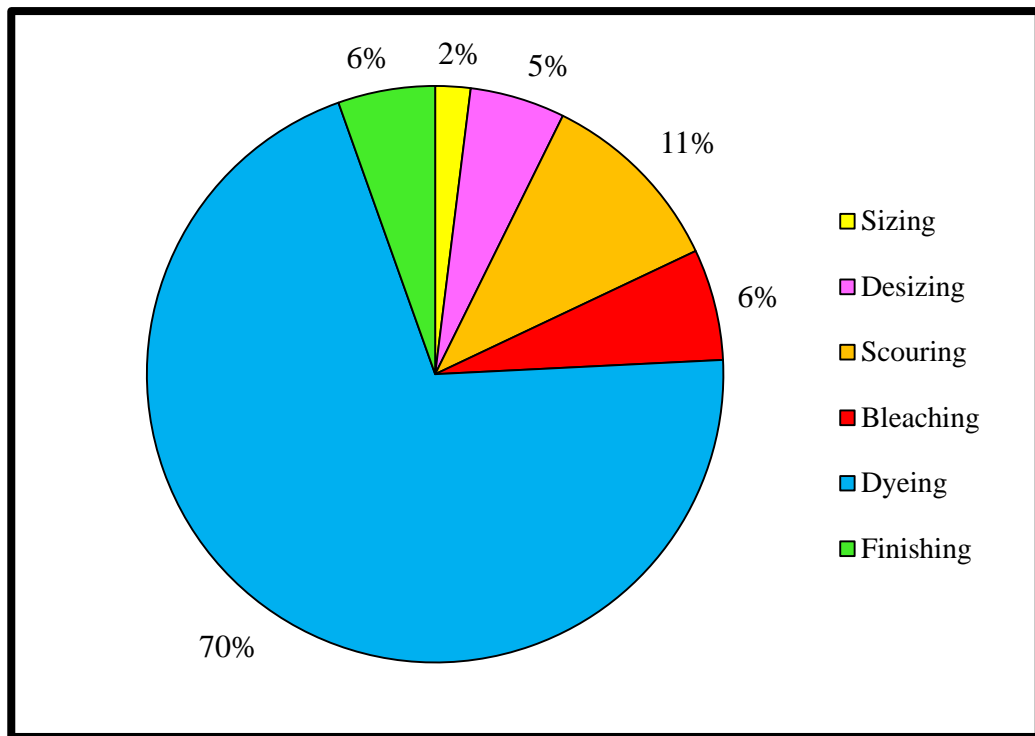
Before proceeding the comprehensive description of the whole research study carried throughout, thesis layout has been designed for the convenience of the reader which has been shown in Annexure A.

# **LITERATURE REVIEW**

## **2.1 Trends of Textile Wastewater Generation**

Textile industry is categorized into primary sectors of cotton, woolen and synthetic fibers in respect to the type of raw material used. The industrial competency in global research and innovation enlightens the concept of sustainability in developing countries where it is well-thought-out as the backbone of country's economy. Among the industrial hub, textile industries are considered as one of the exceedingly engineered and momentous growth potential industries. Specifically being concerned with the large amount of wastewater generation through various textile operations, the management and treatment is still a bottleneck especially in developing countries like Pakistan. Wastewater discharge from industries containing toxic chemicals like acids, dyes, surfactants, metals and dispersing agents is becoming a chief environmental challenge for the industry and the population.

The wastewater discharge from textile industry is in large quantity because of high quantities of water used in its processes in addition to the complex composition of wastewater as it involves energy and time consuming multi operational processes. For a textile fabric production unit of 20,000 lb/day a quantity of 36,000 liters of water is consumed in cotton processing (Shaikh, 2009).

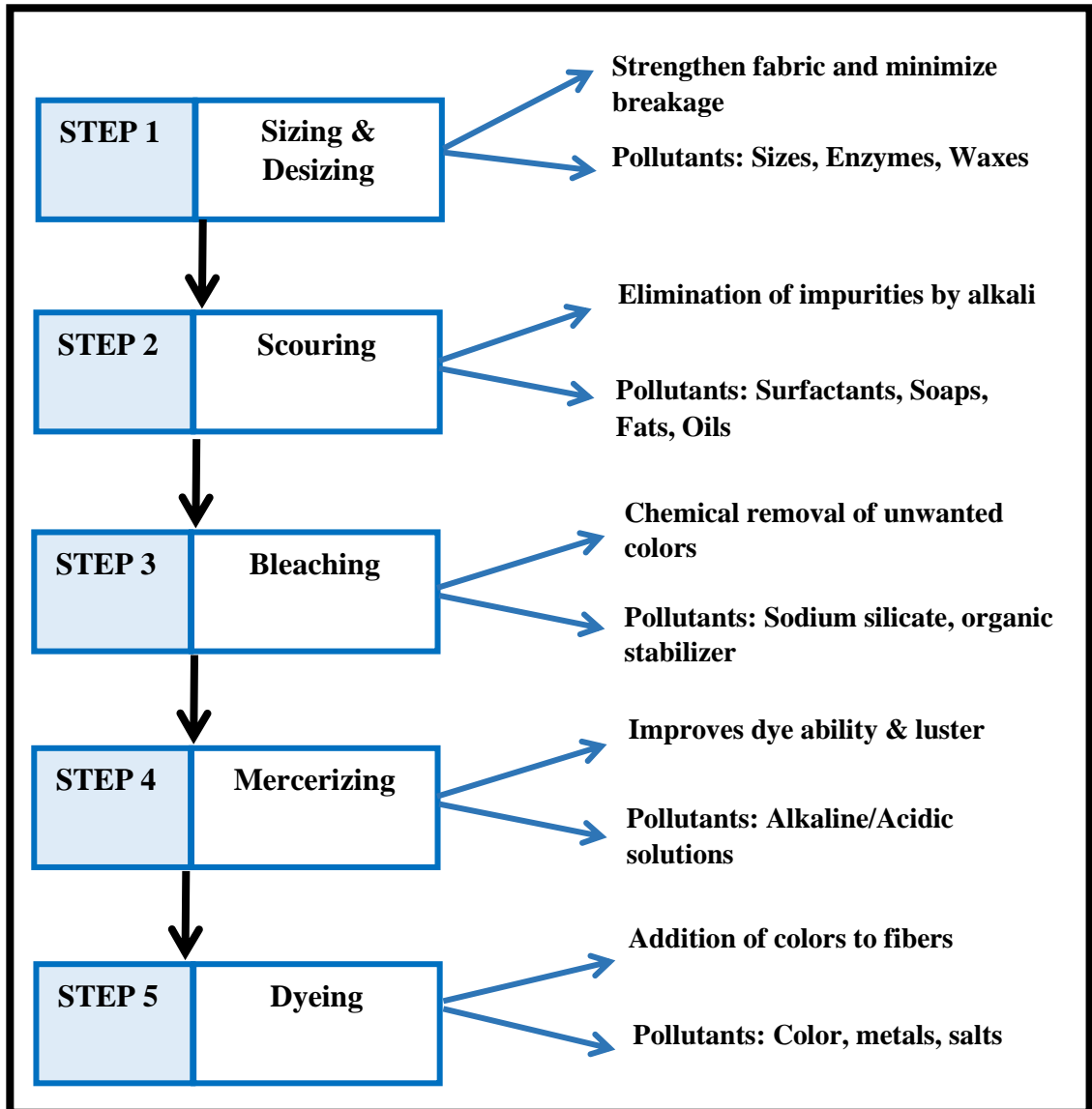


**Figure 2.1: Volume of water required per unit operation of cotton industry**  
(Adopted from (Shaikh, 2009).

Figure 2.1 describes the consumption of water by every operational unit in textile industry. China's industrial wastewater generation has reached to 198.9 million tons increasing with a rate of 1% and is subjected to treatment with several wastewater treatment technologies before being discharged into the water bodies (Meihong, 2007).

### 2.1.1 Textile Operations and Processing

Textile processing technology involves steps like sizing, desizing, scouring, bleaching, mercerizing, dyeing and finishing, that causes various pollutant discharges. Notably, cotton dyeing step involves chromophore groups like azo, carbonyl and nitro which chiefly enhance the unacceptable toxicity of textile wastewater (Rodrigues et al., 2014). The types of pollutants allied to the operational steps are described schematically in Figure 2.2.



**Figure 2.2: Schematic illustration of textile industry unit operations and pollutants generated at each processing level (Adopted from (Rodrigues et al., 2014)).**

### 2.1.2 Toxicity of Textile Wastewater

In addition to high organic loads from textile effluents, the primary target pollutant is color which if remains untreated can be chaotic and therefore these environmental concerns emphasize the importance of reuse of textile effluents (Ciardelli et al., 2001). Colorants releasing in textile outflow are mainly from the dyeing processing step which comprises the use of water as a standard medium for dye application (Verma et al., 2012). The dyes from dyeing operations are usually complex compounds which are

resilient to degrade and released as target pollutant which should be monitored. The structure of dyes also reported as of high stability which makes them very resistant to degrade easily while providing treatment (Hirani et al., 2010).

Wastewater discharge from textile industries in developing countries under unsuitable conditions, results into significant negative impacts affecting local environment and surrounding land area. Aquatic life is diversely affected due to the presence of particulate matter, oil and grease and high organic loading. These industrial effluents contain chromium compounds which destroy the food chain resulting into alteration of the habitat.

### 2.1.3 Pakistan Textile Industrial Discharge

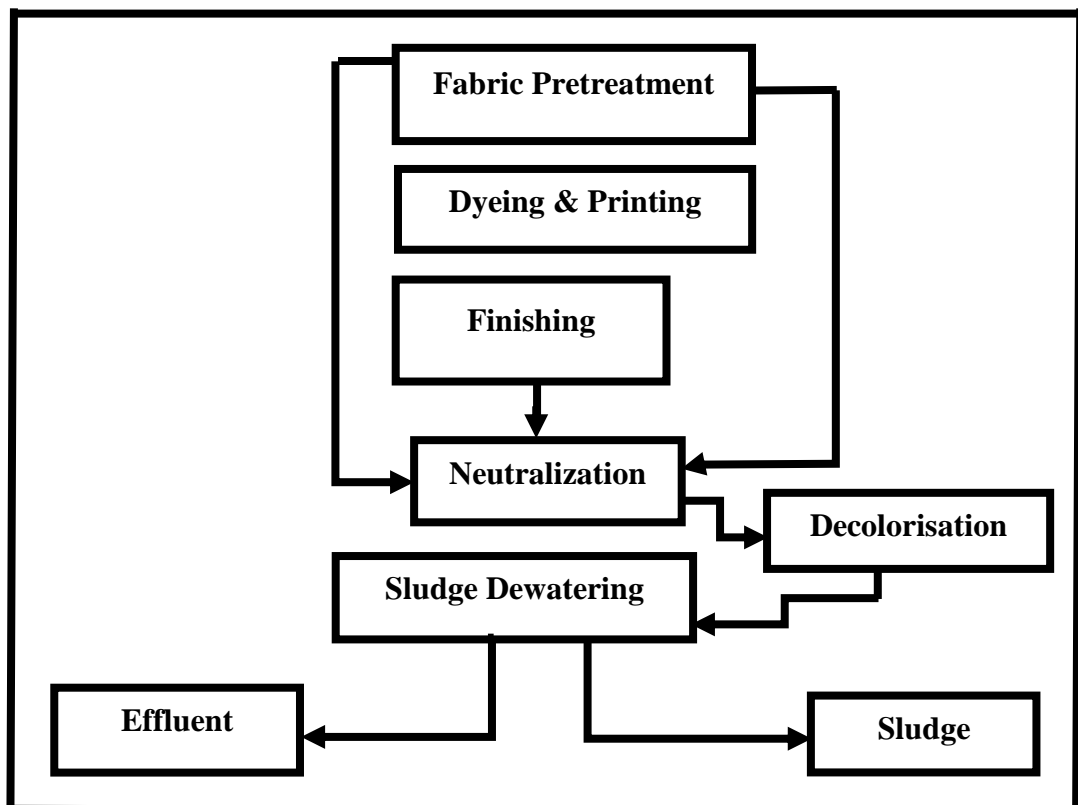


Figure 2.3: Wastewater treatment flowchart from textile processing entities (Adopted from Ohiona et al., 2009).



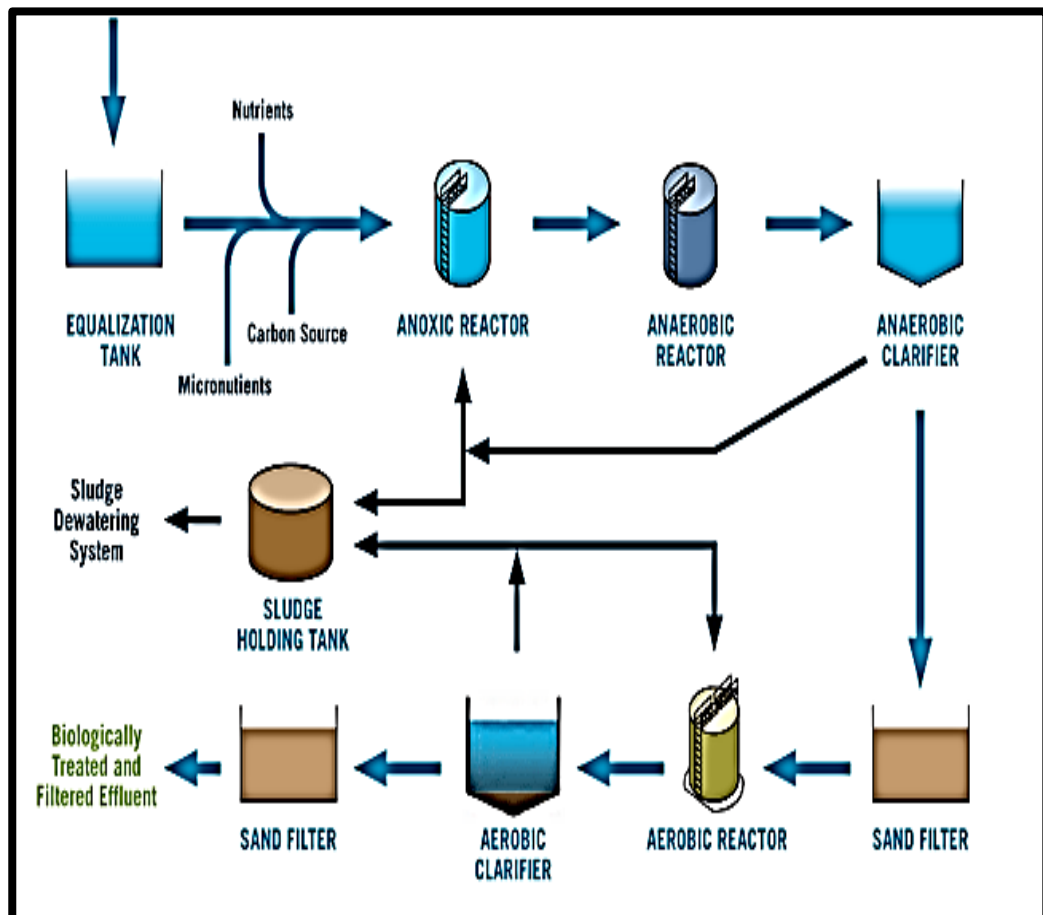
Practicing direct discharge of highly polluted industrial wastewater is worst in developing countries like Pakistan where treatment of industrial wastewater is not in practice relative to domestic wastewater treatment (Ohioma et al., 2009). In developing countries, typical wastewater treatment flow which has been practiced in textile industries is shown in Figure 2.3. Wastewater discharge from industries can be cut down to certain limits but in case of textile industries, major practices are exceedingly dependent on processes that generate a large amount of wastewater (Seif & Malak, 2001). Adoption of anaerobic technology in developing countries like Brazil, Colombia, and Pakistan is in practice especially in arid and semi-arid regions where the warm climates provide efficient wastewater treatment (Chernicharo, 2006). Textile wastewater standards are usually overlooked in under developed countries and the reason of this incomprehension is that either the standards are too stringent or too easy-going that don't even promise the environmental protection. The stringent compliance requirements lead to adopt alternative technologies (Visvanathan et al., 2000). In China, textile industry has set its standards to save the local environment which sets a rousing example for other highly populated countries to take steps in adapting the effluent treatment technologies (Wang et al., 2011).

## **2.2 Biological Wastewater Treatment**

Since 19<sup>th</sup> century, the use of microorganisms, to degrade organic and inorganic pollutants present in water, is in practice with advancement in technologies like trickling filters, activated sludge and septic tanks (Luo et al., 2014). In under developing countries, alternative to unsatisfactory performance of various physico-chemical treatment methods for complete degradation of toxic pollutants is the biological treatment (Ohioma et al., 2009). The wastewater subjected to conventional

biological treatment plant as shown in Figure 2.4, involves the secondary treatment in which biological removal of organics takes place (Silva et al., 2014)

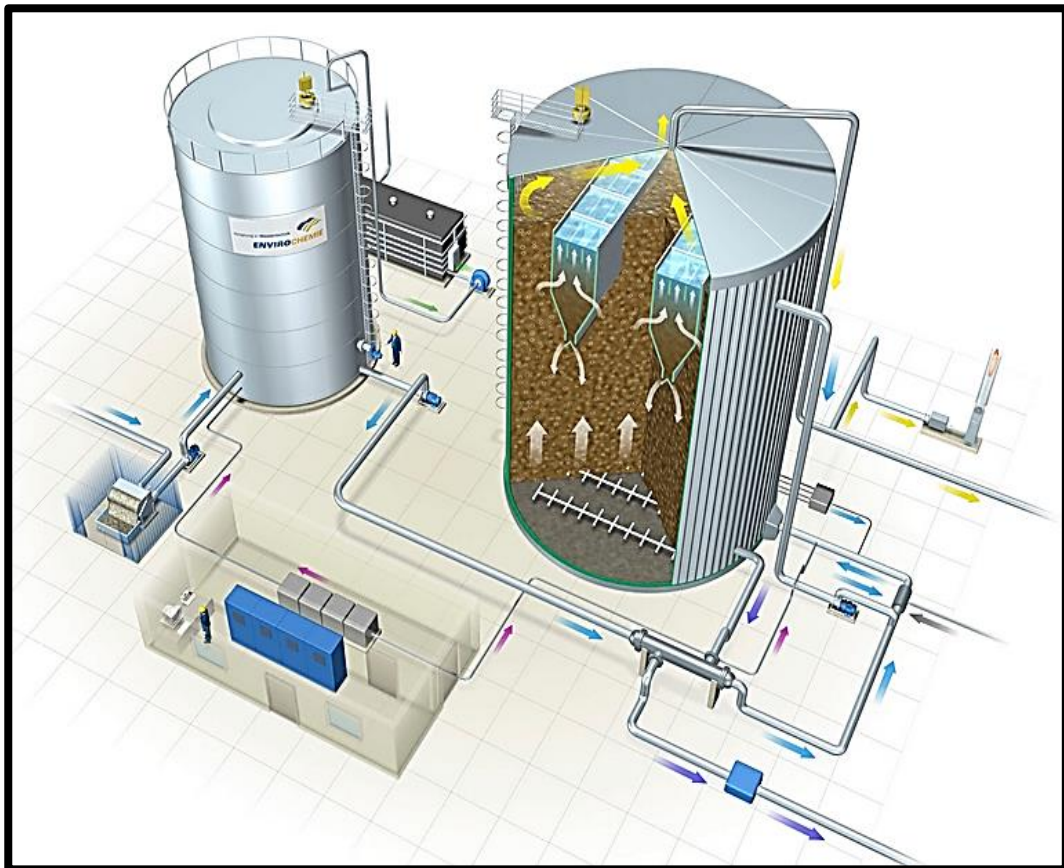
- For industrial wastewater objective is to remove or reduce concentration of organic or inorganic compounds
- Pretreatment of industrial wastewater may be required before being discharged to municipal collection system
- For agricultural irrigation return wastewater, objective is to remove nutrients, specifically nitrogen and phosphorous



**Figure 2.4: Schematic illustration of biological wastewater treatment system (Adopted from (Silva et al., 2014).**

## 2.2.1 Biological Trends in Industrial Wastewater Treatment

Biological treatment can be aerobic and/or anaerobic type and for the treatment of textile wastewater, both technologies coupled with one another can be a mainstream of biological treatment in comparison to the separate treatments exclusively. In textile wastewater, dye removal is of major concern. Typically, the amount of dyes present in textile effluent ranges between 10 to 50 mg/l (Yagub et al., 2014). Color removal from dyebath discharge can be effectively achieved by the use of biologically degradable microorganism consortium and the effect of biological activity on color reduction (Dao et al., 2016).



**Figure 2.5: Biological treatment plant to treat wastewater from paper industry installed at Brazil Envirochemie (Adopted from (Chernicharo, 2006).**

Dyes which are resilient to light and oxidative degradation are most likely to eliminate during biological processes involving the role of bacterial groups having xenobiotic

nature in azo based dyes e.g. genera of *Basidomycetes*.(Pandey et al., 2007). In addition, an anaerobic microbial consortium consisting methanogenic & acetogenic microbes involve unique enzymes, are responsible for wastewater decolorisation (Sem & Demirer, 2003).

### 2.2.2 Categorization of Biological Treatment Technologies

Biological treatment processes are generally classified in two categories and the characterization of both categories conventional activated sludge and membrane bioreactor has been done as shown in Table 2.1 and Table 2.2.

**Table 2.1: Categorization of biological treatment technologies.**

<b>TREATMENT TYPE</b>	<b>CATEGORIES</b>
Aerobic Biological Treatment	Activated Sludge
	Trickling Filters
	Oxidation Ditches
	Membrane Bioreactors
Anaerobic Biological Treatment	Septic Tanks
	Continuously Stirred Tank Reactor (CSTR)
	Upflow Anaerobic Sludge Blanket (UASB)

The activated sludge type biological treatment involves bacterial microbes in the sludge which are responsible to provide degradation in the chemistry of activated sludge treatment, the wastewater doesn't have to be flocculated (Ahmed et al., 2017). The provision of oxygen in advanced technology of membrane bioreactor is key consideration to achieve acceptable level of treatment (Radjenović et al., 2009).

Summarizing the fact that most common distinctive categories of biological wastewater treatment technologies are aerobic and anaerobic. The application of both types of technologies depends upon the types of wastewater subjected to treatment. Typically, the implementation of anaerobic treatment is considered when the wastewater is highly concentrated as the high concentration of wastewater drives the mechanisms of hydrolysis and acidification by microbial consortium present in anaerobic process.

Aerobic biological process is one of the most adoptable cost effective treatment technologies for municipal wastewater treatment. As this type of process involves the variety of aerobic microorganisms which are responsible for organic compound degradation present in low strength wastewater like domestic wastewater.

**Table 2.2: Comparative characterization of conventional activated sludge process vs. MBR (Adopted from Ahmed et al., 2017).**

<b>PARAMETERS</b>	<b>CONVENTIONAL ACTIVATED SLUDGE</b>	<b>MEMBRANE BIOREACTORS</b>
COD Removal Efficiency	94.5%	99.0 %
Dissolved Organic Carbon Removal	92.7%	96.9 %
Floc Size	Large	Small
Filamentous Organisms	Higher amount	Smaller amount
Biomass Fraction Variability	High	Low

### **2.3 Anaerobic Biological Treatment**

Anaerobic processes are considered as the best option for exploitation of biomass retention on membrane (Chernicharo, 2006). Anaerobic technologies are suitably

recommended for industrial wastewater of strength 4-5 g COD/L, as the high loading rate along with heat are required for anaerobic processes (Martin et al., 2011). This technology though facilitates reducing energy demand and sludge production but several studies report that its operational parameters affect the biological treatment performance. In anaerobic biological processes, the end products are usually in the form of biogas.

**Table 2.3: Advantageous of anaerobic systems for wastewater treatment (Adopted from McHugh et al., 2004).**

<b>INDIVIDUALITIES</b>	<b>DESCRIPTION</b>
Budget	Cost effective relative to physic-chemical treatment
Working Operation	Simple in operational procedures and processing
Energy Demand	Zero energy consumption or maximum possibility of energy minimization
Sludge	Less sludge production volume which makes ease of disposal

Anaerobic digestion process is considered as a unique type of biological treatment which is best suited for the treatment of high strength industrial wastewater specifically textile wastewater as heat required to achieve mesophilic temperatures (35 °C) in the reactor is only possible with high strength textile industrial wastewater (Martin et al., 2011). Anaerobic treatment systems are better for use as an effective alternative to treat high strength industrial wastewater at mesophilic temperatures in addition to the other advantages that are enlisted in Table 2.3 (McHugh et al., 2004). The introduction of anaerobic treatment systems in treating textile wastewater are mainly because of the resource recovery, reduction of aeration cost making the technology economically viable and above all less sludge production relative to aerobic treatment which decreases the disposal cost and requirement of landfills (Lin et al., 2009). Table 2.4

shows the flexible degrees of methanisation in AnMBR for wastewater treatment. The extreme methane yields perceived from research experimentations for AnMBRs from domestic wastewater is 0.29-0.33 L-CH<sub>4</sub>/g (Hu and Stuckey, 2006).

**Table 2.4: Variable degrees of methanisation in AnMBRs for actual wastewater influent (Adopted from Hu and Stuckey, 2006).**

WASTEWATER TYPES	DEGREE OF METHANISATION
	L-CH <sub>4</sub> /g
Screened Wastewater	0.20-0.23
Raw Wastewater	0.27
Black Water	0.09-0.12

### 2.3.1 Configurations of Anaerobic Biological Treatment Systems

For the treatment of low strength domestic wastewater, the applicability of anaerobic technology has been presented extensively (Chernicharo, 2006). Different types of wastewater has been exposed to anaerobic treatment provided with proper environmental conditions favorable to anaerobic microbes (Liao et al., 2006). Anaerobic biological systems for wastewater treatment are actually categorized on the basis of two main configurations:

- Upflow Anaerobic Sludge Blanket (UASB)
- Continuously Stirred Tank Reactor (CSTR)

#### 2.3.1.1 Upflow Anaerobic Sludge Blanket (UASB)

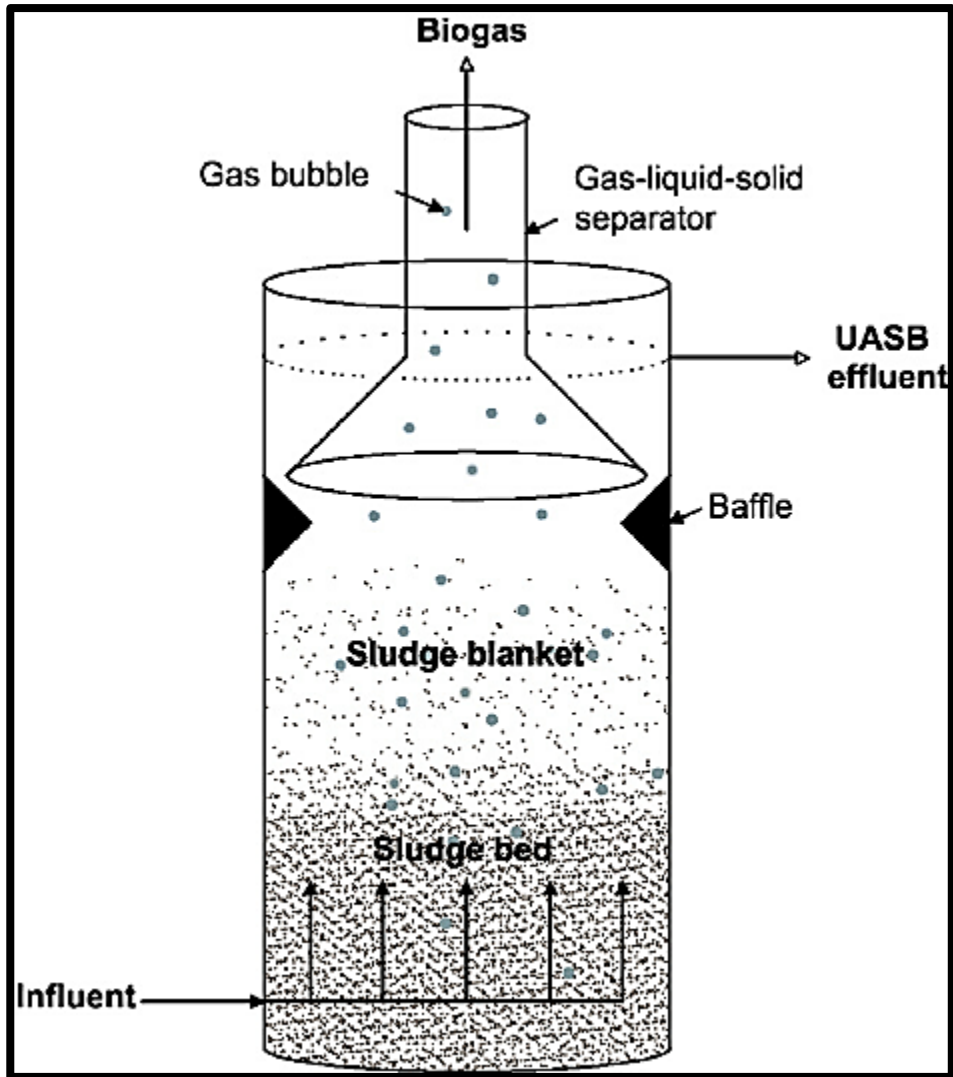
UASB technology is the treatment technique for treating high strength industrial wastewater. This technology consists of expanded granular sludge bed in which

reactions of hydrolysis, acidification, and methanogenesis takes place operating under different hydraulic retention times (HRTs) (Lu et al., 2015).

This technology is an approach to degradation and decolorisation of complex dyes along with the COD removal during anaerobic conditions which makes this technology widely acceptable (dos Santos et al., 2007). Decolorisation efficiency of UASB for textile wastewater containing azo compounds was found to be approximately 95% (Robinson et al., 2001). The removal of microbes i.e. helminths in UASB reactors has been reported on an average of 60-90% which makes it inadequate for direct agricultural usage (Işık and Sponza, 2008). UASB provides the benefit of less sludge production, 30-50% lower than trickling filter technology (Chernicharo, 2006).

Conventionally, UASB technology encompasses the use of granular sludge or films to retain biomass (Lin et al., 2009). The reactor design has multiple gas hoods for the collection of biogas as shown in Figure 2.6. The influent is allowed to enter from bottom of the reactor by peristaltic pump. The sludge bed is maintained in the reactor as the influent move upwards and allow the contact with the bed ensuring the stable effluent quality discharge. The basic principle of UASB allows the 3 phase reaction environment (Gas-Liquid-Solid) allowing the gas and liquid while retaining the solid in the sludge blanket.



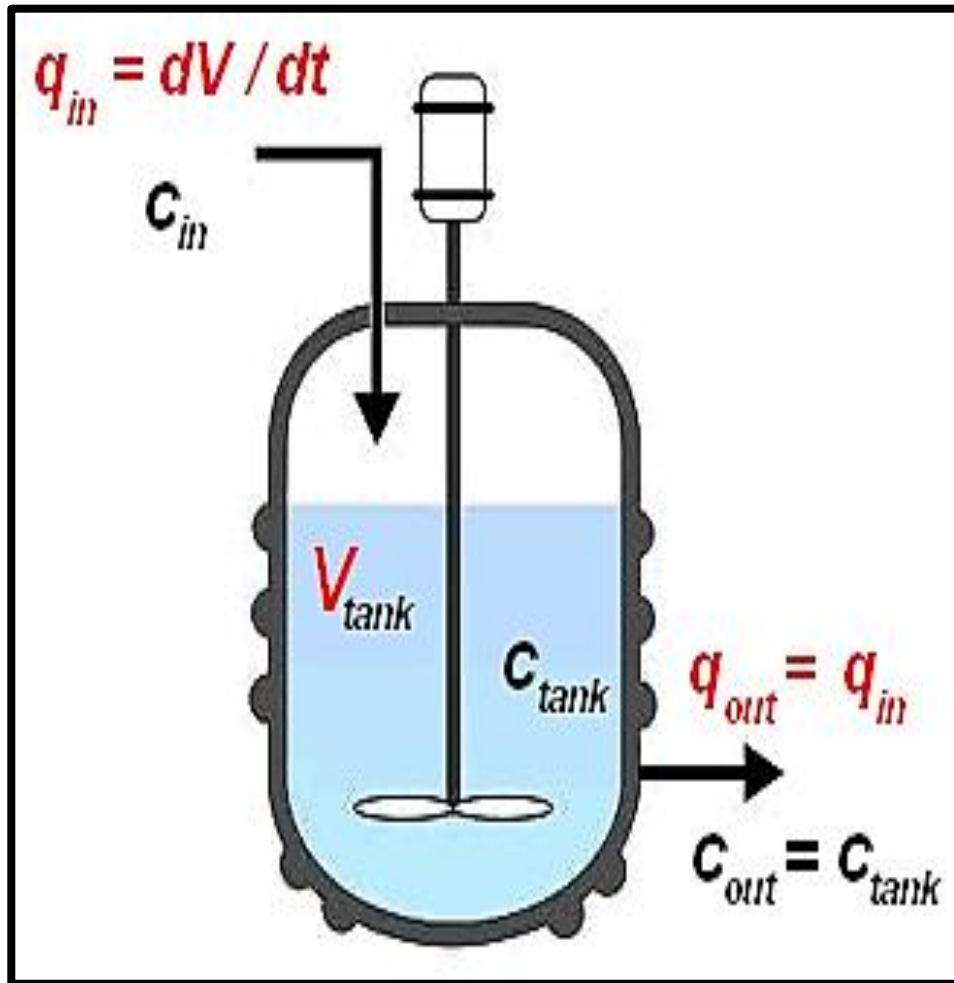


**Figure 2.6: Design of Upflow Anaerobic Sludge Blanket (Adopted from (Lin et al., 2009).**

### **2.3.1.2 Continuously Stirred Tank Reactor (CSTR)**

Anaerobic CSTR has benefits of modest designing, easy operation, low capital costs & importantly self-regulating biomass in the solution for treating high strength wastewater (Chan et al., 2009). The CSTR configuration for activated sludge can be a best approach for several industrial applications by permitting direct control over pH & temperature (Yuan et al., 2010). For the treatment of textile wastewater subjected to CSTR, the influence of bacterial activity fluctuates the performance i.e. increase in

wastewater loading rates provides an average of 90% COD removal in addition to color reduction (Khelifi et al., 2009).



**Figure 2.7: Design of Continuously Stirred Tank Reactor (Adopted from (Yuan et al., 2010)).**

By providing the modifications in existing wastewater treatment system designs, the high stability, greater loading capacities and desired process efficiencies are achieved e.g. CSTR can be upgraded to two phase systems as shown in Figure 2.7. For processes which comprises the process of substrate inhibition or product activation, CSTRs are highly preferred wastewater reactors in addition to providing the effective and promising wastewater treatment specifically the elimination of recalcitrant compounds from textile wastewater (Firozjaee et al., 2013). CSTR ordinarily have certain

limitations like solids settling especially in treating municipal wastewater where COD is of low strength (Gargouri et al., 2011).

### **2.3.2 Limitations of Anaerobic Biological Systems**

There are certain limitations that have been observed throughout the research studies while examining anaerobic treatment systems individually. These limitations can be due to:

- Slow degradation rate in anaerobic treatment; most of constituents removed slightly at higher effluent concentrations which lead to the requirement of secondary treatment (Marchaim, 1992).
- Though the anaerobic degradation of pollutants is a cost-effective natural process but its effluent discharge is typically not in compliance with the environmental standards which means this treatment cannot be recommended exclusively (Chernicharo, 2006).
- Temperature is an important aspect as it plays an important role in degradation of pollutant kinetics. The limitation of anaerobic system at low temperatures and longer SRT is the degradation rate is slow which shows the potential of this treatment is better in warmer climates (Kamali et al., 2016).

## **2.4 Aerobic Biological Treatment**

Aerobic biological treatment is not acclaimed for treating textile wastewater as it involves high organic loading. Bacteria as a primary agent, plays an important role in organic matter biodegradation in the presence of oxygen. The production of active mass of microorganisms involves the principle of stabilizing and converting the organic matter into end products of carbon dioxide and water. Aerobic biological treatment involves series of metabolic activities making aerobic bacteria treat domestic

wastewater, but are inefficient and incapable for degrading complex dye compounds (El Moussaoui et al., 2017; Oh et al., 2004).

### **2.4.1 Configurations of Aerobic Biological Treatment System**

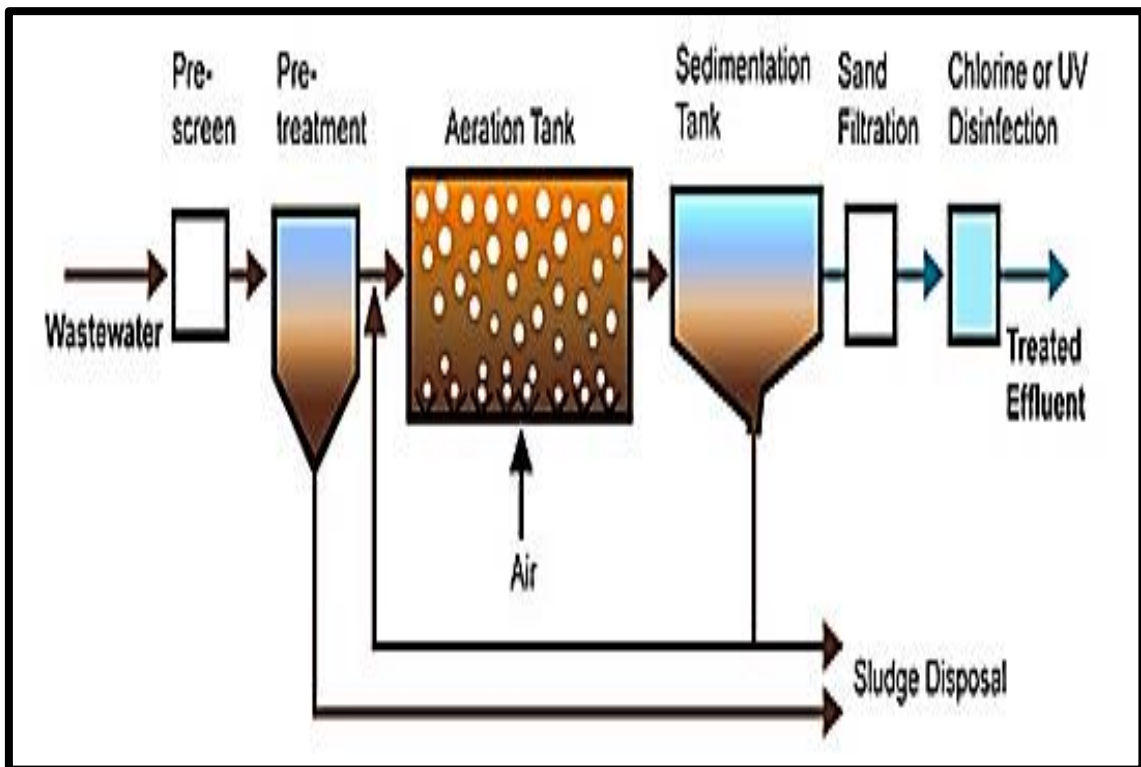
Aerobic biological treatment systems have been practiced to treat municipal wastewater as they are of low strength. For the treatment of low strength wastewater i.e. domestic wastewater having COD < 1000 mg/L, variety of aerobic biological technology is classified in various types. These configurations of aerobic biological systems has been adopted depending upon the type of wastewater.

1. Conventional Activated Sludge Process (CASP)
2. Trickling Filters
3. Oxidation ditches
4. Membrane Bioreactor (MBR)

#### **2.4.1.1 Conventional Activated Sludge Process (CASP)**

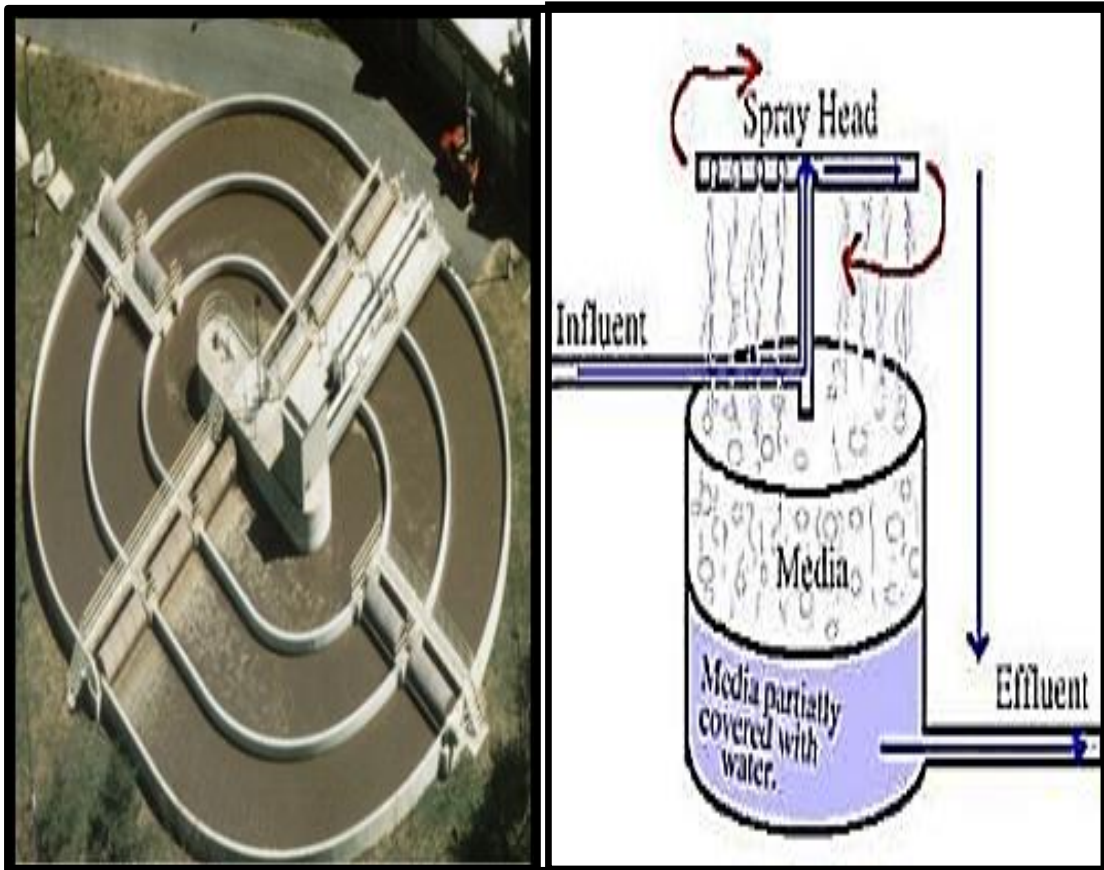
Activated sludge is most commonly adopted technology of wastewater treatment principally involving aeration and biological flocs composed of bacterial species and protozoa. CASP technology is the most typical type technology which has been practiced since earlier to treat wastewater of municipalities. The CASP systems typically involves the biological tank and a secondary clarifier. Aerobic biomass involved in the treatment process reduce the biochemical oxygen demand (BOD) and ammonia concentrations in the aeration tank. This technology was principally adopted to treat domestic or municipal wastewater i.e. low strength and colorless. Previous studies have reported that amongst conventional biological treatment technologies, activated sludge technology is not effective in degradation of azo compounds (Inyang et al., 2016; Sarayu and Sandhya, 2012).

Activated sludge treatment system is one of the most common aerobic type treatment that has been adapted since long and reliable in treating low strength municipal wastewater (Yavuz & Celebi, 2000). Figure 2.8 shows the schematic diagram of activated sludge process which consists of an aerobic process occurring in a biological aeration tank which is followed by a clarifier for the removal of flocs resulting from bacterial growth.



**Figure 2.8: Schematic diagram of Conventional Activated Sludge Process (Adopted from Yavuz & Celebi, 2000).**

Other common categories of aerobic biological treatment in addition to conventional activated sludge technology involves trickling filters and oxidation ditches which have been practiced since early years to treat municipal wastewater but not adequate for industrial wastewater due to its high strength and complex composition. Other aerobic biological treatment technologies have been shown in Figure 2.9.



**Figure 2.9: (a) Oxidation ditches biological treatment (b) Trickling filters involving the microbial growth media to treat wastewater (Adopted from Wu et al., 2006; (Jenssen et al., 2004).**

**Trickling Filters:** Trickling filters is the fixed bed bioreactor type working on a principle of aerobic conditions involving media for biofilm growth over which the wastewater is trickled throughout the process. These systems are compact but require high operational cost (Jenssen et al., 2004). Trickling filters are also known as bio-filters and the effluent coming out through filter consisting of low organic content.

**Oxidation Ditches:** Constructed wetlands or oxidation ditches have been used for the treatment of polluted water like storm water runoff from agriculture land. Oxidation ditches have been practiced in developing countries for in situ rural wastewater treatment (Wu et al., 2006).

#### 2.4.1.1.1 Aerobic Sequencing Batch Reactor (SBR)

SBRs are most commonly adopted in areas which are characterized by low variation wastewater flow patterns. The activated sludge type biological treatment consists of bacterial microbes in sludge which are responsible to provide treatment (Ahmed et al., 2017). The provision of oxygen in aerobic SBR to maintain microbial growth is the key consideration in achieving acceptable level of treatment (Radjenović et al., 2009). SBR technology is considered to be a distinct activated sludge process. They differ from conventional activated sludge process in terms of the components of treatment process. As conventional system requires multiple basins to carry out treatment process, but SBR combine all processes in single basin or biological tank. The schematic of working operation and stages of aerobic SBR are described in Figure 2.10.

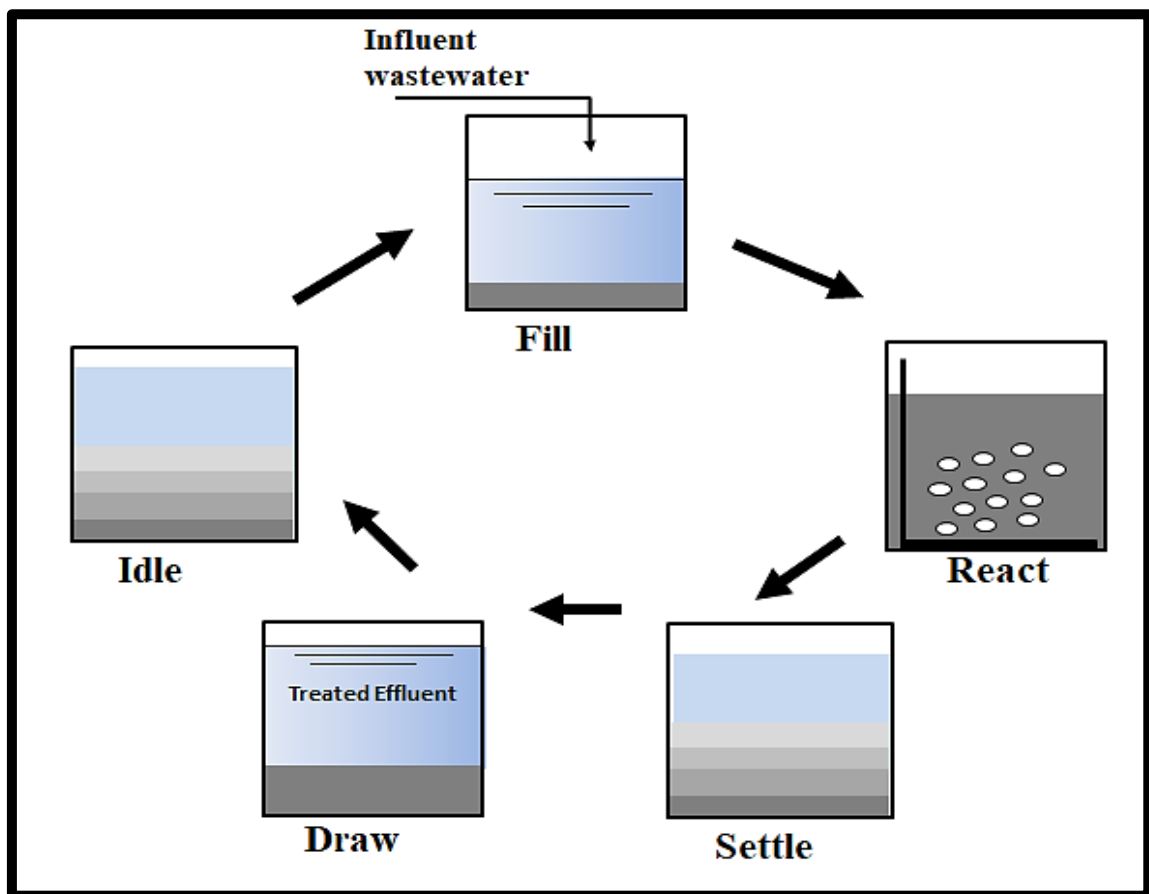
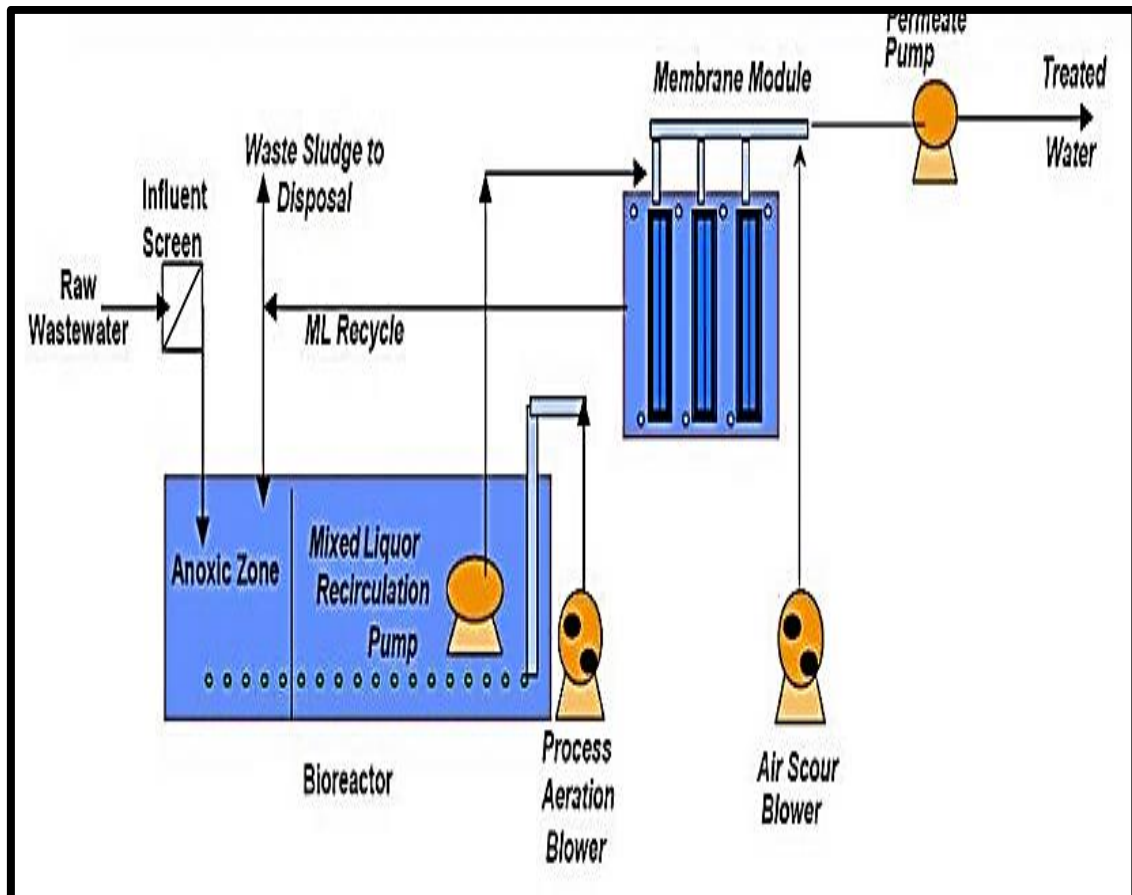


Figure 2.10: Phases of aerobic sequencing batch reactor operations cycle.

### 2.4.1.2 Aerobic Membrane Bioreactors (MBR)

MBRs, the most advanced and efficient treatment technology for treating wastewater are composed of two primary processes, combination of biological treatment and filtration as illustrated in Figure 2.11 (Judd, 2010; Yang et al., 2006).

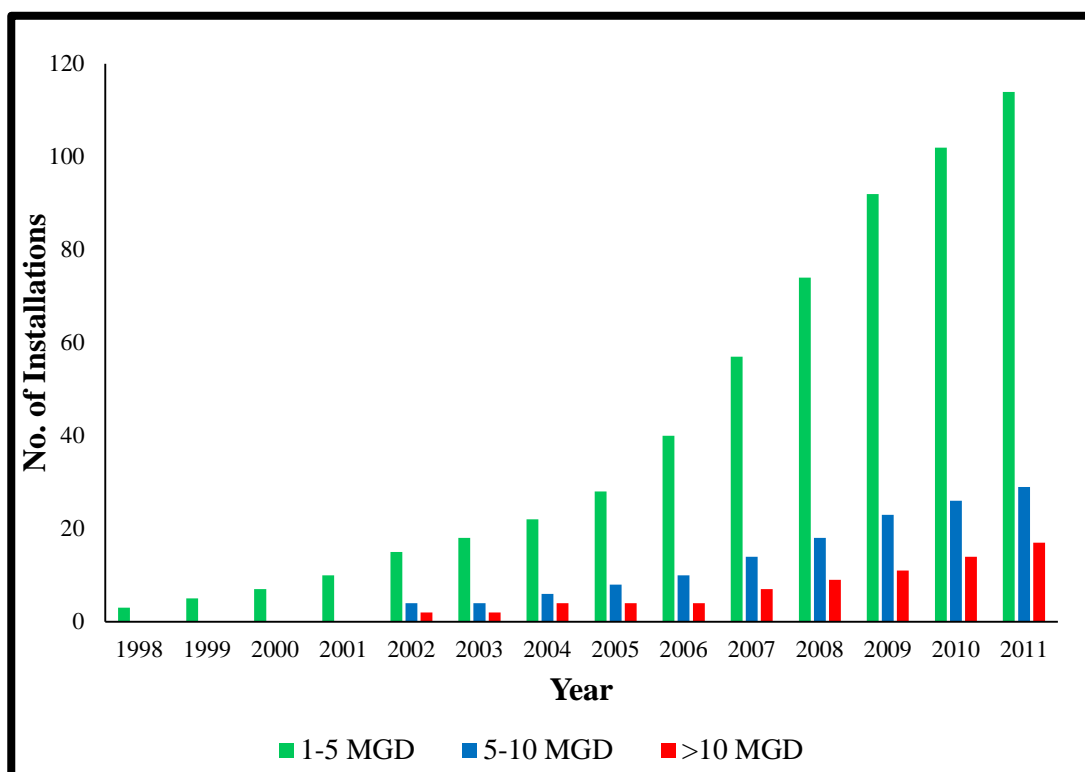


**Figure 2.11: Flow diagram for solid-liquid separation: MBR process provided with filtration process (Adopted from Judd, 2010; Yang et al., 2006).**

The effluent quality of MBR in terms of chemical oxygen demand (COD) is better than any conventional technologies due to the absorbance and enhanced degradation of colloidal compounds (Judd, 2010). With the advantages of MBR technology, there are also certain limitations but the potential of treatment and operational flexibility are perhaps the chief advantages that drag its importance of installation especially for the treatment of high strength wastewaters (Hirani et al., 2010). MBR technology professed novelty, supports the investment decisions for the treatment of wastewater as it



provides the enhanced beneficiaries of energy efficiency, improved operational procedures and longer warranty (Lesjean and Huisjes, 2008). The MBR adoption in developed countries is reported as of variable capacities because of their small footprint treatment processes and 80-95% nutrient reduction (Kraume et al., 2005). Worldwide, larger capacity installations of MBR are still in development of operation, manufacturing or design phase. Figure 2.12 shows that MBRs installation of varying capacity is increasing in Europe region since 1998 (Oppenheimer et al., 2012).



**Figure 2.12: Cumulative capacity installations of MBRs (Adopted from Oppenheimer et al., 2012).**

Membrane bioreactors are designed based on type of target pollutants to be eliminated from wastewater discharges. Numerous modelling studies have reported energy demands on dominant categories of MBR i.e. anaerobic and aerobic MBRs (Chan et al., 2009; Trzcinski and Stuckey, 2016). Despite of the limitations of MBRs, in Europe, the furthestmost practice of wastewater treatment is the MBR technology, especially for the treatment of industrial wastewater (Xiao and Roberts, 2010). The anaerobic MBR is

energy efficient as compared to aerobic configuration because of the recovery of biogas at mesophilic temperatures for wastewaters of strength 4-5 g COD/liters (Martin et al., 2011). In comparison to conventional wastewater treatment, COD removal in MBR can be achieved up to 96-99% (Bérubé, 2010) .

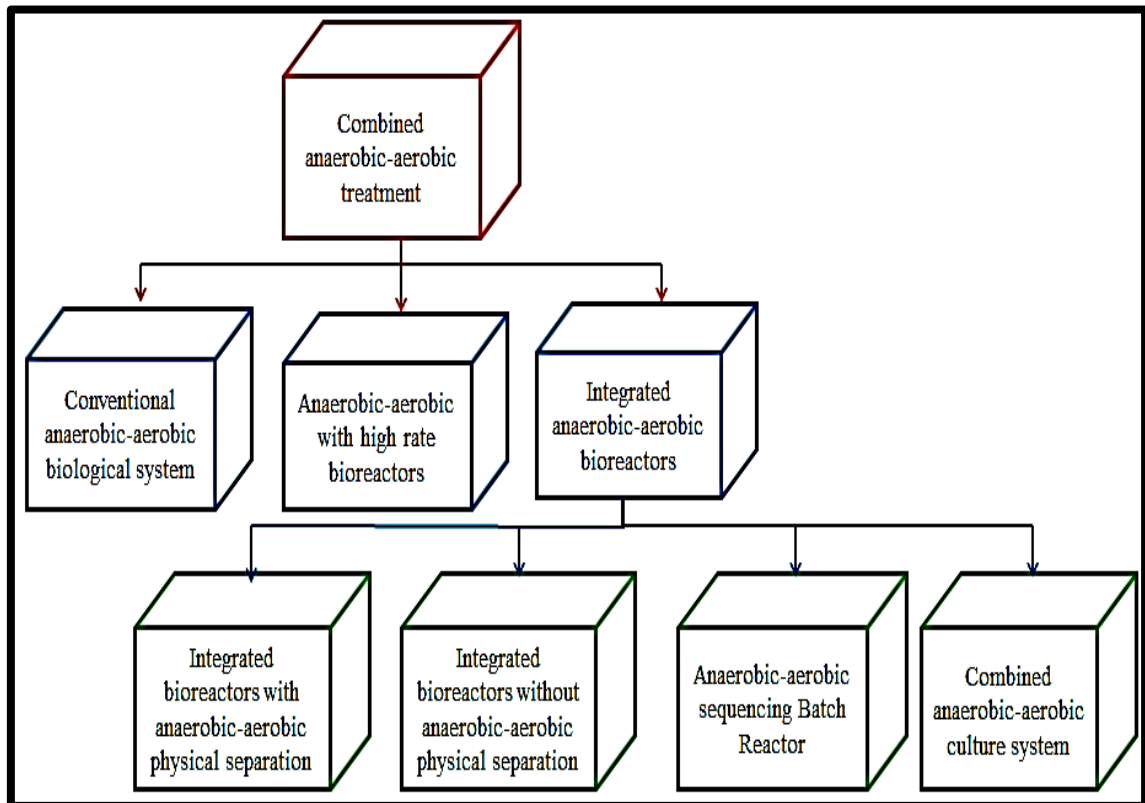
#### **2.4.2 Limitations of Aerobic Biological Systems**

- CAS is not acclaimed for treating textile wastewater as it involves high organic loading and dyes. Since aerobic bacteria are accountable for treatment in CAS, they are inefficient and incapable for degrading complex dye compounds (Oh et al., 2004).
- Due to its sensitivity to industrial wastewater, CAS requires high operational cost and large scale sludge disposal unit therefore not considered as an flexible technology.
- Membrane fouling in MBR is the adsorption of certain materials as a result of attachment and accumulation on the surface of membrane that increases the rate of hydraulic resistance (Simonič, 2013).

#### **2.5 Hybrid Biological Treatment Systems**

For the stringent regulations of textile wastewater discharge, MBR technology is an alternative technology to adopt, but come with certain limitations of operational and maintenance cost (Yigit et al., 2009). In view of the strict regulations and standards of industrial wastewater discharge, both aerobic and anaerobic treatment can be combined to achieve maximum degradation. The two phases of anaerobic- aerobic treatment eliminate the limitations of each treatment independently. For the actual color removal from textile industrial discharge, the continuous treatment system is composed of two stage treatment; anaerobic followed by aerobic reactor. Hybrid biological systems can

be combinations of various treatment technologies as shown in Figure 2.13. Aromatic amines that usually show complexity in degradation, when subjected to aerobic treatment after anaerobic stage, their removal efficiency surges up to 87%, which shows the potential effectiveness of coupled treatment technology (Işık and Sponza, 2008).



**Figure 2.13: Possible combinations for hybrid biological systems (Adopted from (Işık and Sponza, 2008)).**

### 2.5.1 Combined Anaerobic Baffled Tank and Activated Sludge Process

Previously, it was reported that the slaughterhouse wastewater production was subjected to treatment in combined anaerobic baffled reactor and aerobic activated sludge system in order to generate biogas production from meat processing industry (Bustillo-Lecompte and Mehrvar, 2017). The adoption of anaerobic treatment technology either in combination or individually, is considered as a green practice as an environmental protection initiative. The biogas production from high organic strength

industrial wastewater can balance out the operational cost of treatment technology with installation cost. To fulfill the industrial discharge requirement, the provision of aerobic treatment is of main concern in order to protect environmental resources.

### **2.5.2 Combination of Biological and Photocatalytic Treatment**

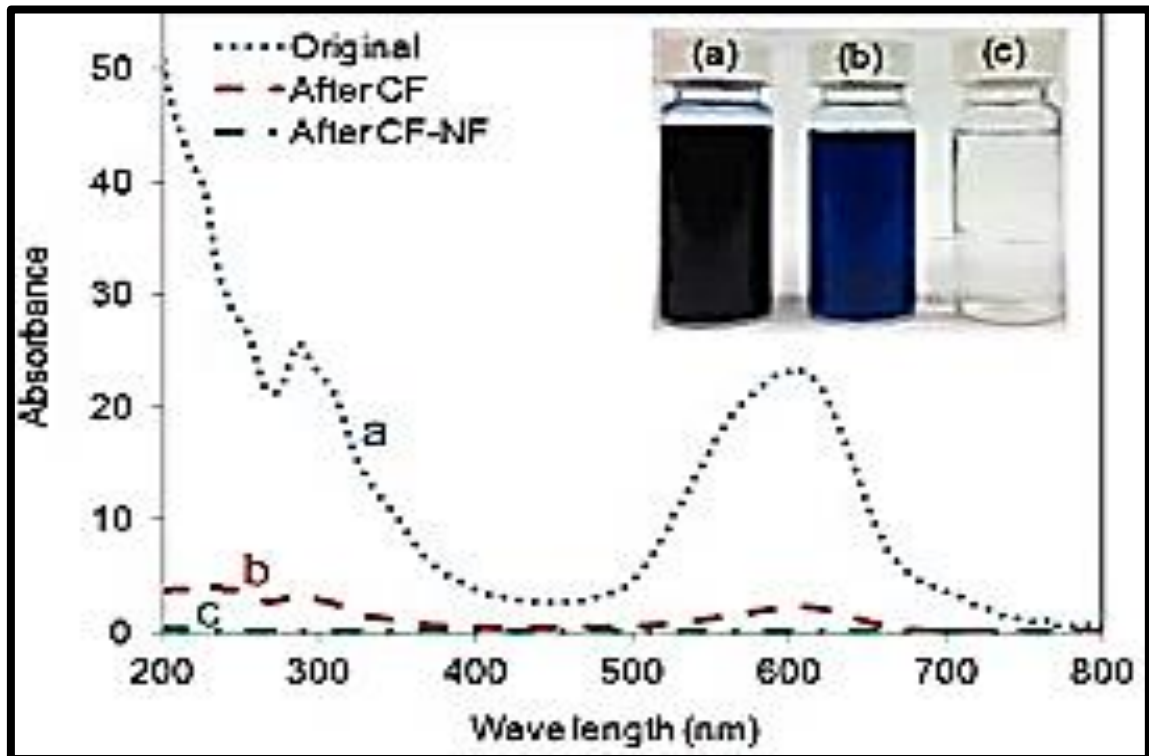
Combination of three bioreactors integrated in a sequence of anoxic-aerobic-anoxic followed by UV-TiO<sub>2</sub> photo catalysis was practiced in removing organics and toxic pollutants from industrial wastewater. This type of treatment was found to be appropriate for treating coke oven wastewater containing a variety of toxic organic and inorganic compounds like phenolic, aromatic hydrocarbons (Sharma and Philip, 2016). The physico-chemical treatment technologies though can be adopted to meet discharge standard requirements but are considered to be uneconomical and unsustainable for the treatment of complex composition industrial wastewater.

The application of anoxic-oxic processes is the provision of high wastewater treatment performance and economical due to naturally growing microbial activity. The provision of anaerobic processes in the combined treatment system is to degrade the refractory and inhibitory compounds into biodegradable intermediates resulting into maximum COD removal rate. The photocatalysis provides the removal of total carbon and total nitrogen effectively as the TiO<sub>2</sub>/UV light process or H<sub>2</sub>O<sub>2</sub>/UV light process has the ability to remove recalcitrant compounds present in wastewater such as pesticides and coloring dyes.

### **2.5.3 Combination of Coagulation/Flocculation and Nanofiltration**

Mostly, industries produce colored wastewater due to the presence of residual dyes that are aesthetically undesirable and harmful to the environment. Colored wastewater typically is generated from textile, tanning and paper industries. An experimental study

was performed on synthetic wastewater containing highly concentrated multiple dyes i.e. acid, basis and reactive dyes through combined technology of coagulation/flocculation and nanofiltration (Liang et al., 2014). The combined treatment technology involving nanofiltration as a post treatment lead to 100% dye removal as described in Figure 2.14.



**Figure 2.14: Effect of individual and combined technology on dye removal in complex colored wastewater (Adopted from Liang et al., 2014).**

Nevertheless, conventionally the typical arrangement adopted for textile wastewater treatment was UASB i.e. anaerobic treatment followed by activated sludge process i.e. aerobic treatment (Oh et al., 2004). The combined anaerobic-aerobic treatment has the following advantages which makes this hybrid technology of the greatest potential to treat textile wastewater proficiently. For pretreatment with anaerobic processes, all the primary sedimentation tanks or use of conventional activated sludge can be eliminated. The combined treatment system has the following advantages (Chernicharo, 2006).

1. For pretreatment with UASB, all the primary sedimentation tanks or use of conventional activated sludge may be eliminated.
2. Lower volumes of sludge from anaerobic system provide an ease of disposal of inclusive collective sludge.
3. Cost effectiveness and environmental feasibility also adds up in compensations.

The savings on operation and maintenance costs usually on an average 45-50% relative to conventional treatment systems (Pérez et al., 2013). Also, the consideration of both aerobic MBR and anaerobic MBR characteristics with respect to energy constraint, the possible sludge treatment and disposal depends on the operational procedures under solid retention time (SRT). In developed countries, an approach to treat industrial wastewater has been made by combining different biological and chemical wastewater treatment technologies.

# MATERIALS AND METHODS

This chapter is focused on the methodology followed throughout the study which has been conducted in Water and Wastewater Laboratory, IESE-NUST. The research was carried out in two phases as a response of which the results were obtained.

### 3.1 Establishment of Laboratory Scale Setup

A bench scale setup of hybrid anaerobic-aerobic system was installed at laboratory scale to perform the proposed objectives of the study elaborated in Figure 3.1. The volume of each reactor was designed to be 6.5 liters consisting of 6 liters of working volume. The acclimatized sludge for anaerobic CSTR was collected from the pre-installed batch study conducted at laboratory scale, while for the aerobic SBR the acclimatized sludge was taken from the NUST Membrane Bioreactor (MBR) Plant at NUST campus, Sector H-12. The initial concentration of sludge in both reactors was kept constant at 8000 mg/L throughout the study. The solid retention time (SRT) for anaerobic reactor was set to be infinite, but for aerobic SBR, the SRT for maintained at 20 days by wasting 300 ml of sludge each day. System specifications and operational parameters of the lab scale hybrid biological system are described in Table 3.1. The sludge wastage was calculated from the following formula.

$$\text{Sludge discharge (ml)} = \frac{\text{Working volume of reactor (V)}}{\text{SRT (days)}} \quad (1)$$



**Figure 3.1: Laboratory scale setup of hybrid anaerobic-aerobic biological system.**



**Table 3.1: Operating conditions of hybrid anaerobic CSTR-aerobic SBR.**

OPERATING PARAMETERS	UNITS	HYBRID SYSTEM	
		Anaerobic CSTR	Aerobic SBR
Reactor Volume	Liter (L)	6.5	6.5
Working Volume	Liter (L)	6	6
Hydraulic Retention Time (HRT)	Hours (hr)	48	6
Solid Retention Time (SRT)	Days (d)	Infinite	20
MLSS	mg/L	6000-8000	6000-8000
Ratio MLSS/MLVSS		0.6-0.7	0.6-0.7
Ratio Alkalinity to VFA		0.3-0.4	-
pH		6.8-7.2	6.8-7.2

## **3.2 Operation of Hybrid Biological System**

### **3.2.1 Anaerobic Continuously Stirred Tank Reactor (CSTR)**

Anaerobic CSTR was operated with adjusted continuous mixing, provided with mechanical mixer (STIR PAK-HDM-5000425) at rotation speed of 180 to 270 rpm. Mixing was provided with optimized mixing and relaxation mode, 3 hours 30 minutes by setting up timers. The sludge concentration of 6000 to 8000 mg/L was also maintained throughout the study by no sludge wastage, with optimized HRT of 48 hours. Heating rod was set up in the reactor in order to maintain the mesophilic temperature between range of 30-35°C, and the required temperature was monitored throughout by temperature sensors. Relay unit with water level controller was used to maintain the level of water in the bioreactor and fed water stored in storage tank from where it was pumped to the overload tank. For the treatment of textile wastewater subjected to CSTR, the influence of bacterial activity fluctuates the performance i.e. increase in wastewater loading rates provides an average of 90% COD removal in addition to the color reduction (Khelifi et al., 2009).

### **3.2.2 Aerobic Sequence Batch Reactor (SBR)**

In aerobic SBR, air was supplied with the help of air compressor (HEALEA ACO-208) at a rate of 8 L/minutes for coarse bubbling throughout the reactor volume to maintain the concentration level of dissolved oxygen (DO) for the microbial growth, organic degradation and to create the turbulence in order to avoid the dead zones formation in the bottom of the bioreactor. Wastewater after anaerobic treatment flowed into the aerobic SBR with the help of solenoid valve adjusted to control the flow from anaerobic reactor to aerobic reactor. In aerobic SBR, the sludge concentration was kept constant between ranges of 6000-8000 mg/L by adjusting the SRT of 20 days, with HRT of 6 hours provided for polishing the resulting effluent.

## **3.3 Wastewater Composition**

The study was carried out in two phases. During 1<sup>st</sup> phase a synthetic textile wastewater treatment performance was evaluated and optimized for setting a reference study conducted in Phase II, in which real textile wastewater was run through the hybrid system for its treatment performance.

### **3.3.1 Preparation of Synthetic Wastewater**

In Phase I, the initial lab scale study was performed for the performance evaluation and optimization of the system performance when subjected to high strength synthetic wastewater. Synthetic textile wastewater of strength of 3000 mg/L COD was prepared while maintaining COD: N :P at 100:10:1 as show in Table 3.2.

**Table 3.2: Formula of synthetic wastewater of high strength i.e. COD = 3000 mg/l.**

CHEMICALS	UNIT	CONCENTRATION
Hydrated D-Glucose	mg/L	3000.00
Ammonium Chloride (NH <sub>4</sub> Cl)	mg/L	1146.00
Potassium Hydrogen Phosphate (KH <sub>2</sub> PO <sub>4</sub> )	mg/L	143.10
Calcium Chloride (CaCl <sub>2</sub> )	mg/L	29.19
Magnesium Sulfate (MgSO <sub>4</sub> .7H <sub>2</sub> O)	mg/L	9.73
Ferrous Chloride (FeCl <sub>3</sub> )	mg/L	1.00
Sodium Hydrogen Carbonate (NaHCO <sub>3</sub> )	mg/L	500.00
Cobalt Chloride (CoCl <sub>2</sub> )	mg/L	0.10
Zinc Chloride (ZnCl <sub>2</sub> )	mg/L	0.10
Nickle Chloride (NiCl <sub>2</sub> )	mg/L	0.10
Mixed Dyes - Cibacron Yellow - Cibacron Blue - Methylene Blue	mg/L	15.00

The formula of the synthetic wastewater involved macronutrients and micronutrients like Glucose (C<sub>6</sub>H<sub>12</sub>O<sub>6</sub>), Ammonium Chloride (NH<sub>4</sub>Cl), Potassium hydrogen phosphate (KH<sub>2</sub>PO<sub>4</sub>), Calcium Chloride (CaCl<sub>2</sub>), Magnesium Sulfate (MgSO<sub>4</sub>), Ferrous Chloride (FeCl<sub>3</sub>) and Sodium Hydrogen Carbonate (NaHCO<sub>3</sub>) to maintain the pH range of 6.8 to 7.2 as shown in Figure 3.2 (b). Sodium hydrogen carbonate concentration was adjusted to maintain pH of 6.8 – 7.2 which was monitored throughout the experiment. Mixture of dyes was added to the wastewater in variable concentrations, starting from 5 mg/L each, allowing the better acclimatization conditions to the sludge.

### 3.3.2 Characterization of Real Textile Wastewater

In Phase II, real textile wastewater was treated through hybrid biological system, which was optimized in Phase I by running synthetic textile wastewater. Phase I study was used as a reference study for the study of real textile wastewater treatment.

**Table 3.3: Characteristic analysis of real textile wastewater.**

PARAMETERS	UNIT	CONCENTRATION
pH		9.5-11.2
COD	mg/L	2200-2800
TDS	mg/L	1870
Turbidity	NTU	371.5
Orthophosphate	mg/L	22.27
Total Nitrogen	mg/L	250-300
Chlorides	mg/L	745.5
Dyes Mixture		Indefinite (Disperse, Reactive, Pigment, Vat)
Conductivity	mS/cm	3.27

Textile wastewater is classically composed of organic matter, suspended solids, dyes and numerous other chemicals like polycyclic aromatic hydrocarbons, solvents, detergents, recalcitrant compounds and heavy metal ions (Miah, 2013). Composite samples of real textile wastewater were collected from Koh e Noor textile industry located in Rawalpindi and analyzed for characterization in Water & Wastewater laboratory, IESE-SCEE, NUST as shown in Figure 3.2 (a). The samples from industry were collected after primary clarification twice a week.

Different analytical techniques were adopted in order to characterize the influent real textile wastewater. The pH of real textile wastewater was measured to be very high and was maintained between 6.8 to 7.2 adding chemicals like hydrochloric acid (HCl). The

analysis of every sample was done in order to calculate the average concentration of various wastewater parameters.



**Figure 3.2: (a) Textile wastewater composite samples (b) Chemicals used in synthetic wastewater preparation.**

### **3.4 Sludge Acclimatization**

Anaerobic treatment systems are recommended to treat high strength industrial wastewater at mesophilic temperatures (McHugh et al., 2004). For anaerobic CSTR, the sludge was collected from NUST MBR Plant at NUST campus and was adapted to anaerobic conditions for duration of two months with synthetic wastewater and real textile wastewater for both phases. At the end of acclimatization, MLSS was found within the range of 6000 to 8000 mg/L along with the production of biogas. Biogas

production symbolizes the complete acclimatization of sludge anaerobically. Additionally, sludge color changes from “golden brown” to “black”. The acclimatization study was carried out in pre-installed batch study at laboratory scale.

For the aerobic SBR, sludge from aerobic MBR was acclimatized at laboratory scale by providing glucose and continuous aeration. Sludge color was maintained as “golden brown” which indicates the presence of active aerobic microorganisms. This aerobic acclimatization continued for duration of 2-3 weeks.

### **3.5 Hybrid System Operational Setup**

Two biological reactors, anaerobic continuously stirred tank reactor (CSTR) and aerobic sequencing batch reactor (SBR) were coupled in a hybrid biological system. Both the reactors were filled with distilled water and operated to check the water leakage. Anaerobic setup was also subjected under nitrogen sparging test in order to insure the anaerobic conditions in a reactor. Schematic of installed setup is shown in Figure 3.3. The hybrid system working operation involves gravity flow of wastewater from feed tank was driven into anaerobic SBR with the help of solenoid valve and level sensor. The anaerobic CSTR was equipped with mechanical mixer for continuous mixing of anaerobic sludge and influent wastewater at 200 rpm speed. The hydraulic retention time (HRT) in anaerobic reactor was set at variable values to check the performance capability of anaerobic process treating textile wastewater. After providing anaerobic treatment in SBR the permeate was streamed to aerobic SBR by gravity flow. Aerobic SBR was provided with proper aeration provided with cylindrical diffusers connected to air compressor. The effluent was decanted from aerobic system with the help of solenoid valve controlled by digital timers. Operation of hybrid system

involved feeding, reaction, mixing/aeration, settling and decanting controlled via digital timers.

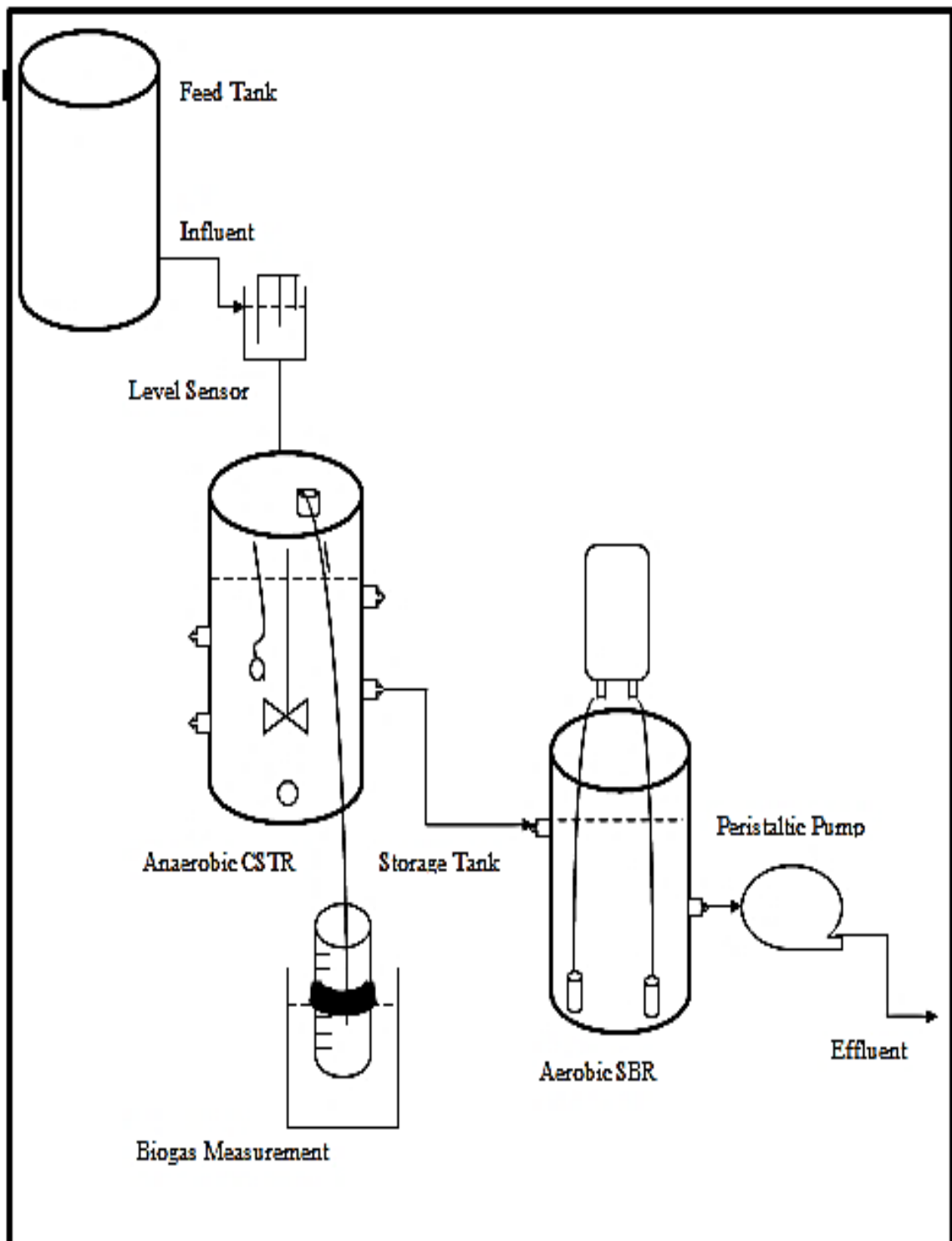


Figure 3.3: Schematic of hybrid anaerobic CSTR-aerobic SBR system.

Anaerobic and aerobic system was operated at HRT of 48 and 6 hours, respectively. Solid retention time (SRT) in anaerobic system was adjusted to infinite to maintain MLSS in laboratory scale reactor, but for aerobic system SRT was adjusted to 20 days.

### **3.5.1 Phase I: Application of Synthetic Wastewater**

- The system was fed with synthetic wastewater at various HRTs i.e. 12, 24 and 48 hours in order to evaluate the performance of system in treating synthetic wastewater.
- Synthetic wastewater was prepared in laboratory as presented in Table 3.2.
- This synthetic wastewater was subjected to various analytical techniques, in order to determine the initial characteristics like COD, pH, TKN, and dyes concentration.
- Wastewater was filled into the feed tank after performing its physical and chemical analysis.
- Reactor Feeding: The wastewater was allowed to enter into the anaerobic CSTR provided with continuous mixing and wastewater level was maintained with the help of relay and level sensors provided in inlet chamber. The samples from CSTR were collected at variable HRTs in order to determine the removal efficiency of anaerobic process by performing sample analysis.
- After anaerobic system optimization, the wastewater was then subjected to aerobic SBR, coupled with anaerobic CSTR, in order to improve the removal efficiency.
- The optimization of aerobic system was also performed at various HRTs.
- Hybrid system was optimized after performance evaluation for synthetic wastewater, which was set as a reference study for performing real textile wastewater treatment.



### **3.5.2 Phase II: Application of Real Textile Wastewater**

- The system was fed with real textile wastewater every 48 hours at a uniform organic loading rate.
- Composite samples were collected from textile industry and subjected to laboratory analysis as presented in Table 3.3.
- Textile wastewater was run into the hybrid biological system which was optimized in Phase I.
- Reactor feeding and decanting steps were followed similar to the Phase I.

### **3.6 Experimental Analytical Techniques**

Hybrid system was operated for a time span of 242 days involving two phase study i.e. synthetic and real textile wastewater. Throughout the study, performance of reactors was evaluated for organics and nutrients removal from textile wastewater. For system performance, different tests of samples were conducted to determine the removal efficiencies of various parameters involved in treatment performance.

The analytical analysis are presented in Table 3.4 carried out throughout the study in experimental phases i.e. Phase I and Phase II. The analyses protocol involved the extent of various analytical techniques from Standard Methods for the Examination of Water and Wastewater (APHA et al., 2012).

**Table 3.4: Performance testing for effluent from anaerobic CSTR & aerobic SBR.**

PARAMETERS	ANALYTICAL TECHNIQUES	TREATMENT METHODS	
		AnCSTR	AeSBR
Chemical Oxygen Demand (COD)	Close Reflux Titrimetric Method	✓	✓
Mixed Liquor Suspended Solids (MLSS)	Oven Dry Method	✓	✓
Mixed Liquor Volatile Suspended Solids (MLVSS)	Muffle Furnace Drying Method	✓	✓
Ratio MLSS/MLVSS		✓	✓
Alkalinity	Titration Method	✓	✗
Volatile Fatty Acids (VFA)	Distillation Method	✓	✗
Ratio VFA/Alkalinity		✓	✗
Total Kjeldahl Nitrogen (TKN)	Standard Macro Kjeldahl Method	✓	✓
Color	Spectrophotometric – Single Wavelength Method	✓	✓
Ph	Electrometric Method	✓	✓
Biogas	Water Displacement Method	✓	✗

### 3.6.1 Analytical Effluent Quality Analysis

#### 3.6.1.1 Chemical Oxygen Demand (COD)

COD of samples i.e. both aerobic & anaerobic, was performed by close reflux method in which following steps were followed:

- COD vial was prepared by adding 2.5 ml volume of sample, 1.5 ml of 0.0166 M  $K_2Cr_2O_7$  and 3.5 ml volume of  $H_2SO_4$  reagent

- Digestion: These vials were then placed in preheated COD digester for a duration of 2 hours at 1500 °C temperature
- After digestion, vials were allowed to cool down to room temperature
- Titration: The vials were titrated against 0.25 M ferrous ammonium sulfate (FAS) using ferroin indicator till the end point reached i.e. yellow to brown.
- COD of sample was calculated by using following formula

$$\text{Chemical Oxygen Demand (mg/l)} = \frac{A - B \times 8000 \times M}{\text{Volume of sample (ml)}} \quad (2)$$

where

A = volume (ml) of FAS used for Blank

B = volume (ml) of FAS used for sample

M = molarity of FAS

8000 = conversion factor

### 3.6.1.2 Alkalinity

Alkalinity test of anaerobic samples only, was performed using titration method in which following procedural steps were followed:

- Alkalinity test of samples from anaerobic CSTR was performed using titration method.
- A volume of 20 ml sample was collected from anaerobic CSTR into a beaker
- pH of the sample was measured using pH meter and noted
- Sample was then titrated against H<sub>2</sub>SO<sub>4</sub> till the pH drop to 3.0 was observed
- At pH 3, volume of H<sub>2</sub>SO<sub>4</sub> used was noted and calculated by using given formula:

$$\text{Alkalinity (mg/l)} = \frac{(\text{Final} - \text{Initial}) \times N \times 50000}{\text{Volume of sample (ml)}} \quad (3)$$

where

N = Normality of H<sub>2</sub>SO<sub>4</sub> consumed

Final-Initial = Volume of acid consumed (ml)

50000 = conversion factor

### 3.6.1.3 Volatile Fatty Acids (VFA)

VFA tests were also performed for anaerobic samples only, and following procedure was adopted throughout the study.

- VFA test for anaerobic samples was performed using distillation method.
- Sample collected was first subjected to distillation and then further to titration
- The sample subjected to alkalinity test was further heated by placing them on a heating plate.
- The sample was removed from the plate when temperature reached 60-70°C.
- Sample was cooled down to room temperature and their pH was measured and noted.
- Titration: Sample was titrated against 0.02 N NaOH till the required pH reached 6.5. Volume of NaOH used was noted and VFAs were calculated by using following formula.

$$\text{Volatile Fatty Acids (VFA)} = \frac{(\text{Final} - \text{Initial}) \times N \times 50000}{\text{Volume of sample (ml)}} \quad (4)$$

where

Final-Initial = Volume of NaOH consumed (ml)

50000 = conversion factor

#### **3.6.1.4 Ratio VFA/Alkalinity**

Ratio of VFA to alkalinity was determined which is considered as an important parameter that stimulates the bio gas production. The ideal range for this ratio was calculated as 0.3-0.4, which was maintained throughout the experiment. Following formula was used to calculate the ratio.

$$Ratio = \frac{Volatile\ Fatty\ Acids\ (mg/l)}{Alkalinity\ (mg/l)} \quad (5)$$

#### **3.6.1.5 Color**

Color removal from dyebath discharge may be effectively achieved by using anaerobic biologically degradable microorganism consortium (Petzoldt, 2014). Color removal in % was calculated for both phases by following procedural steps:

- No. of samples were collected from both systems at various HRTs.
- Influent and affluent sample was collected and placed into the centrifuge for 30 minutes at 3000 rpm.
- Absorbance of pre-programmed calibrated Pt-Co spectrophotometer has a broad maximum Pt-Co absorbance within the wavelength range 450 – 456 nm.
- Initially, spectrophotometer vial was filled with distilled water to zero the instrument error.
- Vial was rinsed with influent sample

- The centrifuged influent sample was diluted 4 times and then placed into the spectrophotometer. An absorbance value was recorded
- Similarly, the centrifuged effluent sample, diluted 4 times, was placed into the spectrophotometer and absorbance value was recorded.
- Color removal efficiency was calculated by using following formula:

$$Color (\%) = \frac{Influent - Effluent}{Influent} \times 100 \quad (6)$$

- Procedure was repeated for no. of samples collected throughout the study.

### **3.6.1.6 Total Kjeldahl Nitrogen (TKN)**

TKN removal was evaluated for both anaerobic and aerobic samples. Standard Macro Kjeldahl method was adopted to calculate TKN removal by following procedural steps.

- Digestion: Sample was collected from the system and the protocol followed for digestion was addition of 100 ml sample diluted to 300 ml volume, 50 ml digestion reagent.
- Sample was placed into the fume hood for 45 minutes to carry out digestion process till the crystal clear liquid left in the flask.
- Kjeldahl Apparatus: Digested sample was then cooled down to room temperature and its pH was observed by using pH meter.
- NaOH-Na<sub>2</sub>SO<sub>3</sub> reagent was added to the flask till pH reached 9.5 and diluted to 250 ml in conical flask.
- The sample was placed into the Kjeldahl apparatus, with the reagent of 50 ml boric acid in a separate beaker for the collection of ammonia.

- The sample was heated and ammonia gas started to collect in boric acid solution and stopped till the volume of boric acid reached 50 ml to 250 ml with an endpoint of purple to green.
- Titration: Ammonia containing boric acid was titrated against 0.02 N H<sub>2</sub>SO<sub>4</sub> and TKN value was calculated by using following formula.

$$\text{Total Kjeldahl Nitrogen (mg/l)} = \frac{A - B}{\text{Sample vol (ml)}} \times 280 \quad (7)$$

where

A = ml volume of H<sub>2</sub>SO<sub>4</sub> used for sample (final – initial)

B = ml volume used for blank

280 = conversion factor

### **3.6.2 Sludge Characterization**

#### **3.6.2.1 Mixed Liquor Suspended Solids (MLSS)**

System MLSS and MLVSS for both reactors were quantified and evaluated thrice a week. MLSS test for both anaerobic and aerobic sludge samples was conducted throughout the experiment by following standard procedure.

- At first, drying of whattman filter paper was done at a temperature of 105°C for duration of 30 minutes in order to remove moisture from filter paper if any.
- After drying, filter paper was placed in desiccator for cooling it down at room temperature. Weight of filter paper was measured using mass balance equipment and noted.
- Filtration: Filter paper was subjected to filtration assembly.

- Sample of volume of 10 ml was collected from both reactors separately i.e. during mixing in anaerobic CSTR and during aeration in aerobic SBR to obtain uniform sludge samples.
- Collected volume of sample was poured onto the filter paper fitted on filtration assembly till the water drained out.
- Filter paper was removed from assembly consisting the sludge cake and then placed into the oven for 1 hour at 105°C.
- After 1 hour, filter paper was weighed again after it cooled down to room temperature in a desiccator.
- MLSS was calculated by using the following formula:

$$\text{Mixed Liquor Suspended Solids (mg/l)} = \frac{A - B \times 1000000}{\text{Sample vol (ml)}} \quad (7)$$

where

A = weight (g) of filter paper + residue after drying at 105°C

B = weight (g) of filter paper

### 3.6.2.2 Mixed Liquor Volatile Suspended Solids (MLVSS)

- Residue containing filter paper obtained in MLSS calculation was further placed into the muffle furnace for 30 minutes at 550°C.
- Filter paper was weighed again, after cooling it down to room temperature.
- MLVSS was calculated by using following formula:

$$\text{MLVSS (mg/l)} = \frac{A - B \times 1000000}{\text{Sample volume (ml)}} \quad (8)$$



where

A = weight (g) of filter paper + residue after drying at 105°C

B = weight (g) of filter paper+ residue after drying at 550°C

### 3.6.2.3 Ratio MLVSS/MLSS

Ration of MLVSS to MLSS was calculated to determine the percentage of biomass present in sample of sludge taken. Ideally, the value of MLVSS/MLSS ratio for both anaerobic and aerobic systems is 0.6 – 0.8.

$$Ratio = \frac{MLVSS (mg/l)}{MLSS (mg/l)} \quad (8)$$

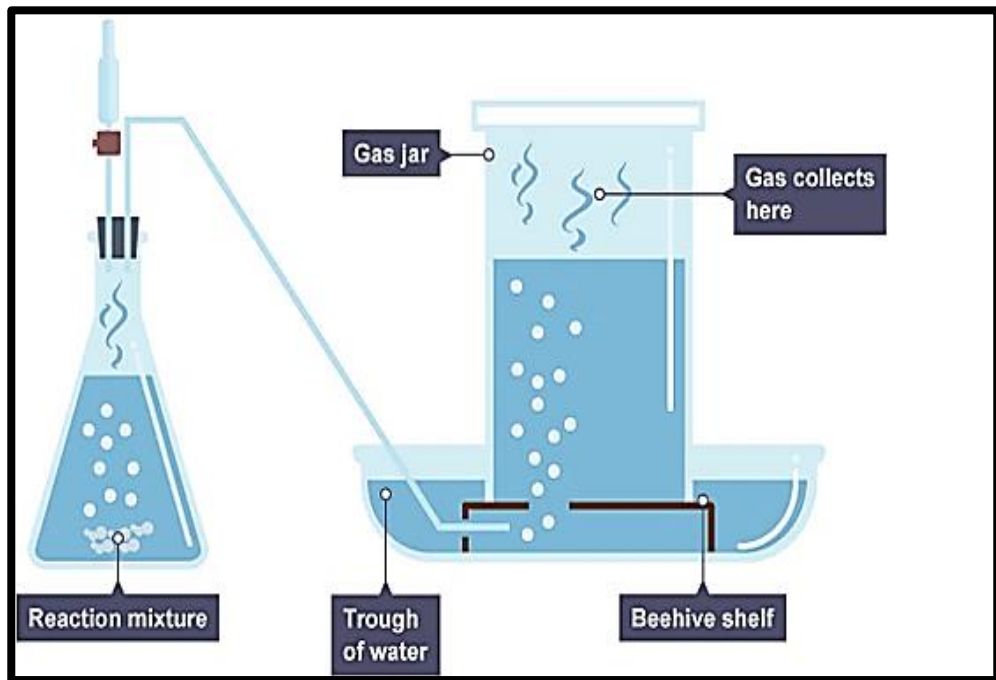
### 3.6.3 Biogas Production Analysis

Biogas production observed in anaerobic CSTR was measured by water displacement method by setting up the equipment with anaerobic CSTR. Figure 3.4. illustrates the water displacement method to measure biogas from system.

The measuring procedure followed during study is as follows:

- An acrylic tank was filled with distilled water along with the addition of sodium chloride (NaCl) in order to remove collected gas absorbed in water.
- The one open ended column connected with the anaerobic CSTR was dipped into the water tank and filled with distilled water, with the valves closed.
- The measurement of gas was taken when gas volume (ml) was allowed to enter the column replacing volume of water.
- Measurement procedure was repeated till the gas production stopped.

- Biogas production was measured on daily basis throughout the experimental study.



**Figure 3.4: Biogas measurement equipment by Water Displacement Method (Adopted from Sem & Demirer, 2003).**

# **RESULTS AND DISCUSSION**

In hybrid system, both coupled bioreactors were fed with the acclimatized anaerobic and aerobic sludge in each reactor separately, having initial MLSS concentration 7000 mg/l. Before and after the application of synthetic and real textile wastewater to the hybrid biological system installed at IESE Wastewater laboratory, the wastewater physiochemical and sludge parameters were analyzed on the basis of which the system performance efficiencies were calculated. In Phase I study, performance of synthetic industrial wastewater of strength of COD = 3000 mg/L was studied under its application to the hybrid system while in Phase II, study of real textile wastewater was conducted in assessing its treatment by hybrid biological system.

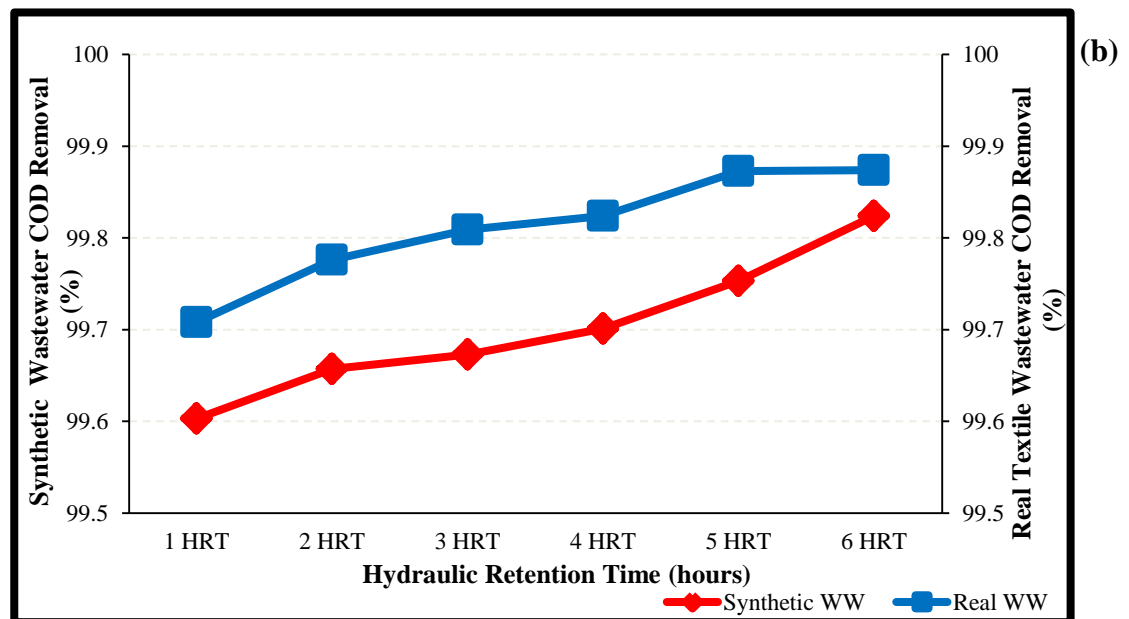
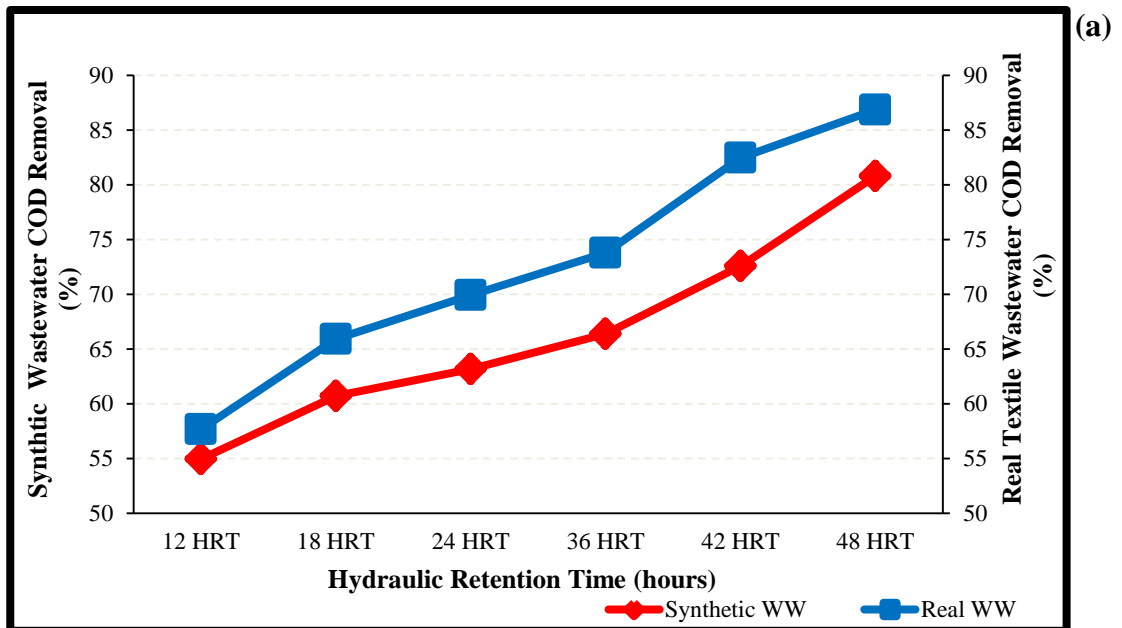
## **4.1 Treatment Performance Analysis**

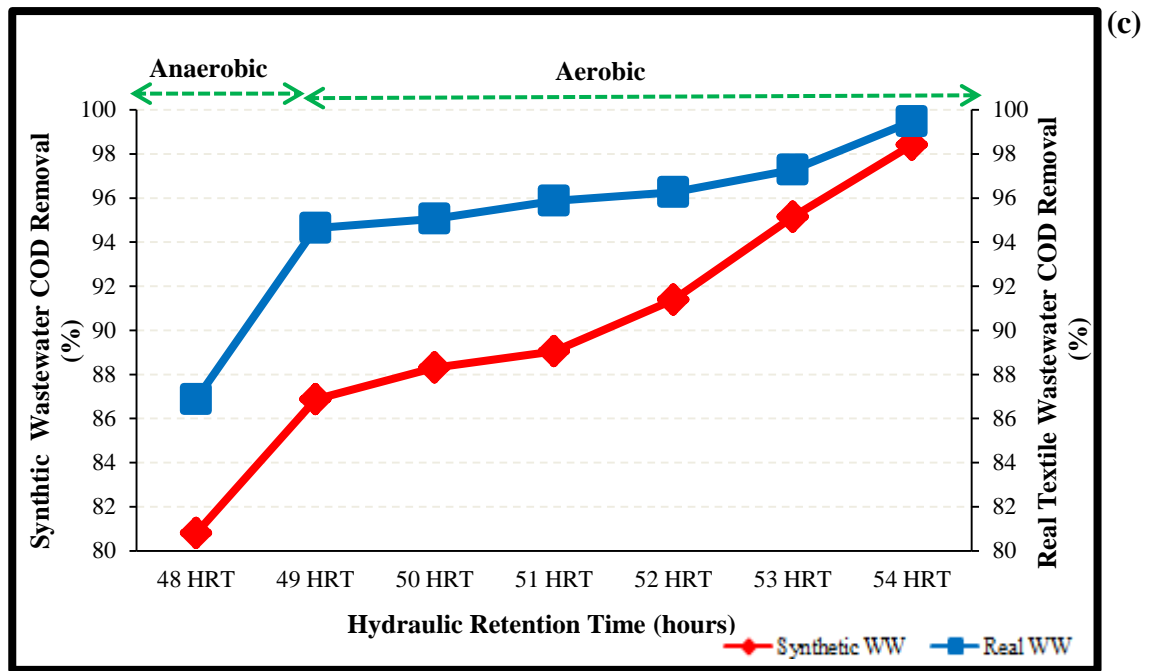
The treatment performance of hybrid system was observed during experimental phases of synthetic and real textile wastewater treatment.

### **4.1.1 Effect on Chemical Oxygen Demand (COD)**

Typically, for textile wastewater treatment, several approaches of chemical treatment methods like coagulation process have been practiced for the effective degradation of organic matter, but the application of these methods is restricted to industrial wastewater of high organic strengths owing to the requirement of large investment on chemical cost, which is considered as a non-affordable option for developing countries like Pakistan. COD removal was examined at different stages of working hybrid system in order to analyze the performance of coupled bioreactors individually. The COD

parameter was examined during the Phase I and phase II study till the system optimization conditions were achieved.





**Figure 4.1: (a) Trends of COD removal for synthetic and real textile wastewater, when subjected to anaerobic treatment only at variable HRTs. (b) Trends of COD removal for synthetic and real textile wastewater when subjected to aerobic SBR (c) COD removal trends for synthetic and textile wastewater when subjected to hybrid anaerobic -aerobic operating at variable HRTs.**

The COD profile during whole experimental study in which synthetic and real textile wastewater fed in two phases of hybrid anaerobic- aerobic system is shown in Figure 4.1. Figure 4.1 (a) shows the COD removal profile at different HRTs in anaerobic CSTR system individually due the action of methanogenic and syntrophic anaerobic microbial species. In anaerobic process, at 48 HRT, the maximum organic degradation takes place which shows this is the optimized HRT for anaerobic CSTR in treating textile wastewater. Figure 4.1 (b) describes the COD removal trend, when anaerobically treated permeate was subjected to aerobic SBR for final polishing. Aerobic treatment at variable HRTs showed that the maximum COD removal was found in aerobic process at a condition of 6 HRT provision to the system. Also the percentage of COD removal by hybrid anaerobic-aerobic system at variable HRTs in treating synthetic and real textile wastewater is shown in Figure 4.1 (c). The advantage of aerobic treatment in sequence with anaerobic is to polish the wastewater in terms of its COD removal up to

the level of 99 % by further running system at 6 HRT aerobically as the system efficiency of anaerobically pretreated wastewater increases by aerobic polishing. Figure 4.1 inclusively describes the positive outcomes that anaerobic CSTR can give the 80 % COD removal in case of synthetic run at an optimized condition of 48 HRT and 87% treatment efficiency of real textile wastewater when subjected to the optimized conditions evaluated in Phase I. The provision of aerobic treatment operating at variable HRTs of 1, 2, 3, 4, 5 and 6 hours combined with anaerobic pretreatment is to polish the anaerobic permeate. It is clear that the treatment in both phases by providing combined anaerobic-aerobic treatment at variable HRTs increases the system efficiency up to the level of 98 % and 99.5 % for synthetic and real wastewater, respectively. Therefore, the optimum HRT for hybrid system in treating real textile wastewater is inferred as 54 HRT for maximum COD removal.

#### **4.1.2 Effect on Color Removal**

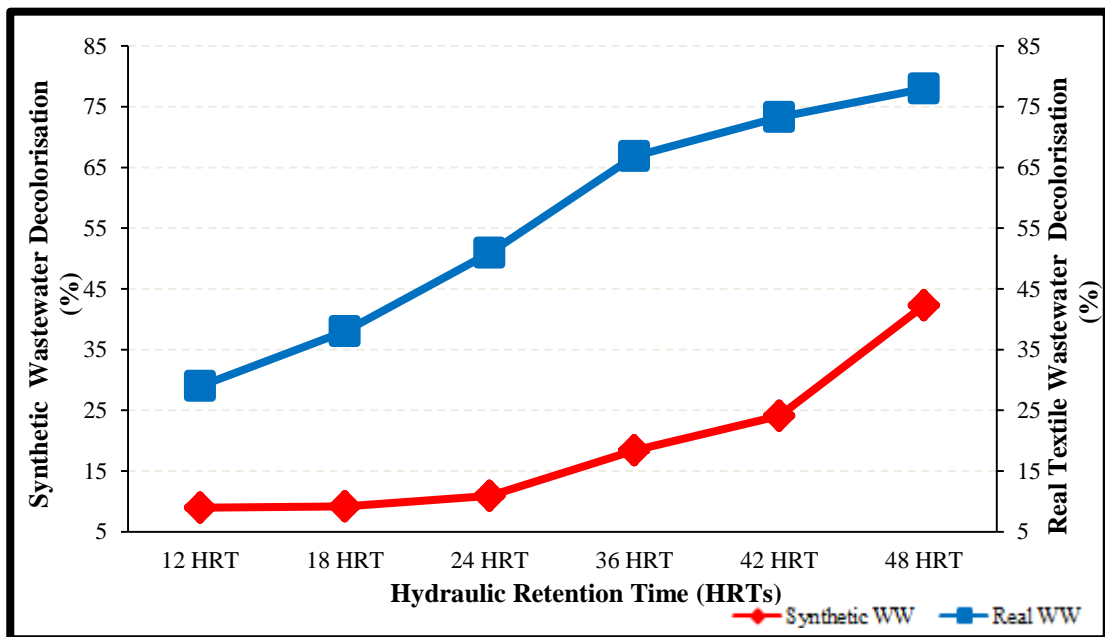
The function of anaerobic microbial species vary when exposed to different levels of pollutants allow their adaptability to the degradation of toxic colored compounds (Khan et al., 2013). The results for decolorisation of textile wastewater are described below illustrating the applicability of hybrid system in treating colored wastewater. During hybrid system operation, the anaerobic phase is the main contributor dealing color removal constraint.

##### **4.1.2.1 Decolorisation in Anaerobic Process**

The maximum efficiency in respect to the color removal parameter during acclimatization of the activated sludge in this experimental study was 80% approximately which is an indication of tremendous microbial adaptability conditions. The purpose of anaerobic CSTR is to maximize the decolorisation efficiency in

addition to the COD removal, lowering the cost of aeration in sequenced activated sludge process. The presence of non-biodegradable complex compounds present in textile wastewater impart color to the wastewater which can be treated anaerobically by slow degradation process (Lee et al., 2005).

Figure 4.2 describes that with the increase in HRT, color removal efficiency of anaerobic microbial community increases and the maximum removal is at 48 HRT. After achieving 78.4% color removal at 48 HRT, the anaerobic decolorisation remains stable because of complexly degradable by products of dyes in case of real textile wastewater. Color removal by aerobic microbial species does not add much value to decolorisation relative to the anaerobic CSTR.



**Figure 4.2: Decolorisation percentage removal trend of synthetic and real textile wastewater in anaerobic CSTR at variable HRTs.**

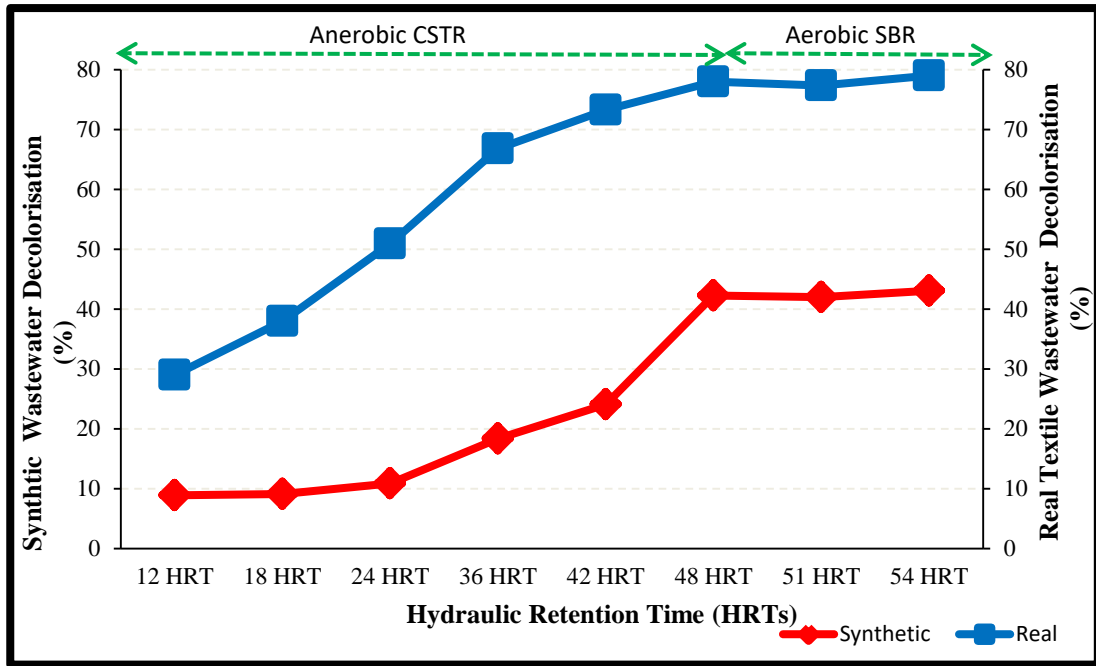
#### 4.1.2.2 Decolorisation in Combined Process

The analysis performed for samples from aerobic SBR was carried out to check the color reduction in the presence of oxygen. It has been reported that for a single azo dye solutions, the anaerobic dye reduction metabolites usually undergo a mechanism of

partial mineralization when the permeate from anaerobic treatment subjected to aerobic type treatment (Papić et al., 2004). The observed color removal in Figure 4.3 depicts anaerobic treatment to be the main process for wastewater in terms of its color removal since the slight increase in removal efficiency was observed when subjected to aerobic treatment. The reason for this behavior of aerobic SBR is in treating textile wastewater, the products release from anaerobic dye degradation like aromatic amines, which are aerobically non-biodegradable so cannot improve color removal efficiency notably and this finding is also reported earlier (Pandey et al., 2007).

Figure 4.3 shows that in both experimental phases, strict color reduction is observed in anaerobic CSTR. The percentage tends to increase in phase II which is due to fact that in case of real textile wastewater, time period for sludge acclimatization was provided longer as compared to synthetic wastewater. Literature shows the microbial capacity to degrade reactive dyes, dominantly azo type dyes, demands the sequence of processes resulting into different byproducts if former treatment technology is further extended to sequenced later technology (Khan et al., 2013; Zuriaga-Agusti et al., 2010).



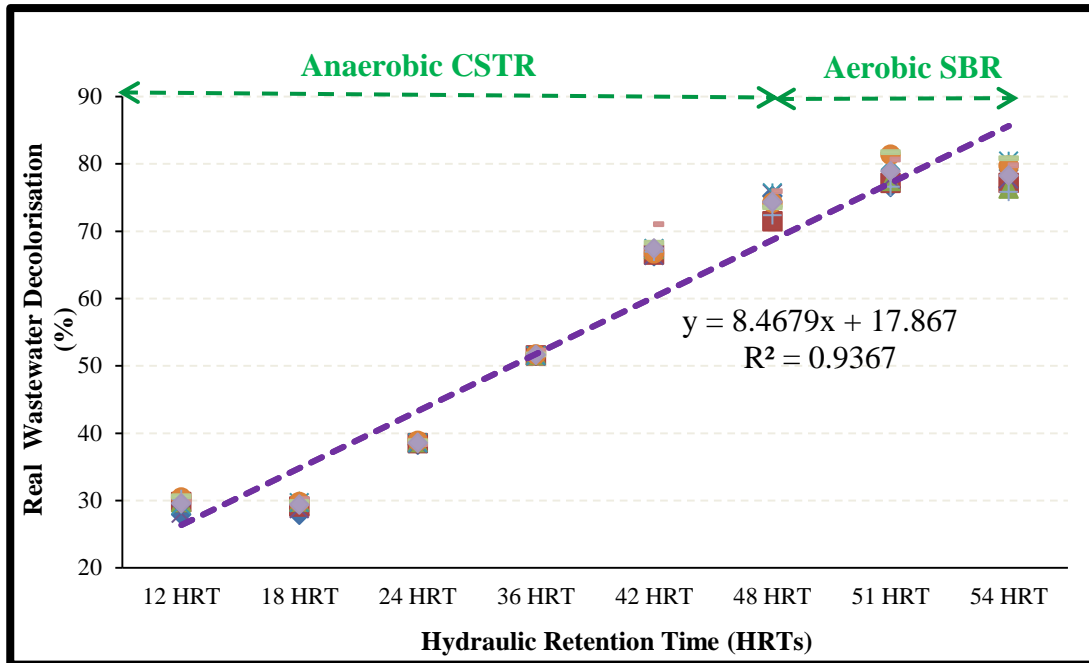


**Figure 4.3: Decolorisation trend of synthetic and real textile wastewater in hybrid anaerobic-aerobic system.**

#### 4.1.2.3 Applicability of Hybrid System in Color Removal

In both phases of the conducted experimental study, strict color reduction was observed in anaerobic CSTR and the removal rate slightly continued to increase in the aerobic phase as well but the observed efficiency was much less than anaerobic phase. The observed behavior supports that two stage system when applied together satisfies the desired color removal efficiency.

Figure 4.4 shows the overall efficiency of hybrid systems from which the equation for decolorisation efficiency is derived to prove the effectiveness and applicability of hybrid anaerobic CSTR-aerobic SBR systems at industrial level treating real textile wastewater. Figure 4.4 describes the linear equation  $y = 8.4679x + 17.897$  of  $R^2$  0.9367 showing the 93.67 % applicability of anaerobic treatment in hybrid systems to treat textile discharge when this technology is subjected to industrial scale.



**Figure 4.4: Equation evaluated from treatment of real textile wastewater treatment through hybrid system in terms of decolorisation percentage.**

### 4.1.3 Effect on Total Kjeldahl Nitrogen Removal

Total Nitrogen (TKN) is the cumulative of ammonia nitrogen (NH<sub>3</sub>-N) and organically bounded nitrogen. An average removal rate of effluent parameter of TKN was calculated throughout the study treating textile wastewater through hybrid biological system. Parameter like total nitrogen (TKN) removal also plays an important role in improving the treatment efficiency of sequential anaerobic-aerobic treatment technologies. Table 4.1 shows that TKN removal in anaerobic type reactor is low for both type of wastewaters (synthetic & real) and the provision of aerobic treatment in sequence increases the TKN removal rate.

Nitrification can be achieved in aerobic sequencing batch reactor at low organic loadings and where suitable environmental conditions are provided. Nitrifying bacteria are considered as slower growing microorganisms than the heterotrophic bacteria, which comprises the greater proportion of the biomass in both fixed film and suspended

growth systems. The key requirement for nitrification to occur, therefore, is that the process should control the net rate of accumulation of biomass, and hence, the net rate of withdrawal of biomass from the system, is less than the growth rate of the nitrifying bacteria. Therefore, SRT for aerobic SBR was maintained to be of 20 days in order to maintain MLSS concentration between 6000 – 8000 mg/L, in order to achieve maximum nitrification from aerobic SBR in hybrid system.

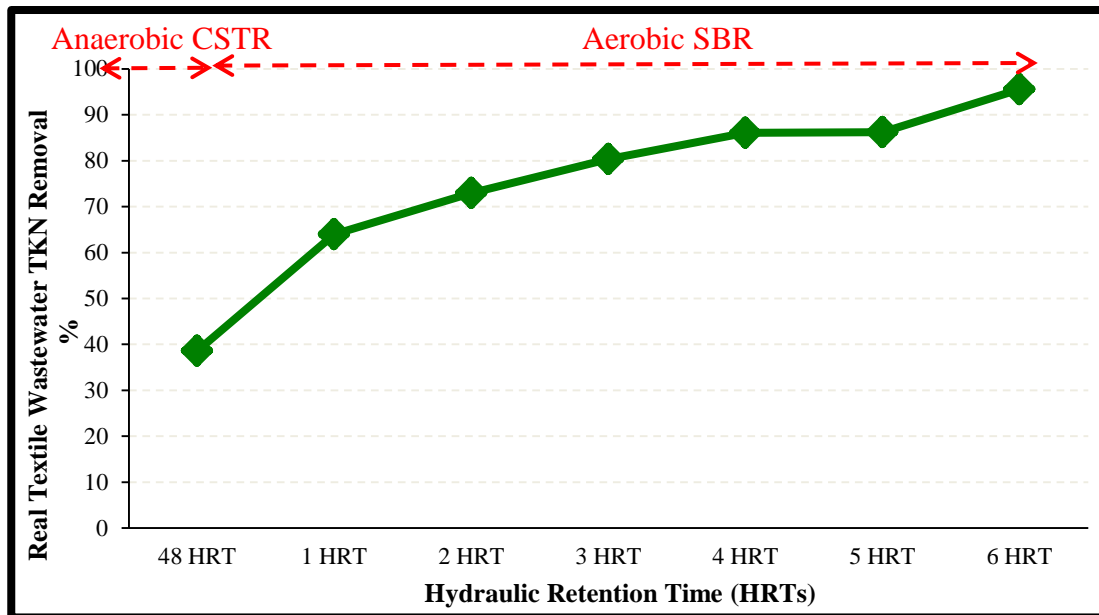
An average removal rate of total nitrogen in hybrid system treating real textile wastewater is observed to be of an average of 95.6 % because the assimilation of influent ammonia nitrogen and organic nitrogen enables total nitrogen to achieve certain levels of removal efficiencies as shown in Table 4.1.

**Table 4.1: Behavior of hybrid anaerobic-aerobic system in Total Kjeldahl Nitrogen (TKN) removal percentage.**

TOTAL KJELDAHL NITROGEN REMOVAL EFFICIENCY (%)								
PHASE	WASTEWATER	Hydraulic Retention Time (hours)						
		AnCSTR	AeSBR					
		48	1	2	3	4	5	6
I	Synthetic	31.8	59.8	66.9	77.2	86.0	93.0	99.3
II	Real	38.6	64.0	73.0	80.4	86.1	86.2	95.6

The maximum TKN removal is found in aerobic reactor at 6 HRT along with the maximum removal rates of color and COD. Studies have reported that microbial population in activated sludge process demands the amount of nitrogen as a nutrition for biomass growth (Zhidong et al., 2009). Figure 4.5 describes the TKN removal rate in hybrid system as the aerobic treatment plays an important role in TKN removal which polishes the treated wastewater. Also at 6 HRT, maximum TKN removal was found and system was optimized at this condition. Biological removal of total nitrogen

was first achieved by the transformation of organic nitrogen into ammonia, followed by the aerobic conversion of ammonia ( $\text{NH}_4^+$ ) to nitrite ( $\text{NO}_2^-$ ) and then nitrate ( $\text{NO}_3^-$ ).



**Figure 4.5: TKN percentage removal trend of real textile wastewater in hybrid biological system at variable HRTs.**

#### 4.1.4 Overall System Performance Efficiency

Removal efficiency for COD during phase I & II of study along with the removal of other constituents shows that hybrid systems are best alternatives in treating industrial wastewater resulting in higher removal efficiencies in cost effective manner. Both types of wastewater (synthetic and real) were subjected to hybrid anaerobic CSTR-aerobic SBR to treat real textile wastewater with reference to system performance by running synthetic wastewater. The effluent characteristics for important parameters providing hybrid type treatment in both phases of experimental study are shown in Table 4.2.

**Table 4.2: Effluent characteristics showing percent removal rate of hybrid anaerobic-aerobic treatment technology, meeting environmental quality standards.**

<b>EFFLUENT CHARACTERISTICS</b>	<b>PHASE I</b>	<b>PHASE II</b>
	Synthetic Type (%)	Real Type (%)
COD Removal (Anaerobic)	80.80	86.87
Total COD Removal	98.41	99.47
TKN Removal	95.60	99.30
Color Removal	43.08	78.40

Table 4.2 describes the overall effluent characteristics observed throughout the study. In treating high strength textile wastewater, hybrid systems provide positive removal efficiencies for parameters like COD (99.47%), TKN (99.30 %), Color (78.40 %) effectively in addition to the biogas production.

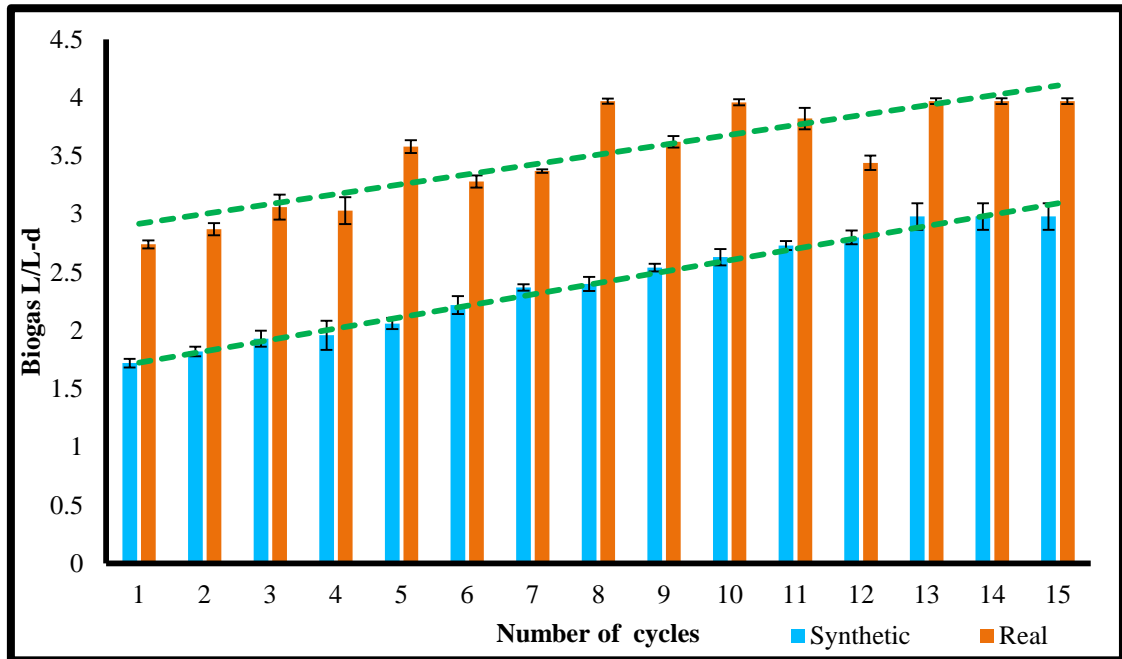
## **4.2 Energy Recovery**

Hybrid system provides the facility of valuable energy recovery in addition to the treatment of textile industrial wastewater, which adds value to the system in terms of its cost effectiveness and performance efficiency. This experimental study reveals that a general production of biogas takes place when textile wastewater is subjected to anaerobic treatment in anaerobic continuously stirred tank reactor (CSTR).

### **4.2.1 Biogas Production**

Figure 4.6 shows the results of anaerobic sludge acclimatization to synthetic and real textile wastewater in number of batch studies. During the initial batch runs, biogas production was found to be low as anaerobic microbial species are considered to be slow growing organisms, but the adaptation ability increased with increase in time till

complete acclimatized conditions were observed. The hydrolysis of wastewater organics into biogas is a slow process because of the activity of slow growing methanogens responsible for biogas production (Umairakunjaram and Shanmugam, 2016).



**Figure 4.6: Biogas production: An indication to Acclimatization of anaerobic sludge by running no. of batches of synthetic and real textile wastewater.**

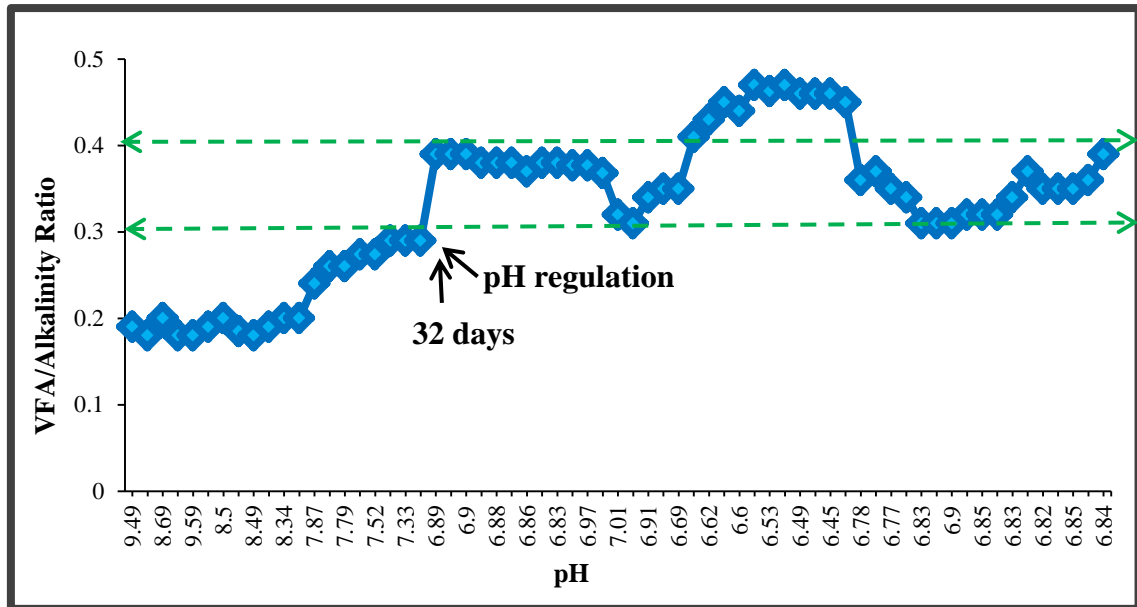
The production of biogas characteristically determine the adaptability of microbial community responsible for organic degradation. During the first phase of study, in application of synthetic wastewater of 3000 mg/L COD along with mixture of dyes, the biogas production was observed to be less due to gradual change in environment of the activated sludge from aerobic conditions to anaerobic conditions. In conventional anaerobic technologies, the acclimatization of sludge in treatment of wastewater to anaerobic conditions is reported to be mandatory as the higher removal rate cannot be possible with aerobic active microorganisms (Hossain et al., 2017). Also Figure 4.6 reveals that an increase in the biogas production in Phase II study of real textile

wastewater is due to the stability of sludge Acclimatization achieved in Phase I and strength declination from 3000 mg/L (synthetic) to 2400 mg/L (real).

#### **4.2.2 Ratio VFA/Alkalinity**

The function of facultative anaerobic bacterial species involves the conversion of carbon sources to volatile fatty acids (VFA) in the complete absence of oxygen. The accumulation of VFAs affects the anaerobic process stability therefore, by maintaining the VFA/Alkalinity ratio to 0.4 is a crucial parameter to keep anaerobic microbes in stable conditions (Somasiri et al., 2008). Figure 4.7 shows the effect of VFA/Alkalinity ratio in anaerobic CSTR that in real textile wastewater treatment an increase in pH happens at initial stages due to alkaline nature of wastewater which disturbed the reactor stability by lowering the VFA/Alkalinity ratio. The stable condition suitable for anaerobic microorganisms is ensured by maintaining the VFA/Alkalinity in the range of 0.3-0.4, which takes time to enhance sludge Acclimatization.

During anaerobic cycle, the sludge was completely acclimatized to synthetic wastewater of pH 6.8 to 7.2. When the application of high pH real wastewater to the optimized system takes place, VFA/Alkalinity ratio drops and initially this pH of 9.5 is adjusted to 7.2 by adding acid solution of HCl, to increase the action of facultative bacteria as shown in Figure 4.7. For pH adjustment, an option of chemical addition i.e. Hydrochloric Acid, Sodium Bicarbonate, can be a possible solution on laboratory scale study but on an industrial level, it will definitely add cost to the operational cost of biological systems. As shown in the graphical illustration of VFA/Alkalinity affecting pH due to the variations in real textile wastewater, it was observed that the anaerobic decolorisation efficiency and COD removal do not get affected by the adjustment of the pH because of better adaptation of microbial population involved in the treatment.



**Figure 4.7: Trend of VFA/Alkalinity ratio in anaerobic CSTR, in treating real textile wastewater.**

This outcome satisfies the literature, that bacterial consortium adapted to the pH of 6.8 to 7.2, does not require any pH neutralization before its application to treatment (Telke et al., 2008). However as Figure 4.7 depicts at initial steps, chemicals addition for pH adjustment was done but later, the increase in microbial adoption to pH self-regulation in anaerobic reactor was observed during experimental study.

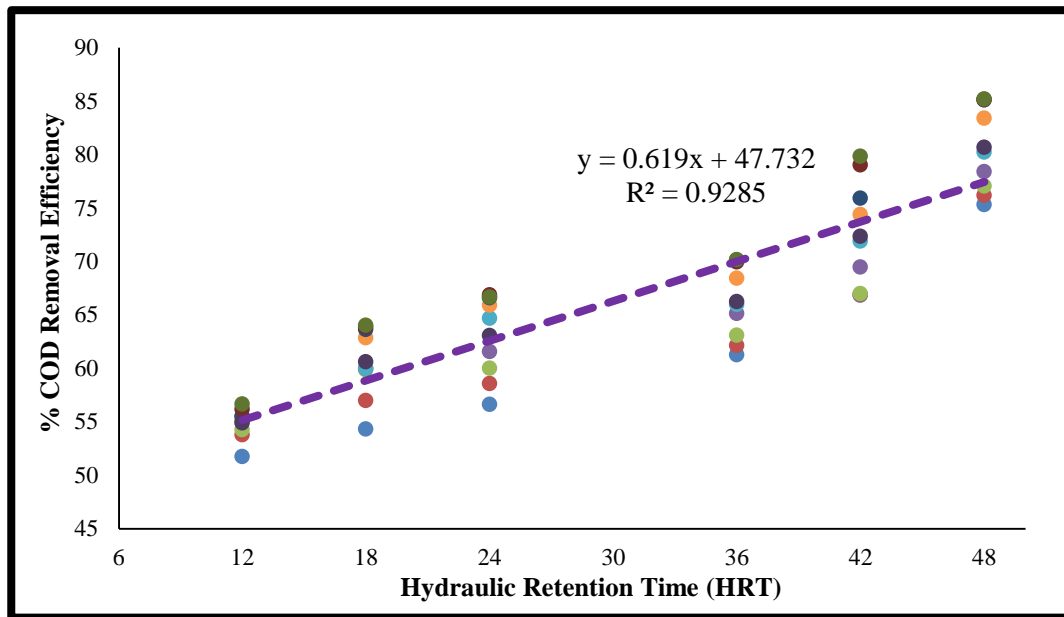
### 4.3 Applicability of Hybrid System to Industrial Scale

#### 4.3.1 Anaerobic Treatment Applicability

Figure 4.8 shows the overall efficiency of hybrid systems from which the equations for treatment efficiency are derived to prove the effectiveness and applicability of hybrid anaerobic CSTR-aerobic SBR systems at industrial level treating real textile wastewater. From Figure 4.8, it is illustrated that the linear equation derived from the experimental results shows the 92.85 % application possibility of anaerobic treatment in hybrid systems for textile wastewater discharged at industrial scale. This derived equation is :



$$y = 0.619x + 47.732 \quad (10)$$



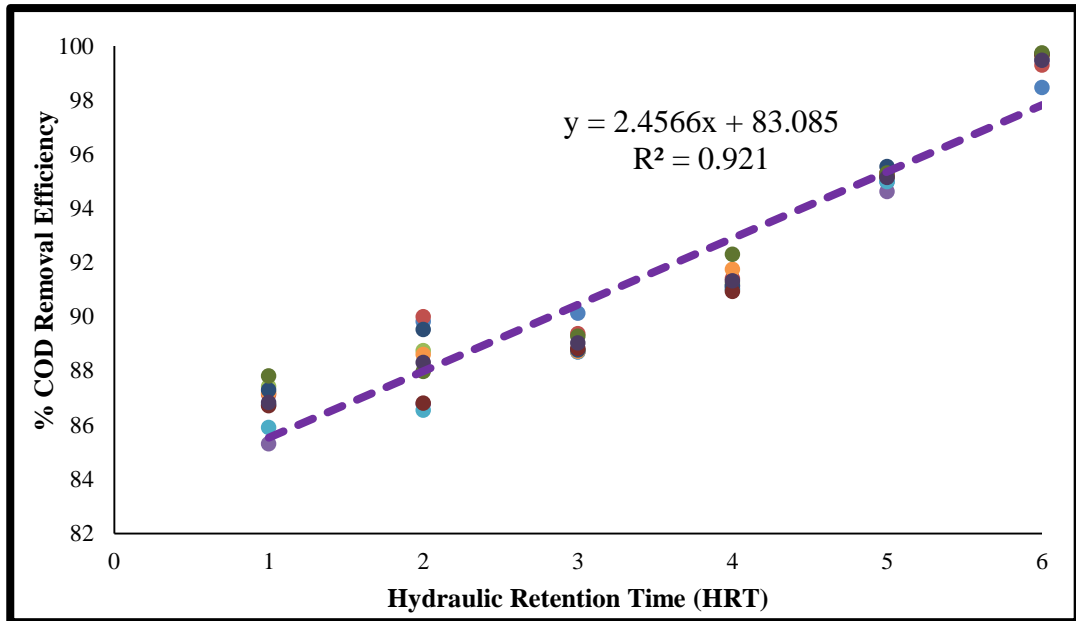
**Figure 4.8: Equation derived for anaerobic COD removal efficiency at different HRTs in treating real textile wastewater.**

### 4.3.2 Aerobic Treatment Applicability

The applicability of aerobic system to the industrial level in providing final polishing to the treated wastewater is also shown in Figure 4.9 by linear relationship equation:

$$y = 2.4566x + 83.085 \quad (11)$$

The derived equation illustrates that the performed experimental study shows 92.1% applicability of the aerobic treatment in hybrid systems when subjected to textile wastewater treatment at industrial level.



**Figure 4.9: Equation evaluated from performance of real textile wastewater treatment aerobically at variable HRTs.**

Therefore, with the help of these model equations, industries can find out their wastewater removal efficiencies at designed HRTs of hybrid system.

# **CONCLUSIONS AND RECOMMENDATIONS**

## **5.1 Conclusions**

A hybrid biological system for treating real textile wastewater was found to be a feasible treatment technology for developing countries. For real textile wastewater treatment, the optimized conditions of system performance were evaluated to be of 48 and 6 HRT for anaerobic CSTR and aerobic SBR, respectively. The production of biogas of  $3.51 \pm 0.30$  L/L-d, provides the value addition to the hybrid technology. Biogas production increased with better sludge acclimatization when VFA/Alkalinity ratio was found between 0.3-0.4. Satisfactory NEQS were obtained with high removal efficiencies of COD (99.5%), TKN (99.3%) and color (78.4%). Acclimatized sludge was found to have an impact in improving system efficiency and biogas production. The biological driving parameters of MLSS and pH of an 6000- 8000 mg/L, and 6.8-7.2 was maintained throughout the study. As compared to running anaerobic and aerobic system individually, maximum removal efficiencies were effectively obtained from the hybrid system. Hybrid Anaerobic-Aerobic Sequencing Batch Reactor (SBR) is a promising technology for treating real textile wastewater providing high treatment efficiency in addition to the recovery of biogas energy source. Therefore, for sustainability, adaptation of the hybrid anaerobic-aerobic system is recommended for textile industrial wastewater treatment.

## 5.2 Recommendations

- The biogas production can be increased for reprocessing it in anaerobic CSTR by substitution of mechanical stirring with biogas bubbling.
- Characterization of microbial species and dyes structure should be performed to bring more understanding in determining the microbial species responsible for efficient treatment in hybrid system.
- Combinations of hybrid system can also be implemented in order to compare the best technology for treatment of industrial wastewater
  - Integrated bioreactors with physical separation of anaerobic-aerobic zone
  - Anaerobic CSTR + Aerobic MBR.

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# ANNEXURE A

## THESIS LAYOUT

