

Photocatalytic Oxidation for Sustainable Management of Textile & Dyeing Industry Wastewater



Synopsis Submission

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Summary

Textile and dyeing consumes a huge amount of water and in turn generates large quantities of wastewater which contains high amounts of colorants, dyes, chemicals, surfactants etc. Notably, textile effluents have high colour, toxicity, odour with high chemical oxygen demand (COD) and low biochemical oxygen demand (BOD). These colour causing complex organic compounds in wastewater streams are not completely degraded/ removed using conventional physicochemical and biological treatment processes, whereas the removal of COD, Colour and BOD is essential for ensuring legal compliance and reusability of treated water.

In order to overcome these challenges, Photocatalysis (PC) which is an emerging advanced oxidation process (AOP) is a potential solution which is viable at commercial scale, easily automatable and doesn't have the limitations of high cost, chemical dosage and toxic sludge generated as compared to conventional AOPs like Fenton, Photo Fenton, Peroxidation, Photolysis of peroxide.

With this background, this thesis aims to evaluate the potential of photocatalysis as a standalone/ integrated approach for sustainable treatment of textile and dyeing effluent. Objectives of this research includes, i) Study of effectiveness and optimization of Photocatalysis in Textile and Dyeing Industry wastewater as an alternative to conventional treatment methods; ii) Identification of parameters for suitable scale up of the process on a Pilot scale reactor and iii) Techno-economic feasibility assessment of Photocatalysis & other Photochemical AOPs in comparison to conventional wastewater treatment methods prevalent in Textile industries in India. Scope of the study was limited to i) Textile industry shall be Dyeing Units generating coloured effluent containing recalcitrant dyeing compounds; ii) Pilot scale reactor treatment capacity developed and utilised in the study is of a maximum designed capacity of 100 Litre per Day; iii) Optimisation studies using simulated wastewater i.e. synthetic dye bath effluent using real dyes and chemicals; and iv) At least 2 real effluent treatment studies from different dye processing operations.

The presented work utilises a novel patented end-to-end pilot scale treatment (100 LPD) involving two stages has been presented to adequately treat real textile and dyeing industry wastewater to obtain process water quality. The pilot scale treatment utilised for the research work has been designed, implemented, operated and patented by The Energy and Resources Institute (TERI). As part of this research work, the plant has been optimised using

synthetic dye bath effluent, thereafter a sample from a real dyeing unit operating in Guntur, Andhra Pradesh, India was treated with the optimised method and technoeconomic assessment has been carried out. The results showed that overall BOD and COD reduction achieved was 95 % and 91% respectively with formation of negligible non-toxic sludge (0.4 kg m^{-3}), with estimated overall operational costs around 1.55 USD/m^3 and complete removal of total hardness, total nitrogen and mineralisation of organic nitrogen into inorganic Nitrogen along with significant reduction in chloride, TSS and TDS content. Major benefits of the overall scheme involves clean & green approach, toxic sludge free, scalable and robust technology with simultaneous disinfection. Bypassing bioremediation and involving around 5 hours in complete treatment ensures lower footprint and high efficiency. Additional advantages include tremendous reduction in use of chemicals, resources and operational costs. Proposed treatment is easily scalable for suitable field scale implementations due to the simplistic approach and design. Reuse of treated water as process water further ensures water re-use efficiency and implementation of Zero Liquid Discharge.

After successful study on a real effluent treatment using photocatalysis, another study has been done to treat combined effluent from 11 cotton, hosiery production and dyeing units from UP, India wherein photocatalysis has been evaluated to meet norms as per regulator in a sustainable manner. Given the complexity of the effluent, photocatalysis resulted in highly biodegradable treated water meeting the norms of downstream biological processing; moreover this study also compared PC with other photochemical AOPs like UV Photolysis, Photolysis of Peroxide and Photo Fenton. This study presents a batch pilot scale study on application of various these AOPs to treat textile wastewater from a Common Effluent Treatment Plant (CETP) to enhance its biodegradability and improve downstream biological system performance. It also compares the technoeconomic feasibility of various photochemical AOPs in terms of treatment efficiency, energy requirement and overall cost of treatment of the integration to treat combined effluent with AOP for better downstream biological treatment. The treatment results showed improved the quality of treated water in all cases with PC resulting in the most promising approach due to efficient removal in COD, BOD and minimal Electrical energy per order ($10.79 \text{ kW-hr/m}^3/\text{order-COD}$ removal and $5.16 \text{ kW-hr/m}^3/\text{order-colour}$ removal). Moreover, UV/ TiO_2 resulted in the most economical treatment with overall cost estimated as 59.75 INR/ m^3 , however UV/ H_2O_2 and UV -Photo Fenton also show promising COD & Colour removal with three fold improvement in

biodegradability but have higher cost of treatment at 126 INR/ m³ and 155 INR/ m³ respectively.

After comparisons of these photochemical AOPs on the basis of their degradation efficiency of COD, Colour and BOD including their energy, cost and overall life cycle potentials, a detailed Life Cycle Assessment (LCA) of the proposed implementation was carried out. Other techno economic parameters like E_{EO} etc have been computed however it is even more important to compute the net footprints of the proposed solution on the environment. Hence, the presented research work also compares the sustainability potential through a gate to gate life cycle assessment for i) optimised PC approach with other photochemical AOPs for its standalone implementation to treat dyeing wastewater; ii) optimised PC approach with other photochemical AOPs for integrated implementation with conventional technologies, and iii) footprint of a typical zero liquid discharge compliant unit with an integrated AOP system. The study showed that the integration holds the potential to contribute to national and international programmes of decarbonisation and meeting Sustainable Development Goals (SDGs).

Hence, the aim of assessing PC as a sustainable approach for treating textile and dyeing effluent was completed and the finalised objectives were complied. Such study shall open up other works in the area particularly towards field scale implementation, large scale continuous plant assessments etc which will enhance water reuse in the textile sector.

1 Introduction

Textile industries play a crucial role in fiscal perspective but this sector is facing various environmental challenges concerning the freshwater scarcity, wastewater management and associated environmental acquiescence due to the release of inadequately treated effluents with abhorrent chemicals, heavy metals at a higher concentrations into the receiving water body. Therefore, the non-conformity from the recommended ranges have undesirable impacts to the terrestrial and aquatic ecological system and human health [1], demand prompt attention and innovative advancement in management technology. The conventional wastewater treatment protocols particularly practiced in the micro, small and medium scale enterprises (MSMEs) and depend upon the excessive consumption of ferrous salts, lime and alum as coagulants-flocculants along with their uncontrolled dosing followed by traditional aerobic biological treatments. The mainstream treatment technologies as coagulation & flocculation, flotation, adsorption and biological treatment [2,3] along with the few advanced systems, *e.g.*, membrane-based methods, evaporation-based techniques [4] may be favourable for few of the physicochemical factors as well as chemical contaminants in textile wastewater.

Thus, the traditional treatment techniques are susceptible towards shock loads and needs diverse coagulants to treat the different effluents with different attributes with low decolourization efficiency of the wastewater and leads to generation of huge resultant sludge thereby it creates the problem of secondary pollution [1,5–7]. Not only that, but also the oxidants which are used in these traditional methods are unable to degrade and/or detoxify the extremely persistent hazardous substances [8–12]. Specifically, the biological treatment of wastewaters has become quite popular and have been well-practiced at the commercial scale for their uncomplicated design, cost-effectiveness and user friendly approaches, involves the methods as Activated Sludge Process, Extended Aeration, Moving Bed Biofilm Reactor or Fluidised Bed Reactor, Membrane Bioreactors, Sequential Batch reactor and Submerged Aerated Fixed Film [1,4,13,14]. A number of researches experimentally established that the bioremediation can be fruitful to treat effluent water from textile units at pilot scale and Sequential Batch reactor [13], Anaerobic- Fluidised Bed Reactor [15] and Activated Sludge Process [16,17] are supposed to be effective in removal of Colour, COD and BOD, but, field scale installations showed lower biodegradability of the wastewater sample due to the

presence of dyes with complex chemical properties and existence of other toxic compounds, which hinder the microbial growth and thereby affecting the efficiency of these processes, that in turn leads to inadequate treatment, where treated water is not free from dissolved organics and cannot be suitable to process wastewater [18–21]. Furthermore, these microbial treatment based systems are time-intensive and consume prolonged treatment duration of around 12 to 24 hours, acquire large area footprint and also generate huge volumes of toxic slurry [22] demands secondary concerns.

Considering the prior indicated shortfalls in wastewater treatment requires more advanced alternative approach as Advanced Oxidation Processes (AOPs) like UV photolysis [23–25], UV photolysis of peroxide [26], UV photo Fenton [27–37], and UV photocatalysis [5,38–42]. in terms of integrated approach with techno-economic competency to treat the effluent by AOPs for improved biological treatment to remove the obnoxious pollutants with high chemical stability and/or low microbial degradability and the basic mechanism of treatment of wastewater by producing highly oxidising reactive hydroxyl radicals (OH•) with strong destruction capability, that can oxidise and degrade stable organic compounds, typically unsaturated molecules quiet promptly and indiscriminately [11,25,43]. Therefore, the combinations of AOPs have the potential to adequately treat the heterogenous effluents and improve health of treatment plants, augment capacities, achieve Zero Liquid Discharge (ZLD) compliance and enhance water reuse. More specifically, several published studies have explored the integrated approach in treatment, even of the challenging streams [6,39,44–77]. Some of the recent studies from India, have integrated Photocatalysis with conventional biological treatment for treating mixed sewage effluent containing Oils, Solvents causing COD, BOD with other Persistent Organic Pollutants (POPs) and Contaminants of Emerging concern (CEC). The result from this works shows a significantly improved treated water quality as compared to conventional treatment alone [78]. Similar improvement has been reported in a recent study of photocatalysis as a standalone approach for treating heterogenous polluted open drain [78,79].

This section shall elaborate on the above points and shall be subdivided into the following sub- sections as follows:

1.1 Challenges in Textile and Dyeing Industry

1.1.1 Environmental

1.1.2 Regulatory Compliance

1.1.2 Wastewater Management

1.1.2.1 Treated Water Quality

1.1.2.2 Energy

1.1.2.3 Chemicals

1.1.2.4 Sludge

1.2 Sustainability

1.2.1 Capital Expenditure CAPEX

1.2.2 Operational Expenditure OPEX

1.3 Life Cycle Assessment

1.4 Role of Advanced Oxidation Processes (AOP) to meet these challenges

1.5 UV- TiO₂ Photocatalysis as a sustainable AOP

2 Aims and Objective

The current research has examined that, the incorporation of advanced technologies like Heterogenous Photocatalysis among Advanced Oxidation Processes (AOPs) can be a promising, potential technology and energy efficient option to meet the existing challenges and to obtain water for reuse. In integration and/ or standalone implementation of TADOX[®] at the pre-biological stage/ post-biological stage or as an alternative, depending on the nature of the effluent under analysis.

Therefore, the aims and objective of this research-based work is, therefore, to assess the accomplishment of the integration of photocatalysis by augmenting the biological treatment systems or as a standalone implementation as per techno-economic perspective to enhance the reuse of treated wastewater. The current work has set the following definite objectives:

- **OBJECTIVE 1:** Study of effectiveness and optimization of Photocatalysis in Textile and Dyeing Industry wastewater as an alternative to conventional treatment methods.
- **OBJECTIVE 2:** Identification of parameters for suitable scale up of the process on a Pilot scale reactor.
- **OBJECTIVE 3:** Techno-economic feasibility assessment of Photocatalysis and comparison with other Photochemical AOPs with conventional wastewater treatment methods prevalent in Textile industries in India.

Scope of the Study:

- Type of Textile industry shall be Dyeing Units generating coloured effluent containing recalcitrant dyeing compounds
- Pilot scale reactor treatment capacity developed and utilised in the study is of a maximum designed capacity of 100 Litre per Day.
- Optimisation studies using simulated wastewater i.e. synthetic dye bath effluent using real dyes and chemicals.
- At least 2 real effluent treatment studies from different dye processing operations.

3 Review of Literature

Conventional prevalent Textile industry is one of the key industries playing vital role in economies of developing countries like China, Bangladesh, India, Pakistan, Vietnam, Indonesia etc. At the same time, this industry faces major challenges of waste management and associated environmental compliances. Among key challenges, inadequate treatment of wastewater, lack of fresh water availability and management of highly toxic sludge residues, obtained as the result of conventional chemical and biological treatment needs immediate attention and innovation.

The current wastewater treatment practices rely on excessive use of chemicals; chemical treatment using conventional coagulants and flocculants and uncontrolled dosing of lime/ alum/ ferrous salts etc. These conventional protocols have been in use at micro, small and medium scale enterprises (MSMEs) for a long time owing to its lower capital and operational costs. However, these chemical treatments are prone to shock loads and application of different coagulants maybe required for treatment of different effluent streams. They also have low decolourization efficiency for the wastewater having reactive and vat dyes and leads to large generation of resultant sludge [1].

Lab scale studies have shown that newer pre-hydrolysed coagulants such as Poly-aluminium chloride (PACl), Poly aluminium ferric chloride (PAFCl), Poly-ferrous sulphate (PFS) and Poly-ferric chloride (PFCl) may prove to be among better choices, owing to their superior colour removal even at smaller dosage and their efficiency at wider pH range [80,81]. However, these primary treatment systems may still be unsuitable for overall sustainability of treatment, as these processes result in mere phase-transfer of dyes and chemicals into toxic sludge whose disposal and management is another crucial matter.

Biological treatment has been used at commercial scale for a long time due to its simplistic design, low cost and easy operation. Common biological treatment systems involve approaches like Activated Sludge Process (ASP), Extended Aeration (EA), Submerged Aerated Fixed Film (SAFF), Moving Bed Biofilm Reactor (MBBR) or Fluidised Bed Reactor (FBR), Sequential Batch reactor (SBR) and Membrane Bioreactors (MBR). Several reports on application of bioremediation for treatment of real textile effluent at pilot scale have shown SBR [82], Anaerobic-FBR [83] and ASP [84] to be successful in removal of Colour, COD and BOD. However, field scale installations face challenges of low biodegradability of wastewater due to presence of complex dyes and toxic compounds, which hinders microbial growth thereby reducing the efficiency of these processes. This leads to inadequate treatment, where treated water is not free from dissolved organics and can not be used as Process Water [20,85,86]. Moreover, these biological systems involve at-least 12 – 24 h treatment, incur large area footprint and produce large amounts of toxic sludge [22].

In view of above shortcomings, innovation in wastewater treatment especially in developing countries is the need of the hour. AOPs are processes which results in in-situ generation of hydroxyl radicals ($\bullet\text{OH}$) which are most powerful and 10^9 times faster than Ozonation which has been used for quite some time in wastewater treatment [87]. AOPs are referred for a group of processes which ultimately generate hydroxyl radicals which acts as oxidising species for targeted organic contaminants. AOPs have been reported to efficiently, effectively and economically destroy organic contaminants in water and wastewater [88]. These processes are classified on the basis of the source of generation of $\bullet\text{OH}$; like by addition of chemicals (Ozonation, Peroxidation and (Fe(II) Salt with Hydrogen Peroxide) i.e. Fenton's Process) known as Chemical AOP, use of artificial light i.e. UV (Ozonolysis, Peroxidation, Photo Fenton, Heterogenous Photo-Catalysis) known as Photo Chemical AOP; use of ultrasonic waves known as Sono-Chemical AOP and when electrochemical reactions result in formation of hydroxyl radicals then these are Electrochemical AOPs. Several published reviews and research work have shown that AOP based technologies are promising, environmentally friendly, and emerging solution for complete destruction of POPs and toxic organic matter present in water [87,89–94].

Heterogeneous Photocatalysis (HP) is class of emerging AOP that made significant advancement in recent years by utilising action of artificial light on TiO_2 suspensions and this has been widely accepted as an efficient technology [95]. Some of the advantages of this

technology are minimal addition of chemicals and simultaneous destruction of bio-pollutants including pathogens like viruses and Bacteria [96]. This technology is self-sufficient for treating a substantial range of inorganic, high and low molecular weight organic contaminants and can be used as a standalone technology and can be easily integrated with biological systems [96]. This AOP has the potential to treat water having heavy metal interferences it can reduce heavy metals and degrade organic contaminants at the same time.

HP involves use of UV light to excite semiconductor (like TiO_2 / ZnO) to induce oxidation- reduction reactions since the energy gap (between the valence and conducting bands) is overcome by the UV radiation, causing formation of electron-hole pairs which migrate on surface and then react with adsorbed chemical species [97]. The photo-generated holes are strong oxidants, and the photo-generated electrons are reducing enough to yield superoxide from dioxygen. In these potential conditions, the photo-generated holes can either directly oxidize the absorbed pollutants or oxidize the hydroxyl groups located at the TiO_2 surface to form $\bullet\text{OH}$ radicals, whose redox potential is only slightly decreased. Consequently, the degradation of pollutants contained in the contaminated waters can take place either directly at the semiconductor surface or indirectly through interactions with the $\bullet\text{OH}$ radicals, the indirect oxidation by the radicals being the most favoured degradation pathway. In addition, it is possible to again increase the number of $\bullet\text{OH}$ radicals by adding into the photoreactor H_2O_2 or O_3 which can be photolyzed by UV irradiation [98].

Several published review articles and research papers show that AOPs including ozonation and use of UV light for wastewater treatment particularly for degradation of difficult organic contaminants in wastewater [1,87,96,99–104]. Some recent studies have reported that photocatalysis and electrochemical AOPs like have been successful in complete dye treatment as end to end treatment [105–109]. Several reports have cited use of AOPs for achieving net zero greywater/ municipal sewage waste recycle through complete degradation of recalcitrant organics [110]. Other reports also suggest its tremendous potential for treatment of municipal wastewater systems with integration [52,99,110–117]. Photocatalysis using TiO_2 with Visible/ solar and UV radiation has also been shown to be particularly useful in treatment of POPs in water [118–121]. Recent report of Jallouli et. al shows tremendous potential of energy efficient photocatalysis using UV-LED and TiO_2 for treating trace Ibuprofen in ultrapure and pharmaceutical industry wastewaters [122].

Hence, use of advanced and promising technologies like Heterogenous Photocatalysis (HP) among Advanced Oxidation Processes (AOPs) where it utilises n-TiO₂/UV is widely accepted as a promising and efficient technology to meet these challenges [95]. HP is among the class of AOPs that made significant advancement in terms of upscaling in recent years owing to improved membrane based systems which can recover used nanomaterials which could be further regenerated and reused [123,124].

It has been shown previously that coupling of biological treatment and photocatalysis for treatment of real dye wastewater, resulted in the treated water meeting discharge norms with operational and economic feasibility [125,126]. In general, there has been success of integration of biological treatment, chemical treatment and AOPs for successful treatment of real textile wastewater [51,126–129].

AOPs have been shown in particular to be effective on integration with biological treatment systems, like in some cases wherever biodegradability in terms of BOD₅/ COD₅ is very low, AOP is used for pre biological stage to break down complex organics and enhance biodegradability [130]. Photocatalysis has also been used for pre-treatment of textile wastewater prior to its treatment using bioremediation technique in several reports and this greatly improved biological treatment [131]. It has also been shown to improve biodegradability of pesticide and emerging pollutant rich wastewater [132]. Jamil et. al have also shown photochemical AOPs to greatly improve biodegradability of difficult real pulp wastewater [133]. Several reports have shown use of photochemical, photocatalysis and electrochemical AOPs to improve biodegradability of wastewater [134]. Parra et. al proposed a photocatalytic flow through system for complete mineralisation of Isoproturon (IP) within 60 min with 95% removal of dissolved organic carbon (DOC), the coupling was found to be useful because the derivative of IP which was formed post PC was biologically compatible and hence lead to complete removal of the pollutant post biological treatment [119].

Photocatalysis has been even studied for its use for treatment of tertiary wastewater for 'polishing' wastewater and the photo-catalytically treated water is free from dissolved organics and pathogens. Several reports show use of solar driven PC for treatment of tertiary wastewater for treating trace contaminants of emerging concern [53,135,136]. Xu et. al were able to successfully demonstrate coupling of MBBR and TiO₂ Photocatalysis for advanced treatment of real coal gasification wastewater [48]. Combination of biological systems with photocatalysis has also been extensively used for treatment of urban wastewater and leachate

treatment and report cited that the combination maybe effective method to treat such difficult streams owing to the synergistic effect from the two [137]. The coupling of biological treatment and photocatalysis has also been extensively applied for treatment of real dye wastewater and results show that the treated water was within discharge norms with economically feasible running cost [23,138,139].

As conventional methods use biological treatment and physicochemical treatment for treating wastewater however the obtained treated water is unsuited to meet characteristics for reuse in flushing, cooling or gardening. Therefore, advanced treatment like photocatalysis or other AOPs are expected to reduce sludge generation, reduce footprint, improve automation of such system and completely treat water which is free from emerging contaminants of concern.

The above RoL summary shall be elaborated in the final thesis

4 Materials and Methods

This section shall cover the overall approach and methodology adopted for carrying out the study. It will give detailed information of the overall treatment used in conducting the study, experimental setup, chemicals, analytical techniques used for characterisation. It shall also give a detailed description of the sample collection, sample pre treatment, preservation, storage etc. The tools used for conducting the techno economic feasibility, equations and theory used with the details of Life Cycle Assessment (LCA) model shall be added.

A simplified process flow diagram of the TERI advanced oxidation technology (TADOX) is shown in Fig. 1. Technical information and detailed methodology of the TADOX[®] Technology based pilot scale plant treating 100 litres per day (LPD) and its operation has been published in another recent article. Details of plant operation, analysis of the wastewater quality parameters, computation of energy requirements and evaluation of the Figures of merit has been already given in the earlier publication of this series of case studies. It involves UV-TiO₂ Photocatalysis as the secondary treatment followed by nanomaterial recovery at source. Such a photocatalytic treatment has been established to be useful in Dye intermediates, Basic organics, Dye molecules, Synthetic textile effluent, Real textile and dyeing wastewater treatment systems and has been successful in eliminating need of biological treatment at any stage.

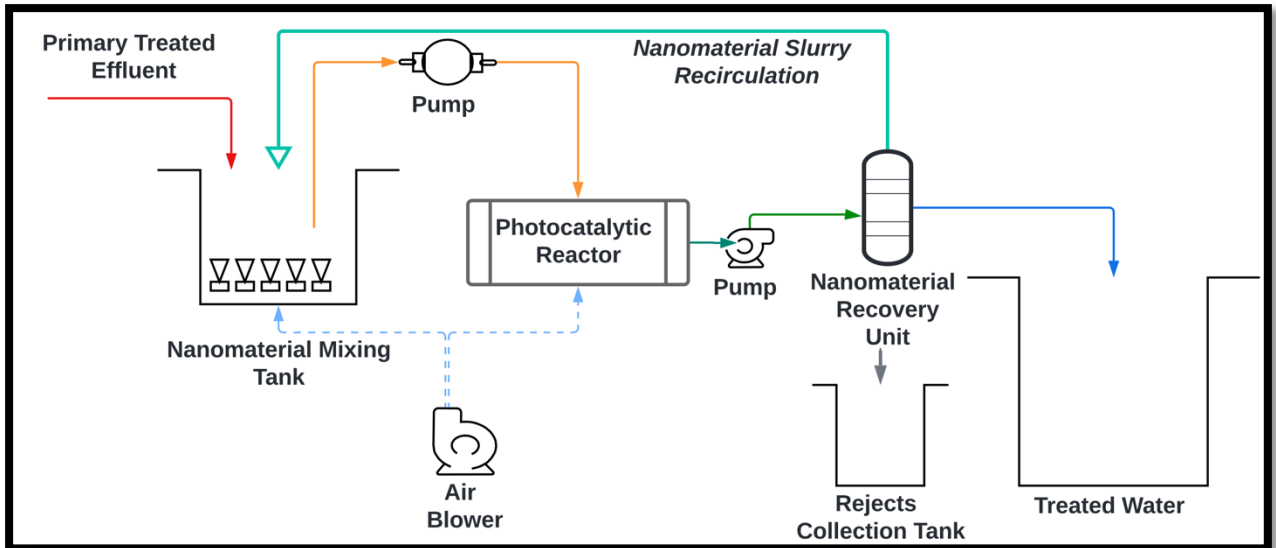


Fig. 1. Flow diagram of the heterogenous photocatalysis system based on TERO advanced oxidation technology (TADOX®)

During the TADOX® treatment, the treated water was transferred via a built-and-developed mechanism equipped with suitable membrane filters to remove the spent nanoparticles. Clean water was thus obtained, and the used nano catalyst recovered. For other three processes illustrated earlier, there was no need for separation of nano catalysts. Hence, to explore whether the nano catalyst from TADOX® can be reused for treating the next batch, all reject material from the nanomaterials recovery unit was stored in a reject-collection tank. Reject water from this tank was oven-dried repeatedly and regenerated after adequate washing and air drying to remove inorganic salts.

Further detailing on the working of the plant, methodology for optimisation of the plant parameters like pH, TiO₂ dose, UV irradiation time etc, sample selection, sample working and characterisation of wastewater shall be detailed in this section. Moreover, the complete information on computation of technoeconomic feasibility parameters like Electrical energy per order pollutant ($E_{EO, COD}$ and $E_{EO, CU}$) as figures of merit, cost of treatment etc shall be discussed in this section. Finally, complete information about life cycle assessment tools used in the study for comparison of photocatalysis with other photochemical AOPs, conventional plants etc shall be detailed.

An outline of the sub sections under this chapter are as below:

4.1 Experimental setup of the pilot plant

4.1.1 Overall PFD of the pilot plant

4.1.2 Description of unit processes

- 4.1.2.1 Physicochemical treatment using Flash Mixer and Tube Settler
 - 4.1.2.2 Nanomaterial Mixing unit
 - 4.1.2.3 Photocatalytic Reactor (PCR)
 - 4.1.2.4 Nanomaterial Recovery Unit (NMRU)
 - 4.1.3 Optimisation of the operational parameters
 - 4.1.3.1 Flow
 - 4.1.3.2 pH
 - 4.1.3.3 Catalyst dose
 - 4.1.3.4 Oxidant usage
 - 4.1.3.5 Reusability studies of spent nanomaterials
- 4.2 Preparation of Simulated wastewater - Synthetic dye bath**
 - 4.2.1 Constituents
 - 4.2.2 Preparation
 - 4.2.3 Storage
- 4.3 Study of Real Samples**
 - 4.3.1 About the Sampling location
 - 4.3.1.1 Individual Effluent Treatment plant (IETP)
 - 4.3.1.2 Common Effluent Treatment plant (CETP)
 - 4.3.2 Sampling, Preservation and Transportation
- 4.4 Testing and Analysis of Samples**
 - 4.4.1 Wastewater Characterisation
 - 4.4.1.1 Physicochemical parameters
 - 4.4.1.2 Organic parameters
 - 4.4.1.3 Trace metals parameters
 - 4.4.2 Nanomaterial Characterisation
 - 4.4.2.1 XRD
 - 4.4.2.2 BET
 - 4.4.2.3 SEM and EDAX
 - 4.4.2.4 FTIR
 - 4.4.3 Sludge Residue Characterisation
 - 4.4.3.1 TCLP
 - 4.4.3.2 XRD
 - 4.4.3.3 SEM
 - 4.4.3.4 EDAX
- 4.5 Techno-economic assessment**
 - 4.5.1 Computation of Figures of Merit
 - 4.5.1.1 Electrical Energy per Order (E_{EO})
 - 4.5.1.2 Electrical Energy per Volume (E_{EV})
 - 4.5.2 Computation of cost of operation of PC
 - 4.5.3 Computation of overall cost of treatment
- 4.6 Life Cycle Assessment**
 - 4.6.1 Scope of the study
 - 4.6.2 Establishing the system boundary

- 4.6.3 Parameters considered for the modelling
- 4.6.4 Assessment of Flow and Load
- 4.6.5 Computation of Inputs and Modelling
- 4.6.6 Data Analysis
- 4.6.7 Assumptions and Limitations

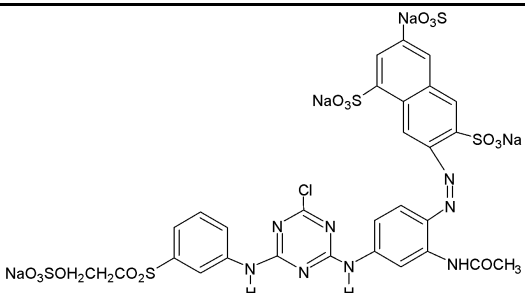
5 Optimisation of Pilot plant using Simulated wastewater

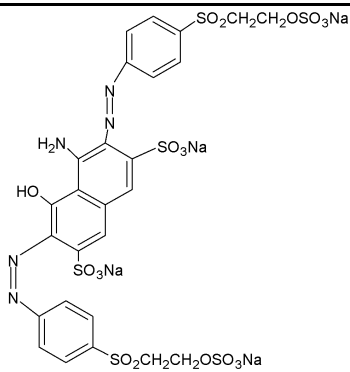
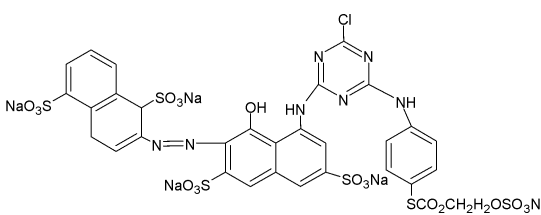
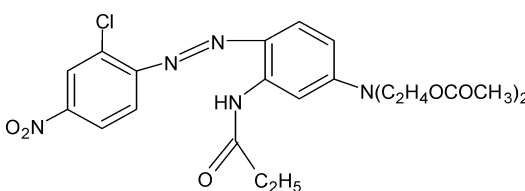
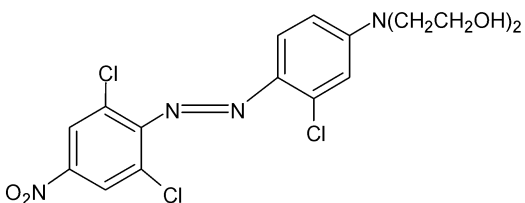
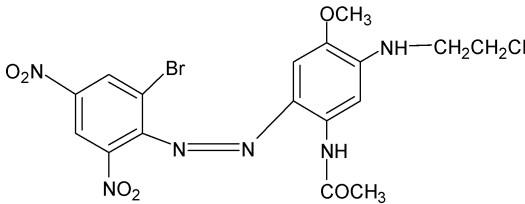
This section shall discuss the data generated from the studies on synthetic dye bath treatment done using the pilot plant. The optimisation studies on the pilot plant for pH, Catalyst dose, UV light irradiation time etc were also carried out on dye bath and have been discussed in this chapter. Finally, the degradation of colour causing compounds, chemicals, COD, BOD, TSS etc are discussed with the approximations on energy consumption and incurred overall cost of treatment.

Preparation of Synthetic dye bath effluent

For the photocatalytic experiments and optimisation of the pilot plant, a synthetically prepared dye bath solution was prepared with 6 dyes, 2 salts and 6 auxiliary liquids in Tap Water Matrix, to simulate an industrial effluent for the pilot scale optimization studies. All dye bath ingredients and the entire recipe was provided by Archroma India Pvt Ltd, Mumbai which is a global leader in dyes and specialty chemicals in the coloration industry. Table 1 provides necessary information regarding all 6 dyes used in the dye bath; this includes their chemical and common names, chemical structure, classification of dye and its molecular weight.

Table 1: Details of the Dyes and chemicals used to prepare synthetic dye bath/ simulated wastewater

S.No.	Dye	Dye Structure	Type/Class of Dye	Molecular Weight (g mol ⁻¹)
1	Drimaren Yellow CL-2R (Dispersed yellow 176)		Single Azo class	1025.26

2	Drimaren Navy CL-R (Reactive Black 5)		Double class	Azo	991.82
3	Drimaren Red CL-5B (Reactive Red 241)		Single class	azo	1026.41
4	Foron Rubine S-9053 (Dispersed Red 167)		Single class	azo	519.93
5	Sodycron Yellow Brown S.IN (Disperse Brown 1)		Single class	azo	433.67
6	Sodycron Navy S.IN (Dispersed Blue 281)		Single class	azo	534.327

From the data, it could be clearly seen that the synthetic solution is a mixture of dyes mainly single and double azo class. As per the protocol of dissolution of dyes, each dye was appropriately weighed and then mixed to form a homogeneous solution. Other auxiliaries and salts were directly added without heating. The two salts include Soda Ash and Glauber Salt while 6 auxiliary chemicals being i.e. Setmol WS, Opticid PSD, Ladipor RSK, Lyocol RDN, Ladipur

MCLI and Eganil PS. The effective dye bath concentration was 400 mg L⁻¹ (200 mg L⁻¹ for dye mixture and 200 mg L⁻¹ for the salts and auxiliaries).

Complete details of this work have been published under Bahadur et. al 2020 under manuscript titled “**Improving energy efficiency and economic feasibility of photocatalytic treatment of synthetic and real textile wastewater using bagasse fly ash modified TiO₂**” [7].

A brief outline of the section is as below:

5.1 Optimisation of operational parameters

5.1.1 pH

5.1.2. Retention time

5.1.3 Catalyst Dosage

5.2 Discussion on pollutant degradation

5.2.1 Effect on Physicochemical parameters

5.2.2 Effect on organic pollutants

5.3 Computation of techno economic assessment of the implementation

5.3.1 Energy consumption

5.3.2 Cost of treatment

6 Studies on Real Effluent treatment using Photocatalysis

Real wastewater sample from equalization tank of a cotton dyeing unit in Guntur, Andhra Pradesh, India was delivered by Air and treated as soon as received in the installed Pilot plant of 100L per day treatment capacity. It was subjected to PC treatment for a total of 5 hours including physicochemical treatment, photocatalysis and nanomaterial separation. Fig 1. Shows a depiction of the UV-Vis Spectra of three subjected to PC treatment. Details of the wastewater characteristics are tabulated in **Table 2**.

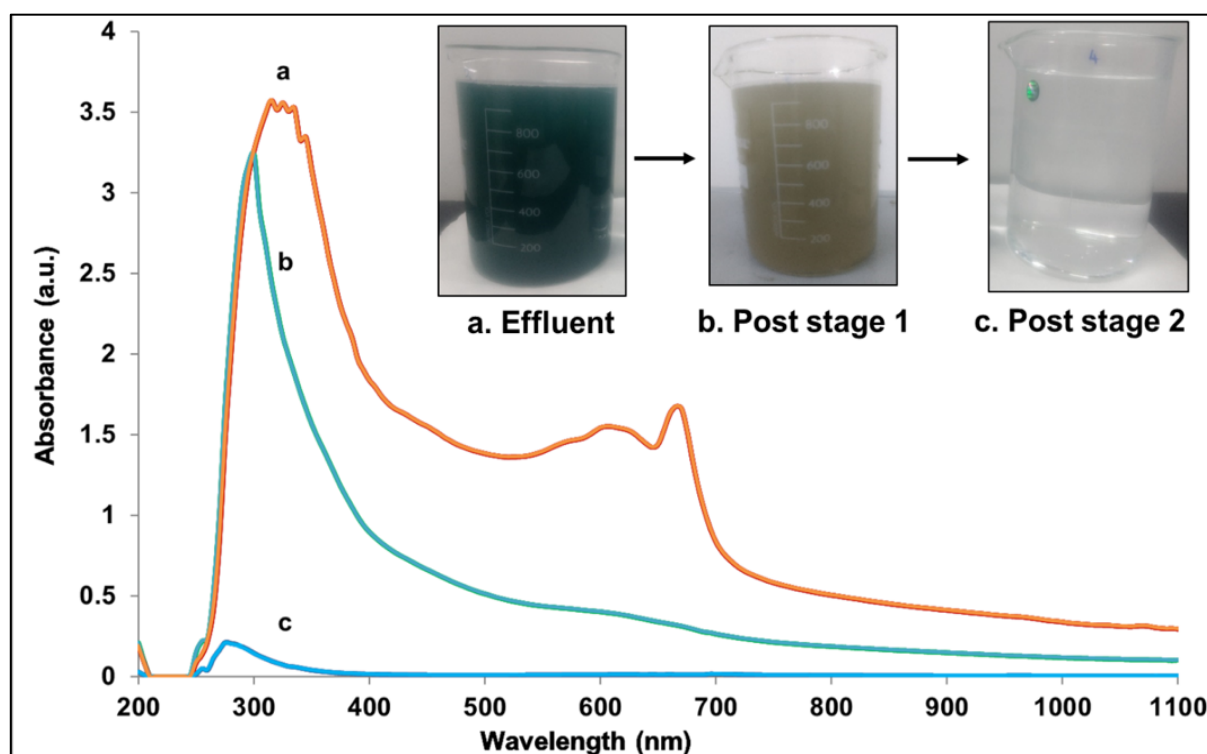


Fig.1. UV- Vis spectra of textile dyeing effluent (a) Untreated/Raw effluent (b) Post Stage 1 and (c) Post Stage 2 treated. Inset: Respective pictures of samples.

Table 2: Wastewater characteristics of Untreated and Treated samples

Parameters, unit		Results		
S.no		Raw Sample	Post Treated	Standards notified by CPCB for treated effluents from Integrated Textile Units
1.	pH	7.62	9.1	6.5-8.5
2.	Salinity [#] , ppm	3470	130	-
3.	Conductivity [#] , $\mu\text{mho/cm}$	7644	294	-
4.	Total Suspended solids (TSS), mg L^{-1}	850	4	100
5.	Total Dissolved Solids (TDS), mg L^{-1}	33350	264	2100
6.	Chloride [#] , mg L^{-1}	240	30	-
7.	Total Hardness [#] , mg L^{-1}	60	ND	-
8.	Calcium [#] , mg L^{-1}	22	1.8	-
9.	Magnesium [#] , mg L^{-1}	3.125	0.1	-

10.	Iron (Fe) [#] , $mg L^{-1}$	3.24	ND	-
11.	Total Chromium (Cr), $mg L^{-1}$	1.13	ND	2
12.	BOD ₅ , $mg L^{-1}$	255	12	30
13.	COD, $mg L^{-1}$	1360	128	250
14.	Total Nitrogen [#] , $mg L^{-1}$	158.4	60.7	-
15.	Total Kjehldal Nitrogen (TKN), $mg L^{-1}$	102.1	3.4	50*
16.	Nitrite Nitrogen (NO ₂ -N) [#] , $mg L^{-1}$	45.1	9.2	-
17.	Nitrate Nitrogen (NO ₃ -N) [#] , $mg L^{-1}$	11.2	48.1	-

ND- Not Detectable. *Ammonical Nitrogen has been notified in the prescribed standard. #Standards not notified by regulator for textile sector.

Overall BOD and COD reduction achieved was 95 % and 91% respectively with formation of negligible non-toxic sludge ($0.4 kg m^{-3}$), with estimated overall operational costs around 1.55 USD/ m^3 and complete removal of total hardness, total nitrogen and mineralisation of organic nitrogen into inorganic Nitrogen along with significant reduction in chloride, TSS and TDS content. Major benefits of the overall scheme involves clean & green approach, toxic sludge free, scalable and robust technology with simultaneous disinfection. Bypassing bioremediation and involving around 5 hours in complete treatment ensures lower footprint and high efficiency. Additional advantages include tremendous reduction in use of chemicals, resources and operational costs. Proposed treatment is easily scalable for suitable field scale implementations due to the simplistic approach and design. Reuse of treated water as process water further ensures water re-use efficiency and implementation of Zero Liquid Discharge. Which is expected to reduce freshwater requirement and ensure sustainable management of water resources in water stressed regions of developing countries.

Findings from this study were published as Bahadur & Bhargava 2019 in **Journal of Water Process Engineering** under MS titled “**Novel pilot scale Photocatalytic Treatment of Textile & Dyeing Industry Wastewater to achieve Process Water Quality and enabling Zero Liquid Discharge**” [5]. This study shall be further discussed in detail under this chapter as per the outline as below:

6.1 Discussion on pollutant degradation

6.1.1 Effect on Physicochemical parameters

6.1.2 Effect on organic pollutants

6.1.3 Effect on trace metals

6.2 Computation of techno economic assessment of the implementation

6.2.1 Energy consumption

6.2.2 Cost of treatment

7 Studies on Real Effluent treatment from Common Effluent Treatment Plant using Photocatalysis and comparison with other Photochemical AOPs

Untreated mixed Effluent sample from a common effluent treatment plant running in a textile and dyeing cluster was obtained and treated using various photochemical AOPs. This batch-scale study of photochemical Advanced Oxidation Processes (AOP) to treat textile wastewater from a common effluent treatment plant to improve biodegradability and downstream performance. The assessment compared the techno-economic feasibility of integrating four photochemical AOPs with existing biological treatment plant in terms of their efficiency, energy requirement, and overall cost of treatment. Photochemical AOPs considered in this study were UV photolysis, UV/H₂O₂, UV photo Fenton, and UV/TiO₂ photocatalysis. Although every treatment improved the quality of treated water, UV/TiO₂ photocatalysis was the most promising for removing COD and BOD and required the least electrical energy per order (10.79 kWh/m³/order-COD removal and 5.16 kWh/m³/order-colour removal) whereas UV/TiO₂ was the most economic (0.77 US dollar or INR 59.75/m³). Fig 2 – 6 illustrate the treatment results from the study and the summary of techno economic analysis carried out in this study.

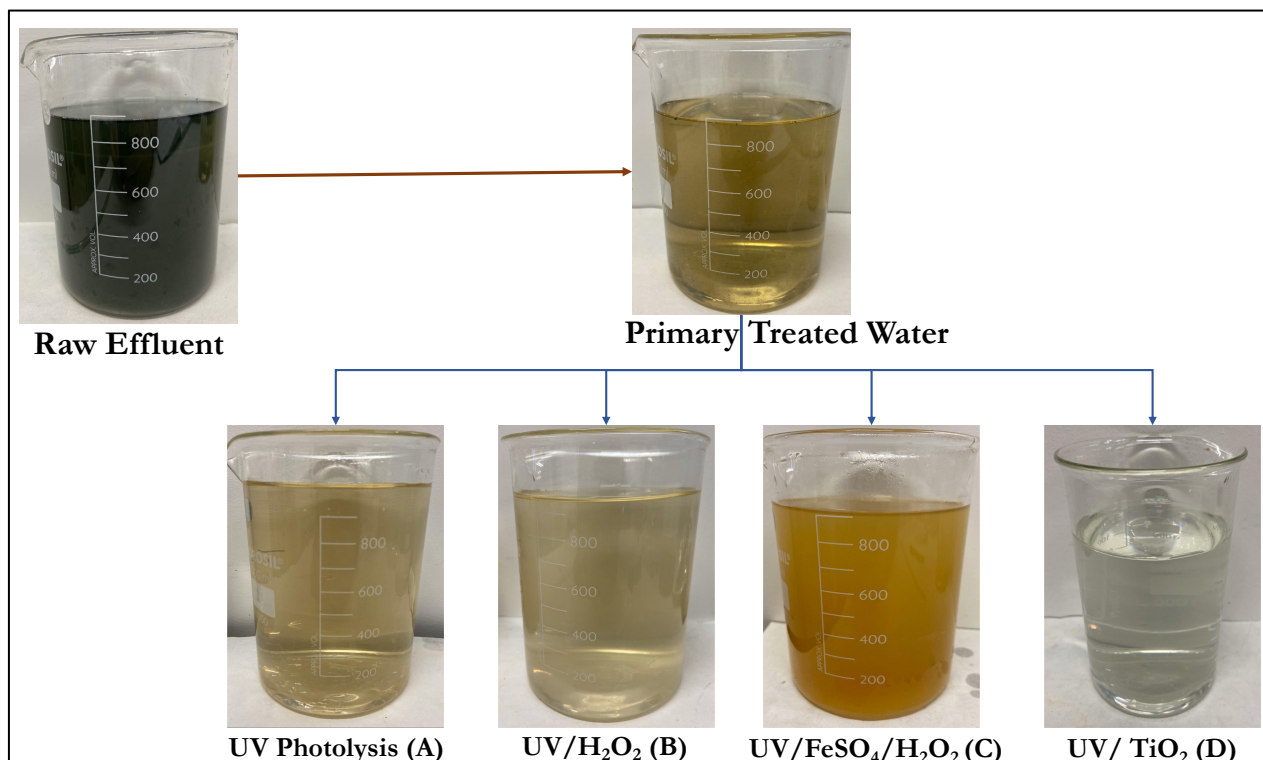


Fig 2. Untreated effluent and effluent after each of the four treatments, all based on advanced oxidation processes.

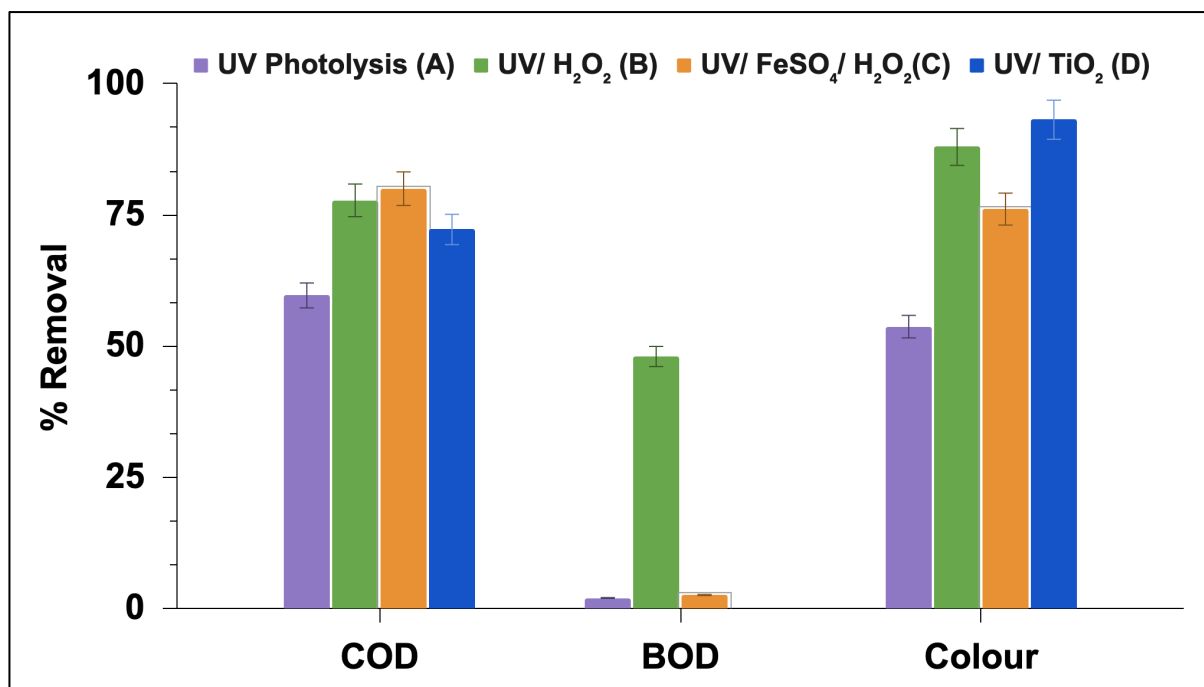


Fig 3. Reduction in chemical oxygen demand (COD), biological oxygen demand (BOD), and colour units following treatment with various advanced oxidation processes measured as a

percentage with respect to the levels in untreated effluent from textile units. Note: UV/TiO₂ treatment, instead of lowering BOD level, increased it instead.

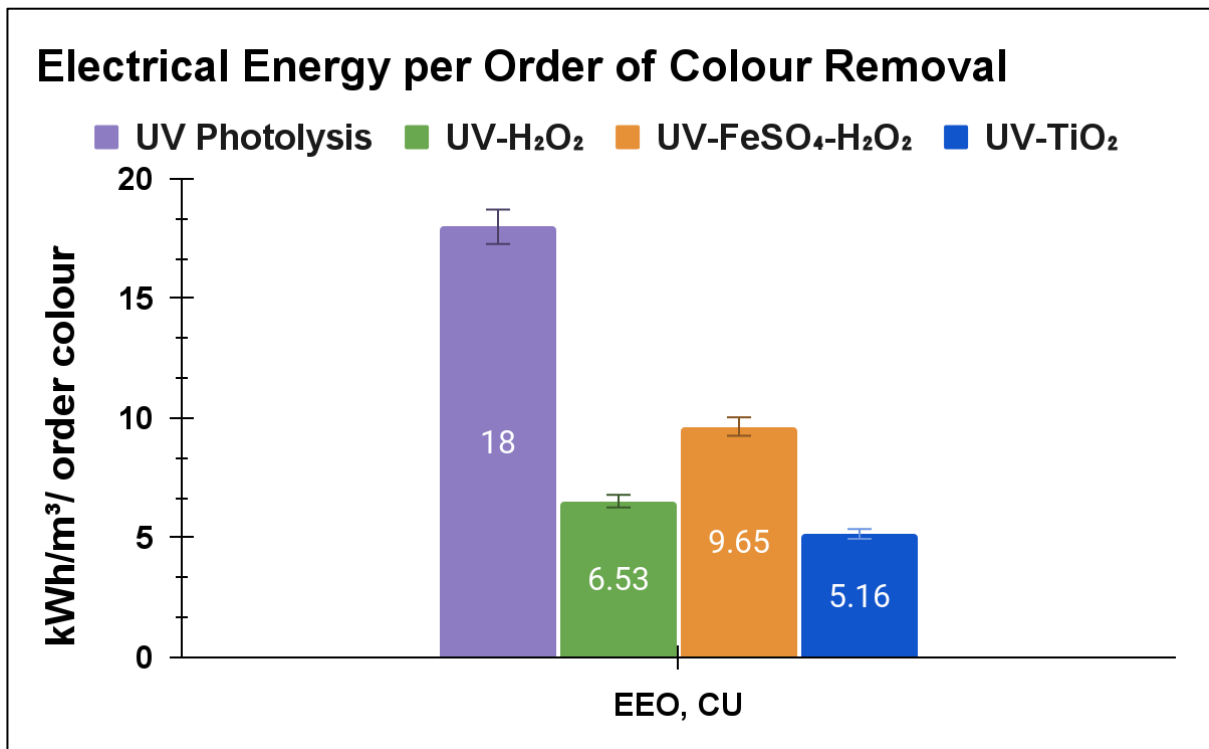


Fig 4. Comparison of EEO, CU for four photochemical AOPs subjected to the same sample in optimal conditions

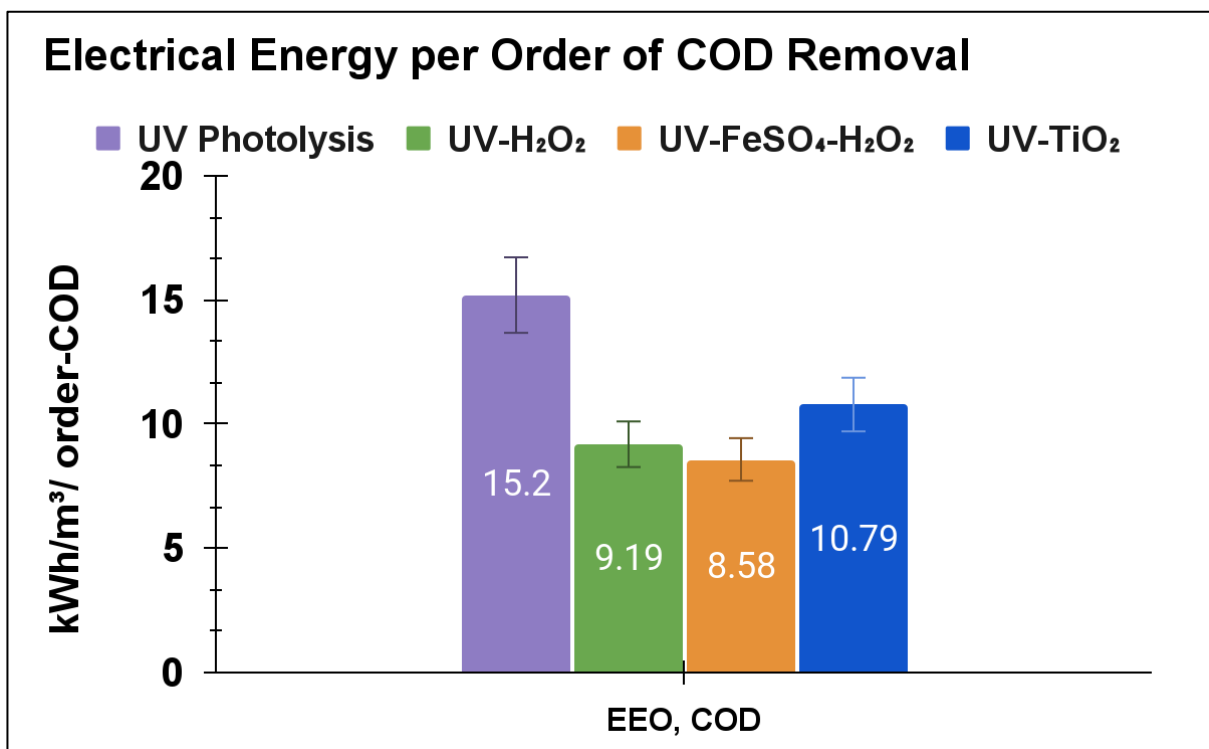


Fig 5. Comparison of EEO, COD for four photochemical AOPs subjected to the same sample in optimal conditions

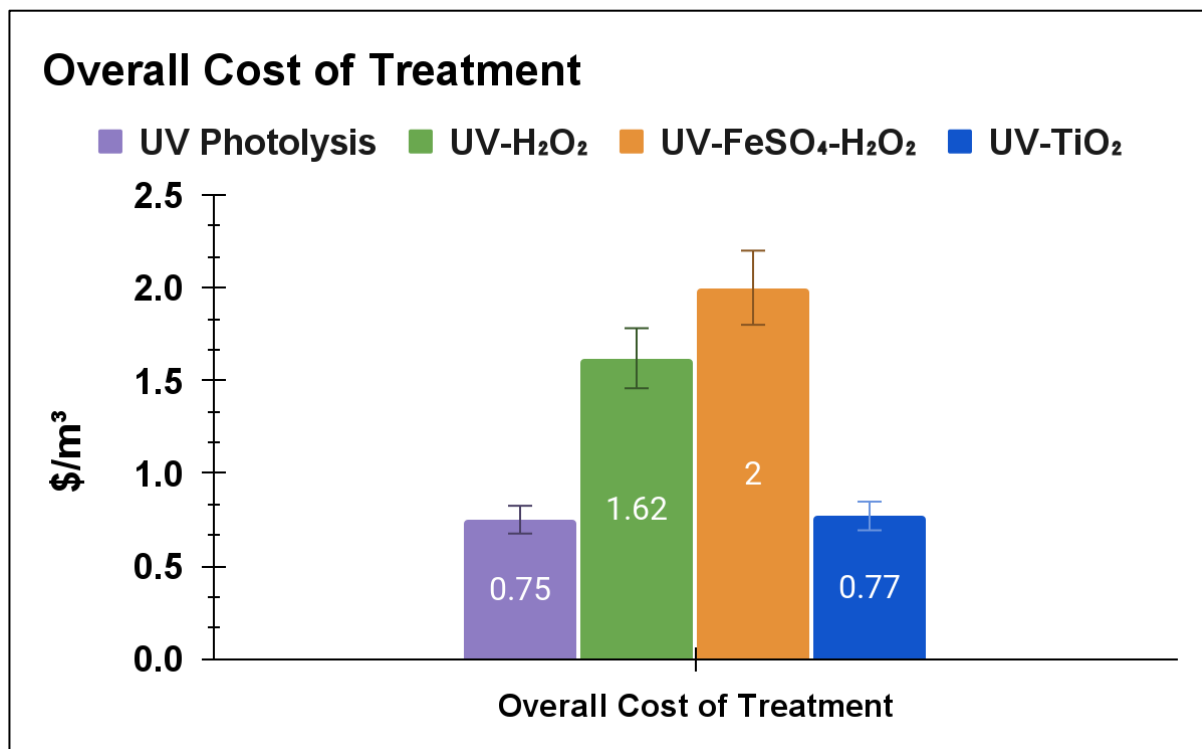


Fig 6. Comparison of Overall cost of the treatment for integration of photochemical AOPs at pre biological stage.

The findings from this study have been compiled into a manuscript and submitted to Journal of Water Process Engineering for consideration for publishing. This study shall be compiled in this chapter in detail and the outline of the chapter in sub headings is as under:

7.1 Optimisation of Primary Treatment for highest colour and TSS removal

7.1.1 Selection of coagulant and flocculant

7.1.2 Optimisation of dose of coagulant and flocculant

7.2 Discussion on pollutant degradation

7.2.1 Effect on Physicochemical parameters

7.2.2 Effect on organic pollutants

7.2.3 Effect on trace metals

7.3 Computation of techno economic assesment of the implementation

7.3.1 Energy consumption

7.3.2 Cost of treatment

8 Life Cycle Assessment of Conventional Treatment and Photocatalytically Integrated Treatment

This chapter illustrates a study carried out as per ISO 14064:2006 for carbon footprint assessment on a 'gate to gate' basis. Table 3 shows the results from the computation wherein, it is assumed that conventional treatment shall not allow for any reuse of water whereas the integration of photochemical AOPs shall enable 50% reuse of treated water and balance treated water maybe discharged on ground.

Table 3. Life Cycle mid points impacts for Global warming Potentials (GwP) computations - conventional and photochemical AOPs with 50% treated water as reuse and balance as discharge to land.

Type of System Parameter	Case I - Conventional	Case II- AOP Integrated (with Discharge)			
	Conventional	A. UV Photolysis	B. UV H ₂ O ₂	C. UV H ₂ O ₂ FeSO ₄	D. UV TiO ₂
A. Energy Consumption	2.56	6.86	5.84	6.09	5.59
B. Residual Pollutants in Outlet: (COD, BOD, TN removal)	1.1	0.75	0.38	0.50	0.26
C. Direct Emissions: (CO ₂ , CH ₄ and N ₂ O)	8.1	6.19	5.13	8.94	6.48
D. Transportation (Computed from chemical dose)	1.1	0.55	0.31	0.62	0.05
E. Chemical consumption (Production of chemical and its fate in environment)	0.45	0.23	1.17	1.71	0.49
F. Total CO₂ Eq. (A+ B+ C+ D+ E)	<u>13.31</u>	<u>14.58</u>	<u>12.83</u>	<u>17.87</u>	<u>12.86</u>
% Reduction	-	<u>-9.51</u>	<u>3.64</u>	<u>-34.23</u>	<u>3.36</u>

Table 4 tabulates the results of a ZLD approach with conventional treatment system involving tertiary treatment involving 3 stage RO plant and multiple effect evaporator operating on coal fired boiler; whereas in case of the photochemical integrated ZLD compliant plant requires 50% water to be sent for tertiary treatment.

Table 4. Life Cycle mid points impacts for Global warming Potentials (GWP) computations - conventional and photochemical AOPs with 100% treated water reuse i.e. compliant to zero liquid discharge

Type of System	Case III -	Case IV - AOP Integrated with ZLD			
	Conventional with ZLD	A. UV	B. UV	C. UV H ₂ O ₂	D. UV
Parameter	Conventional with ZLD	A. UV Photolysis	B. UV H ₂ O ₂	C. UV H ₂ O ₂ FeSO ₄	D. UV TiO ₂
A. Energy Consumption	2.56	6.86	5.84	6.09	5.59
B. Residual Pollutants in Outlet: (COD, BOD, TN removal)	1.1	0.75	0.38	0.50	0.26
C. Direct Emissions: (CO ₂ , CH ₄ and N ₂ O) -	8.1	6.19	5.13	8.94	6.48
D. Transportation (Computed from chemical dose)	1.1	0.55	0.31	0.62	0.05
E. Chemical consumption (Production of chemical and its fate in environment)	0.45	0.23	1.17	1.71	0.49
F. Tertiary Treatment Unit	21.4	19.251	12.834	11.7645	10.695
G. Total CO₂ Eq.	<u>34.70</u>	<u>33.83</u>	<u>25.66</u>	<u>29.63</u>	<u>23.56</u>
(A+ B+ C+ D+ E+F)					
% Reduction	-	<u>2.51</u>	<u>26.05</u>	<u>14.61</u>	<u>32.11</u>

The above data clearly shows that PC resulted in the most sustainable integration with discharge of treated water with 3% reduction in carbon footprints whereas in case of ZLD compliant system, PC resulted in a significant 32% reduction in carbon footprint.

The details of the chapter shall include

8.1 Derived Material Balance

8.1.1 Conventional plant flow and load balance

8.1.2 PC Integrated plant flow and load balance

8.2 Data Analysis

8.3 Comparison of PC integration vs Conventional Treatment

9 Conclusions & Way Forward

This section shall summarise the findings of the research and the future work in the domain area.

10 List of Expected journal publications and conference papers

International Papers

1. Article under consideration in Journal of Water Process Engineering (JWPE) MS no. JWPE-D-23-01629:- **Nipun Bhargava**, Nupur Bahadur*, Arun Kansal; Tentatively titled- 'Techno-economic assessment of integrated advanced oxidation processes for intensification of biological treatment systems to enhance water reuse in textile clusters', 2023. (Minor revisions received by reviewers and response submitted to editor)
2. Nupur Bahadur*, **Nipun Bhargava**, Novel pilot scale photocatalytic treatment of textile & dyeing industry wastewater to achieve process water quality and enabling zero liquid discharge, Journal of Water Process Engineering, Volume 32, 2019, 100934, ISSN 2214-7144, <https://doi.org/10.1016/j.jwpe.2019.100934>. [IF 7, 53 Citations]
3. Nupur Bahadur*, Paromita Das, **Nipun Bhargava**, Improving energy efficiency and economic feasibility of photocatalytic treatment of synthetic and real textile wastewater using bagasse fly ash modified TiO₂, Chemical Engineering Journal Advances, Volume 2, 2020, 100012, ISSN 2666-8211, <https://doi.org/10.1016/j.ceja.2020.100012>. [IF 15.1 (Mirror for CEJ), 9 Citations]

Conference Papers

1. Sustainable Treatment of Textile and Dyeing Wastewater using AOP – ICBASET 25/08/22
2. Integration of Photocatalysis To Improve Biodegradability For Enhancing Water Reuse Efficiency In Textile Wastewater Treatment – Institution of Engineers (India) - 05/08/22
3. Potential For Improvement In Water Management Of Textile And Chemical Industries In India – Institution of Engineers (India) - 07/06/2022

11 Thesis structure

Chapter 1: Introduction

This chapter shall briefly explain the present challenges in textile sector pertaining to environment, specific wastewater treatment issues, present norms of the industry and the respective pain points. Hence this will give a background of the research and justification of the proposed study. Organisation of the thesis shall also be given in this chapter

Chapter 2: Aim and objectives

Aim, objectives, relevance and scope of the study shall be enlisted in this chapter.

Chapter 3: Literature Review

The chapter shall provide a systematic review of the work done in the subject area.

Chapter 4: Materials and Methods

The Chapter shall provide the details of the treatment technology, engineering details of the PC based pilot plant, methodology of operation, optimisation and maintenance of the plant, complete details of the methods utilised for characterisation of nanomaterials, effluent, sludge etc. It will also contain the basis, formulae and approach towards computation of figures of merit, cost of treatment and life cycle assessment studies on photocatalysis as a standalone, integrated approach for achieving ZLD in textile sector.

Chapter 5: Optimisation of Pilot Plant using simulated Wastewater/ Synthetic Dye Bath

This chapter shall contain the initial optimisation studies on operating parameters like pH, TiO₂ dose, Oxidant dosage, UV irradiation time, characterisation of spent nanomaterials, optimisation of nanomaterial recovery unit, reuse of nanomaterials, comparison of various TiO₂ materials etc. and rate kinetics of the degradation. The chapter shall also include the estimated electrical energy consumption per order COD, Colour removal and BOD reduction (EEO COD Colour and BOD respectively) with a tentative cost estimation of the end to end treatment.

Chapter 6: Treatment of Real Effluent from Individual ETP of Textile and Dyeing industry

This chapter shall contain the studies done on a real effluent from cotton dyeing unit from AP, India; the studies contain the detailed characterisation of the obtained sludge, treated effluent, compliance to textile industry norms with associated techno economic feasibility in terms of EEO COD, EEO Colour and overall cost of treatment.

Chapter 7: Treatment of Real Effluent from Common ETP of Textile and Dyeing Industry

This chapter shall detail out the study of PC application on effluent from common ETP and its associated comparison with other photochemical AOPs in terms of degradation of COD, Colour removal, electrical energy per order in terms of COD and Colour; and overall cost of treatment. Finally, an implementation model is given at the end along with tentative material balances to aid future studies in the subject area.

Chapter 8: Life Cycle Assessment of Conventional Treatment and Photo-catalytically Integrated Treatment

This chapter extends the work presented in chapter 7 and details a LCA study carried out on comparing the midpoint impacts of the global warming potential of conventional wastewater treatment in integration with various photochemical AOPs for complying the discharge norms and/ or achieving the zero liquid discharge norms.

References, Annexure, List of Publications

At the end a detailed section on references, annexure and list of publications shall be given.

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