Advanced Oxidation of ECF Bleaching Wastewater Using TiO_2 Photocatalysis

P. Kumar, S. Kumar, and N. K. Bhardwaj

***Abstract*—Advanced oxidation of ECF (elemental chlorine free) bleaching wastewater (D_1 and E stage of DED and ODED sequences) using TiO_2 photocatalysis has been investigated for environmental load reduction. The wastewaters were generated in the laboratory by chlorine dioxide (D) bleaching and alkaline extraction (E) of the mixed hardwood kraft pulp under controlled conditions. The bleaching wastewaters were subjected to photocatalytic oxidation under UV radiation with 0.5 g/L of TiO_2 at a pH 7.0 for 4 h in a slurry-type reactor. Higher removal efficiencies have been observed for ODED sequence wastewaters as compared with DED sequence. UV/TiO_2 oxidation removed 46.3% BOD (biochemical oxygen demand), 64.2% COD (chemical oxygen demand), 88.3% color, and 59.3% AOX (adsorbable organic halides) for D_1 and 55% BOD, 66.5% COD, 86.7% color, and 64.2% AOX for E stage wastewaters of ODED sequence. Higher improvement in BOD/COD ratio has been observed for ODED sequence wastewaters as compared with DED. This shows that wastewaters from ODED sequence are more amenable to photocatalytic degradation.**

***Index Terms*—Advanced oxidation, AOX, biodegradability, TiO_2 photocatalysis.**

I. Introduction

The pulp and paper sector comes under twelve most polluting industries in India due to the huge quantity and quality of wastewater generated [1]. Among the various sections, the wastewaters from pulp bleaching are responsible for most of the color, organic matter, and toxicity of the water discharges of this industry [2]. The pulp produced by chemical pulping requires bleaching to produce bright pulps. These operations generate high environmental load, i.e. BOD (biochemical oxygen demand), COD (chemical oxygen demand), color, and various chloroorganics (chlorinated phenols, resin and fatty acids, and dioxins and furans originating from lignin and/or extractions of wood) in the bleach plant wastewater, which are collectively termed as AOX (adsorbable organic halides). Some of these chloroorganics are bioaccumulative, toxic, mutagenic, and resistant to biodegradation. Hence these compounds pose a serious threat to the environment [3]. AOX in the bleaching wastewater originates from chlorination of residual lignin in the pulp. Thus, prebleaching step, i.e. O_2 delignification, which decreases the incoming pulp kappa number, decreases AOX correspondingly. The replacement of molecular chlorine by chlorine dioxide in the ECF (elemental chlorine free) bleaching process decreases the amount of chlorinated organics formed [4].

The conventional wastewater treatment processes are not effective for the complete degradation of color and low molecular weight chloroorganics [5]. Hence, there is an urgent need to adopt some advanced treatment methods i.e. advanced oxidation processes (AOPs) for meeting increasingly stringent wastewater discharge standards. AOPs rely on the in-situ generation of very reactive oxidizing species, i.e. hydroxyl radicals ($OH^{\bullet}$), for the degradation of organic compounds [6]. Their production can be accelerated by combining various systems, i.e. UV/catalyst, UV/catalyst/H_2O_2 (semiconductor photocatalysis), O_3 (ozonation), UV/O_3, UV/H_2O_2, O_3/H_2O_2, UV/O_3/H_2O_2, UV/O_3/catalyst, Fe(II)/Fe(III) with H_2O_2 (Fenton reaction), and UV/{Fe(II)/Fe(III)+H_2O_2}(photo-Fenton) [7]. Among various AOPs, semiconductor photocatalysis is an attractive technique because it causes the complete mineralization of a wide range of organics without any harmful environmental impact [8]. TiO_2 is the most widely used semiconductor photocatalyst for wastewater treatment because it is chemically and biologically inert, photo-stable over a wide range of pH, low cost, and relatively easy to produce and use [6].

II. Mechanism of Photocatalysis

The photocatalysis is based on the absorption of light radiation by semiconductor particle. When a photon of light ($h\nu$, $\lambda < 390$ nm) strikes the catalyst surface, an electron is raised from the valence band (vb) to the conduction band (cb) leaving behind a hole (h^+_{vb}) (1). The h^+_{vb} can either directly oxidize a wide range of adsorbed pollutants or produce $OH^{\bullet}$ radicals (from H_2O/OH^- ion) (2 and 3) which can also oxidize organics non-selectively (5 and 6). The electron (e^-_{cb}) is readily taken by adsorbed O_2 to produce superoxide radical ($O^{\bullet-}_{2ads}$) (4), thus prevent e^-_{cb}/h^+_{vb} re-combination. The $O^{\bullet-}_{2ads}$ radical can further participate in contaminant degradation reactions [6], [9].

$$TiO_2 + h\nu \rightarrow e^-_{cb} + h^+_{vb} \quad (1)$$

$$h^+_{vb} + H_2O_{ads} \rightarrow OH^{\bullet}_{ads} + H^+ \quad (2)$$

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$$h_{vb}^{+} + OH_{ads}^{-} \rightarrow OH_{ads}^{\bullet} \quad (3)$$

$$e_{cb}^{-} + O_{2ads} \rightarrow O_{2ads}^{\bullet -} \quad (4)$$

$$OH^{\bullet} + Contaminants \rightarrow Intermediates \quad (5)$$

$$Intermediates \rightarrow CO_2 + H_2O + end - products \quad (6)$$

TiO_2 as a photocatalyst has been successfully utilized for the remediation of pulp and paper mill wastewater during the recent years. Reference [10] reported that photocatalysis can efficiently reduce the organic load from ECF bleaching wastewater. About 80% AOX, 64% COD, and 72% color removal was reported with UV/TiO_2/O_2 process. Reference [11] reported increase in the biodegradability of the remaining pollutants in bleaching wastewater after photocatalytic oxidation. Reference [12] reported about 50% removal of both toxicity and COD from bleached kraft pulp wastewater with supported TiO_2. Reference [13] reported that photocatalysis is efficient for the reduction of AOX (95%), TOC (total organic carbon) (50%), total phenols, and toxicity from the cellulose bleaching wastewater. Reference [14] found out that photocatalysis with TiO_2 is an effective method for the degradation of COD and toxicity from pulp and paper mill wastewater with initial COD concentration of 500 mg/L or less. Reference [15] investigated that sequential, biological, and photocatalytic treatment resulted in about complete degradation of bleaching wastewater in terms of COD, color, and chlorophenols.

The present work is aimed to study the advanced oxidation of ECF bleaching wastewater (D_1 and E stage of DED and ODED sequences) with UV/TiO_2 process for environmental load reduction.

III. Experimental

A. Wastewater and Reagents

The wastewaters for photocatalytic oxidation studies were generated in the laboratory by the bleaching of mixed hardwood kraft pulp through DED and ODED sequences. The first chlorine dioxide (D_1) and alkaline extraction (E) stage wastewaters were subjected to photo-degradation. Unbleached mixed hardwood kraft pulp of kappa number 15 was procured from a paper mill in India. The pulp was hand washed with plenty of water, screened, air dried, and stored in air tight polythene bags for further bleaching studies. Titanium dioxide (TiO_2) was obtained from Fisher Scientific (SQ grade). Other reagents and chemicals used were of analytical grade and used without any further purification. The pH of the pulp and aqueous solutions were adjusted with 1M H_2SO_4 or 1M NaOH solutions. Chlorine dioxide was produced in situ from sodium chlorite ($NaClO_2$). The NaOH solution was used for alkaline extraction of pulp.

B. Bleaching

Bleaching of the pulp was carried out to 87% ISO target brightness with the sequences DED and ODED; where D, E, and O refer to chlorine dioxide oxidation, alkali extraction with NaOH, and oxygen delignification stages, respectively. The pulp bleaching conditions for various stages are listed in Table I. Fig. 1 depicts the flow diagram of pulp bleaching process for DED and ODED sequences. Before bleaching and oxygen delignification, the air dried pulp was soaked in water and disintegrated at 3% consistency for 4-5 min using a pulp disintegrator. The dispersed pulp was then filtered and stored in refrigerator for further bleaching studies. Oxygen delignification was performed in the laboratory autoclaves revolving in hot ethylene glycol bath. Calculated amount of NaOH solution and $MgSO_4$ were added to 100 g O.D. (oven

TABLE I: Pulp Bleaching Conditions for Various Stages.

Parameters	DED Sequence			ODED Sequence			
	D_1	E	D_2	O	D_1	E	D_2
Kappa no.	15			15 (after O stage 8.3)			
Kappa factor	0.35			0.30			
Cl_2 demand (%)	70	-	30	-	70	-	30
NaOH (% O.D. pulp)	-	0.7	-	2	-	0.7	-
Consistency (%)	10	10	10	10	10	10	10
End pH	3.4	11.5	3.5	-	3.5	11.4	3.2
Temperature (°C)	70	70	70	100	70	70	70
Time (h)	180	90	180	75	180	90	180
O_2 Charge (kg/cm^2)	-	-	-	6	-	-	-
$MgSO_4$ (%)	-	-	-	0.2	-	-	-
Brightness (% ISO)	87			87			

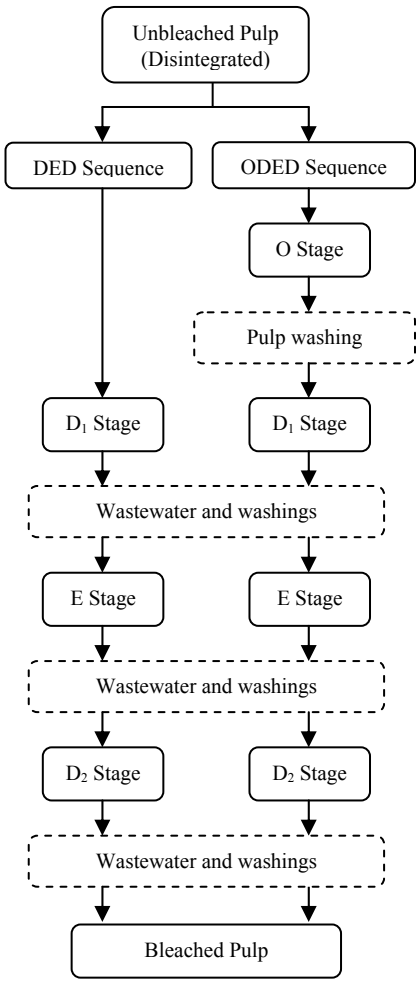

Fig. 1. Flow chart of the pulp bleaching process.

dried) pulp, hand mixed, transferred to autoclaves, and put in ethylene glycol bath. After oxygen stage, pulp was washed with plenty of water on a screen, squeezed, air dried, and stored in polythene bags for further bleaching experiments. The oxygen delignification optimization was targeted at a kappa number drop between 40-50%. Chlorine demand on the pulp was calculated using (7):

$$\text{Chlorine demand (\%)} = \text{kappa no.} \times \text{kappa factor} \quad (7)$$

All the bleaching stages were performed in polythene bags with 200 g O.D. pulp in two sets (100 g each). The pulp was adjusted to desired consistency and pH. Bleaching chemicals were added and well kneaded by hand mixing from time to time during bleaching. After completion of the reaction, the pulp slurry was filtered and filtrate was collected. Then the pulp (100 g O.D.) was washed with 900 (3×300) mL of distilled water after each bleaching stage. The filtrate and washings of both set (each 100 g O.D.) were mixed and used for characterization and wastewater treatment studies. All the experiments were performed in duplicate and average values reported.

C. Photoreactor

The photodegradation experiments were carried out in a borosilicate glass bowl (1L). The glass bowl containing the reaction mixture was placed in a timber-framed UV reactor (77 cm × 36 cm × 71 cm) equipped with 4 UV tubes (λ = 365 nm) each of 18 W (Philips) on the top side, located at a distance of 15 cm from the sample (Fig. 2). The reaction mixture was subjected to irradiation under UV light with continuous stirring. A fan fitted on the side wall was used to lower the heat generated by UV lamps. All the experiments were carried out in completely mixed and batch mode.

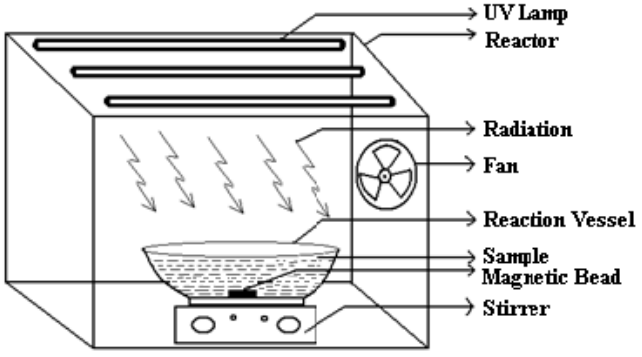

Fig. 2. Photoreactor for bleaching wastewater advanced oxidation.

D. Advanced Oxidation

A 500 mL aliquot of pulp bleaching wastewater was adjusted to pH 7.0 and transferred to the glass bowl. 0.5 g/L of TiO_2 was added and the aqueous suspension magnetically stirred for 30 min before switching on the UV lamps. The experiments were carried out at ambient conditions. After completion of the reaction, water loss was made up by distilled water and allowed to settle overnight. The supernatant was collected and TiO_2 agglomerates removed by centrifuging before analysis. The rate of degradation was quantified in terms of the percentage removal of COD, BOD, color, and AOX after photocatalysis (8). The change in biodegradability index (BOD/COD) of the wastewaters was also investigated for assessing the biodegradability of the treated wastewaters.

$$\text{Degradation (\%)} = [(C_0 - C)/ C_0] \times 100 \quad (8)$$

where

C_0 = Concentration of pollutants before photo-degradation.

C = Concentration of pollutants after photo-degradation.

E. Analytical Techniques

The following standard analytical procedures were followed for pulp characterization: kappa number of pulp - Tappi T 236, disintegration of chemical pulps - SCAN C 18: 65, forming hand sheets for physical testing of pulp - SCAN C 26: 76, brightness of pulp - ISO Standard 2469, preparation of indicators and standard solutions - T 610 om-87. The analysis of the bleach liquor and residual chlorine was carried out according to the standard procedure. The wastewater was characterized before and after photocatalysis in accordance with the Standard Methods [16]. COD was determined by closed reflux titrimetric method and BOD by measuring dissolved oxygen before and after incubation at 20 °C for 5 day. The color was measured at 465 nm using a UV-VIS double beam spectrophotometer (SPEKOL 2000, Analytic Jena). A bench scale pH meter (TOSHNIWAL) was used to measure the pH. Adsorbable organic halides were analyzed on the AOX analyzer ECS 1200 using column method.

IV. Results and Discussion

The average bleaching wastewaters characteristics utilized for the present study are summarized in Table II. The D_1 and E stage wastewaters of DED sequence presented high organic load in terms of BOD, COD, color, and AOX as compared to ODED sequence. The difference in the pollution load of the wastewaters is due to the reduction (44.7%) of initial kappa number (i.e. residual lignin) of the pulp in ODED sequence after O_2 stage, which in turn reduces the active chlorine multiple required for the next bleaching stages and environmental load [4]. The presence of chlorinated organics (AOX) renders these wastewaters unsuitable for biological degradation. Hence, there is a need for advanced chemical oxidation for degrading bio-refractory organics. According to the literature, for the successful catalysis, the initial COD of the wastewater should be below 800 mg/L because excess of the organic matter tends to recover the catalyst surface through adsorption and causes scattering of light radiation [17]. Hence, D_1 and E stage wastewaters of DED sequence were diluted to bring the initial COD values to approximately 500 mg/L before photocatalytic oxidation while ODED sequence wastewaters were used directly.

The optimum treatment conditions for photocatalytic degradation of pulp and paper mill wastewater were searched out in the laboratory using UV/TiO_2 process. It was observed that photo-catalysis can efficiently degrade the organics from the pulp and paper mill wastewater. The best experimental conditions obtained were: pH 7.0, 0.5 g/L of TiO_2, and reaction time of 4 h [18]. The D_1 and E stage wastewaters of

DED and ODED sequences were subjected to photocatalytic degradation under these optimized conditions.

TABLE II: CHARACTERISTICS OF THE BLEACHING WASTEWATERS

Parameters	DED Sequence			ODED Sequence		
	D_1	E	D_2	D_1	E	D_2
COD (mg/L)	1053	853	200	431	340	106
BOD (mg/L)	253	205	60	121	99	51
AOX (mg/L)	21.3	18.7	4.7	10.7	9.3	2.6
Color (mg Pt/L)	1040	1227	86	122	133	62
pH	3.4	11.5	3.5	3.5	11.4	3.2

The degradation of organic matter present in the wastewaters was monitored in terms of COD removal. The wastewaters were pre-equilibrated with TiO_2 for 30 min before illumination. About 8.2 and 7.6% COD removal for D_1 and 8.9 and 7.1% COD removal for E stage wastewaters of DED and ODED sequences, respectively, was observed prior to photocatalysis. This may be due to the initial dark adsorption of organic matter on the catalyst surface [10]. After 4 h of treatment, 49.8 and 64.2% COD removal for D_1 stage, and 61.9 and 66.5% COD removal for E stage wastewaters of DED and ODED sequences, respectively, was achieved, Fig. 3. A higher COD removal efficiency was obtained for ODED sequence wastewaters as compared to DED sequence. The difference in COD removal for different wastewaters may be due to the difference in the molecular weight and structure of the dissolved organic pollutants present in the wastewaters and organic load [11]. E stage wastewaters of both the sequences were more amenable to photodegradation when compared with D_1 stage wastewaters.

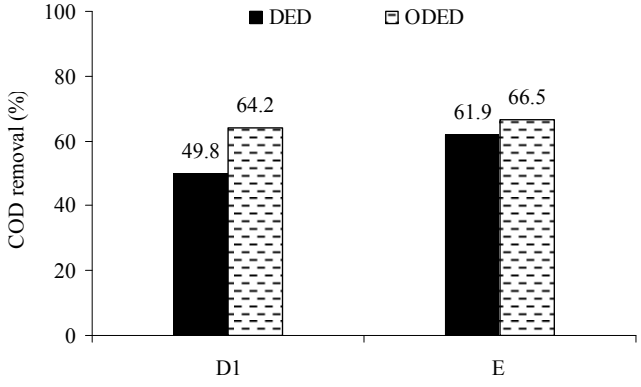

Fig. 3. COD removal (%) for D_1 and E stage wastewaters of DED and ODED bleaching sequences.

A noticeable removal of BOD was observed for all the wastewaters, probably due to the photochemical degradation of the biodegradable species and low toxicity of the wastewaters [19]. After 4 h of treatment, 33.2 and 46.3% BOD removal for D_1 stage, and 47.6 and 55% BOD removal for E stage wastewaters of DED and ODED bleaching sequences, respectively, was obtained, Fig. 4.

These findings are in concordance with the literature [19], [20]. The biodegradability of the pollutants present in the wastewaters was estimated in terms of BOD/COD ratio. The initial biodegradability index of the wastewaters was low i.e. 0.24 and 0.28 for D_1 and 0.24 and 0.29 for E stage wastewaters of DED and ODED sequences, respectively. For complete biodegradation, the wastewater must have a biodegradation index of at least 0.40 [21]. As can be seen in Fig. 5, significant improvement in biodegradability index has been observed for ODED as compared with DED sequence, the values being 0.08 and 0.14 points for D_1 stage, 0.09 and 0.1 points for E stage wastewaters of DED and ODED sequences, respectively. These observations are in accordance with those reported earlier i.e. titanium dioxide photocatalysis increases the biodegradability of organic matter present in the wastewater [22]. For oxygen delignified pulp bleaching wastewaters the biodegradability raised over the optimal values under advanced oxidation conditions.

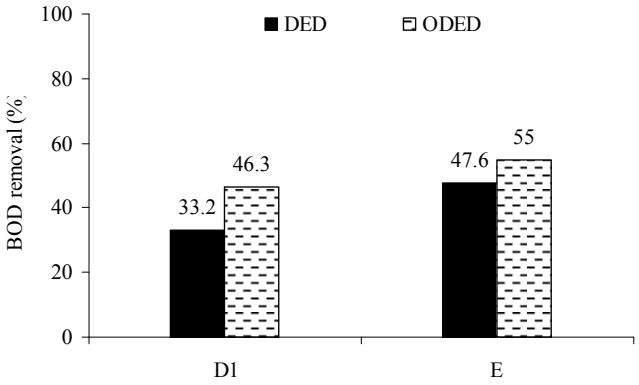

Fig. 4. BOD removal (%) for D_1 and E stage wastewaters of DED and ODED bleaching sequences.

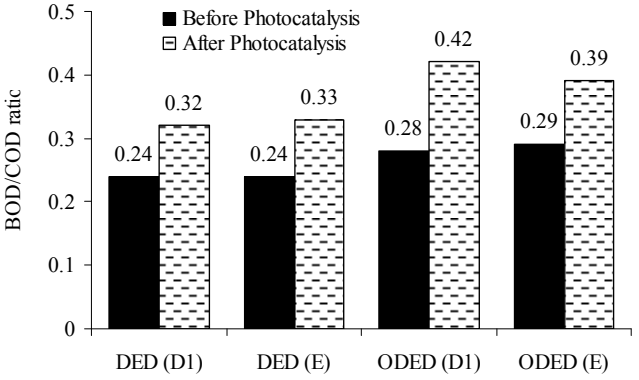

Fig. 5. Change in BOD/COD ratio for D_1 and E stage wastewaters of DED and ODED bleaching sequences.

The brown color in the bleaching wastewaters is primarily due to the presence of lignin and it's derivatives (high molecular weight substances) that are released from the substrate during bleaching [23]. Fig. 6 depicts the color removal efficiency of titanium dioxide photocatalysis for DED and ODED sequence wastewaters. Color of ODED wastewaters was already very low as compared to DED sequence. According to the literature oxygen pre-bleaching decreases the color of bleaching wastewaters by 63-80% [24]. For ODED sequence wastewaters more color removal was observed, i.e. 88.3% for D_1 and 86.7% for E stage wastewater, as compared with DED (78.1% for D_1 and 77.2% for E stage) sequence after photocatalysis. Heterogeneous photocatalysis has been reported to be most efficient for the discoloration of wastewaters [20].

AOX is an approximate measure of the chlorinated organics present in the pulp bleaching wastewaters and toxicity, which must be reduced in any wastewater treatment system [4]. After 4 h of treatment, AOX removal obtained was 43.9% for D_1 and 50.1% for E stage wastewater of DED and 59.3% for D_1 and 64.2% for E stage wastewater of ODED sequence, Fig. 7. Reference [22] reported 85 and 90% removal of AOX, after 60 and 120 minutes of reaction, respectively, for eucalyptus E_1 wastewater using immobilized catalysts. They emphasized that the high reduction in a short period of treatment must be a consequence of the high irradiation power of the lamp. In our case the low removal efficiencies may be due to the low irradiation power of the lamps.

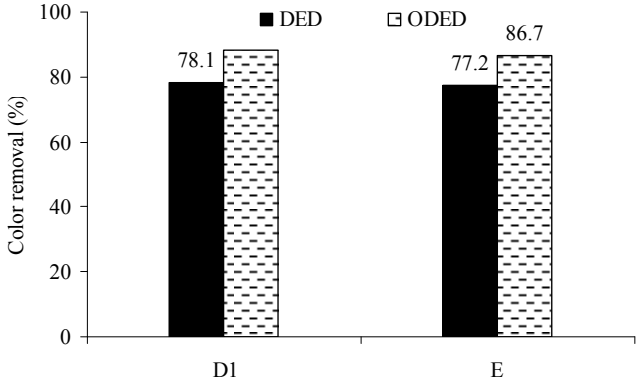

Fig. 6. Color removal (%) for D_1 and E stage wastewaters of DED and ODED bleaching sequences.

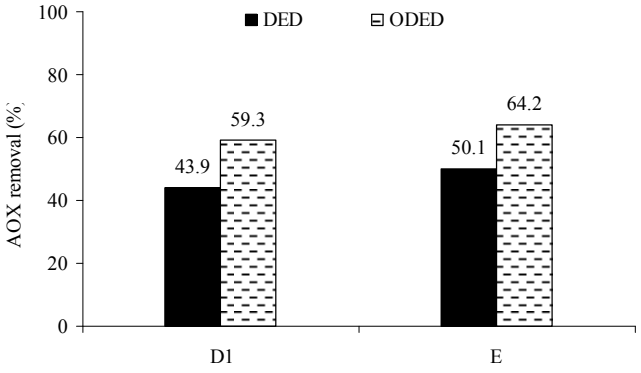

Fig. 7. AOX removal (%) for D_1 and E stage wastewaters of DED and ODED bleaching sequences.

V. Conclusion

Experimental results indicate that TiO_2 photocatalysis can be effectively utilized for the remediation of pollutants from pulp bleaching wastewaters. Higher pollutants removal efficiency was achieved for ODED sequence wastewaters as compared with DED sequence. The biodegradability index of the wastewaters improved after photocatalysis. This shows easy removal of pollutants during biological treatment. The photocatalysis has a strong potential for large scale industrial application by utilizing solar light as an economical irradiation source. To achieve this goal further research is needed in the areas of catalyst development, reactor designing, and interaction of pollutants, catalyst, radiation, and oxidant.

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