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Experimental Investigation on Photocatalytic Degradation of Refractory Organics in Biologically Treated Tannery Effluent Using Photocatalysis

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ABSTRACT

There is a pressing demand for the introduction of environmentally safe technologies for the industries that supply the basic needs of industrialized societies. Advanced Oxidation Processes may become one of the answers to these uprising pollution management problems in the near future. The present investigation aimed to reduce the refractory organics present in the biologically treated (Activated Sludge Process) tannery effluent using Photocatalysis. The optimum time, pH, dosage of H_2O_2, and mass of NPAC required for the effective treatment using photocatalysis were found to be 60 mins, 8, 0.2 $mg.L^{-1}$, and 1g. 100 mL^{-1}, respectively. Although the efficiency of homogeneous photocatalysis was found to be higher than that of heterogeneous photocatalysis, the biodegradability was higher in the latter, with a value of 0.26. The experimental results have proved that photocatalysis could be a promising technology to reduce the refractory organics present in the tannery effluent.

INTRODUCTION

Industrialization has taken up a rapid pace of growth in recent times due to the growing needs of the population, urbanization, and technological advancements. The leather processing industry is one of the major economic shareholders, with an approximated annual worldwide leather production of 1.67 billion m^2 amidst a total trade value of around seventy million Dollars per year. South Asia is one of the major contributors to this production, satisfying approximately 20% of the world's demand in total (Rao et al. 2003). It is understood that, unlike other industries, leather processing is said to be highly water intensive and also the major contributor to water pollution (Subramani & Haribalaji 2012, Haydar et al. 2007, Mannucci et al. 2010), producing 30-35 m^3 of wastewater per tonne of raw hide on average. However, the quantity and quality of wastewater are highly variable based on the characteristics of raw materials used in the process (Tunay et al. 1995). The processing of leather in tanneries generates an enormous amount of organic and inorganic waste materials, namely phenolic compounds, metals, dyes, etc., as effluents. The effluent from tanneries has been thus identified as the main factor for environmental degradation affecting aquatic life, flora, and fauna. It also causes severe illnesses in human beings (Shakir et al. 2012), such as hepatocellular cancer, sperm damage, feto-maternal death, etc., Due to these towering problems, the treatment of tannery effluent has gained more emphasis than ever before. Apart from serious environmental and health issues, the presence of recalcitrant organic substances in these effluents renders their treatment incomplete and poses a serious ecological issue when it is continuously let into the water and soil environment.

Recalcitrant organic is a general term that represents all organic substances that are resistant to bio-degradation. The biological treatment processes were found ineffective in the complete removal of residual organics, whereas the membrane treatment processes show drawbacks of membrane fouling and failure due to the dye molecules and other impurities present in the tannery effluent. The development of an economical method for the removal of low-concentration refractory organics in industrial wastewater is a difficult environmental challenge (Ran et al. 2021). In particular, for the treatment of wastewater's refractory organic constituents, scientific treatment approaches should be matched with the demands of the wastewater treatment technology. In other words, only via the scientific treatment of refractory compounds in wastewater, can the quality of wastewater treatment be improved (Runzhe 2019). Such treatment requires sustainable methods to minimize environmental impact through resource recovery, recycling, and reuse. One of the sustainable new technologies for treating refractory organic pollutants found in various

industrial wastewaters, such as those from paper and pulp, pharmaceuticals, petrochemicals, refineries, and textiles is the advanced oxidation process (AOP) (Manna & Sen 2022). AOP has gained attention in the past few years for the effective treatment of tannery effluents. The process involves complete mineralization of both organic as well as inorganic compounds. Owing to this reason, AOP is preferred to be used in conjunction with biological treatment. Combined tertiary processes like ozonation and activated carbon adsorption have drawbacks, including the potential for the generation of oxidation intermediates and ineffective disinfection (Giusy et al. 2019). On the other hand, membrane separations (Koltuniewicz 2010) have proven to be a viable integration option (Suthanthararajan et al. 2004) to have a more robust treatment process using both the AOPs and membrane separations since they have demonstrated better performances than the standard treatment methods and produce fewer byproducts. However, further effort is required to improve these membrane's efficiency, anti-fouling capabilities, and large-scale module design (Mirzaa et al. 2020).

The following two steps of oxidation are involved in AOPs: a) the production of potent oxidants (such as hydroxyl radicals) and b) the interaction of these oxidants with organic pollutants in water. But the phrase "advanced oxidation process" especially refers to procedures where organic pollutants are oxidized largely through interactions with hydroxyl radicals (Xu et al. 2010, Glaze et al. 1987, Wang & Xu 2012, Hengtao et al. 2021). AOPs often relate to a certain group of procedures that use O_3, H_2O_2, and/or UV radiation in water treatment applications. All of these procedures have the potential to generate hydroxyl radicals, which can chemically react and eliminate a variety of organic pollutants. Although some of the processes mentioned above might have additional methods for eliminating organic pollutants, an AOP's efficiency is often inversely correlated with its capacity to produce hydroxyl radicals. The possibility of post-treating a toxic as well as non-biodegradable effluent with AOP using optimum chemical and energy consumption and producing an output that is completely and rapidly biodegradable (Ticiane et al. 2006, Manna et al. 2022), has been extensively reviewed and published in many research works. There is now a chance for comprehensive and inexpensive elimination of refractory organics in the tannery effluent (Ambaye et al. 2020). Thus, the present study focuses on the degradation of refractory organics using photocatalysis with a view to the application of the same in industrial sectors on a large scale, offering an effective treatment. The second objective is to evaluate the effectiveness of bio-degradability of biologically treated tannery effluent through Ultraviolet radiation, thereby assessing the compatibility of Nanoporous Activated Carbon and hydrogen peroxide when used as catalysts in the process.

MATERIALS AND METHODS

Ultraviolet radiation is one of the most effective advanced oxidation processes. It involves the dissociation of water molecules by the incident ultraviolet radiation into hydroxyl radical species. Hydroxyl radicals are proven to be the most powerful oxidants which readily attack the organic as well as inorganic contaminants until they are reduced to simple inorganic molecules such as carbon dioxide and water. The most frequent and efficient electron acceptor in photocatalytic oxidation is oxygen, which works to further impede the electron-hole recombination action. Hydrogen peroxide has been the subject of several researches to examine its potential as an alternative electron acceptor (Burlacu et al. 2020) because of the potential for H_2O_2 reduction (0.72 V) to be higher than that of oxygen (−0.13 V) reduction. Hydrogen peroxide can present beneficial (Pillai et al. 2009, Poulios et al. 2003) or detrimental effects (Bandala et al. 2002, Dillert et al. 1996) on the photocatalytic degradation of organic contaminants. However, the majority of past photocatalytic research on the impact of hydrogen peroxide has noted that the ideal catalyst concentration depends on the properties of the wastewater. Hydrogen peroxide and nano-porous activated carbon corresponding to a mean pore size of 3.6 nm were used as catalysts in the ultraviolet radiation experiment. A multi-lamp photoreactor (Heber scientific model No HML-SW-MW-LW-888) was used for the study. The multi-lamp photoreactor (Paul et al. 2013) consists of six UV lamps arranged circularly, with six vials each of 100 mL capacity. It can produce UV light sources of intensities 254 nm, 312 nm, and 365 nm, respectively. The effluent sample was collected from the outlet of the secondary clarifier after the biological treatment from Pallavaram Common Effluent Treatment Plant, Chennai, Tamilnadu.

The collected samples were immediately stored at 20°C to maintain the humidity and temperature at constant values. The samples were analyzed for the physico-chemical characteristics in the laboratory according to the standard procedures (Chun & Park 1994, APHA 1990), and the results are presented in Table 1. The COD value is 656 $mg.L^{-1}$ with a standard deviation of 54 $mg.L^{-1}$. The BOD value of secondary treated tannery effluent is a range of 20 - 35 $mg.L^{-1}$, and the average TOC value is 212 ppm. The very low BOD/COD ratio in secondary-treated tannery effluent illustrates the presence of very poor biodegradable refractive organics in secondary-treated tannery wastewater.

RESULTS AND DISCUSSION

An experimental investigation was performed to reduce the refractory organics present in the biologically treated (Activated Sludge Process) tannery effluent using

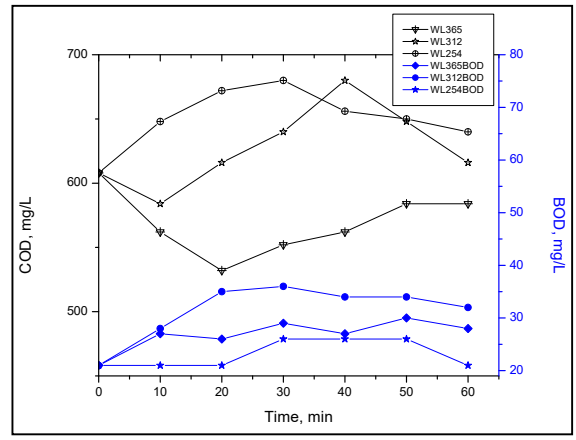

Fig. 1: Effect of wavelength on COD and BOD characteristics.

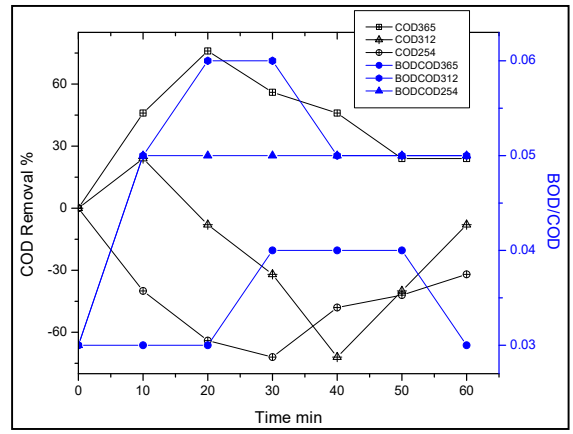

Fig. 2: Effect of wavelength on degradation and biodegradability index.

Photocatalysis. Furthermore, the process parameters such as time, pH and catalysts were analyzed in the oxidation process.

Effect of Wavelength in Photolysis of Organics

The photodegradation of wastewater mainly depends on the light absorption properties of its constituents. The light-absorbing properties of STTE could be studied by investigating the degradability of organics under different wavelengths. The effect of wavelength in the photodegradation of organic constituents of STTE in terms of the COD and BOD of STTE is shown in Fig.1. The characteristic organic constituents of the secondary treated wastewater after UV photodegradation at natural pH showed the refractoriness of organics towards photolysis. The biodegradability pattern over the study time is presented in Fig. 2. The biodegradability, i.e., the BOD/COD ratio, doesn't seem to have shown improvement after photodegradation. The COD content of the STTE was found to increase after the treatment, which may be due to the oxidation of inert organic material into an active state.

Table 1: Characteristics of secondary treated tannery effluent.

S.No.	Parameters	Mean ± SD
1.	pH	7 ± 0.273
2.	COD, $mg.L^{-1}$	656 ± 54
3.	BOD, $mg.L^{-1}$	35 ± 10
4.	TDS, $mg.L^{-1}$	4500 ± 530
5.	TOC, ppm	213 ± 46
6.	BOD/COD	0.08 ± 0.012

Effect of pH in Photolysis of Organics

The pH is one of the most important parameters that influence the photodegradation of organic pollutants. Therefore, the degradation of STTE was studied at different pH values namely, 5.5,6,8 and 8.5, using 254 nm, 312 nm, and 354 nm

UV light, respectively. The results are represented in Figs. (3-6). The photodegradation of STTE at pH 5.5 with UV light of 254nm, 312 nm, and 365 nm (Fig. 3) was observed to produce COD removal efficiency of 13%, 13%, and 17%, respectively. The BOD value reached a maximum of 108 mg.L^{-1} after degradation under 312 nm, whereas the biodegradability of the wastewater increased from 0.03 to 0.19. Although the COD removal efficiency was maximum at 365 nm, the biodegradability of the wastewater showed marginal improvement from 0.03 to 0.10, suggesting that the higher degradation percentage may correspond to the oxidation of volatile organics rather than the target refractory organics.

The photodegradation of STTE at pH 6 showed comparatively lesser efficiency in terms of degradation. The UV range of 312 nm and 365 nm was efficient by 21% and 18% in terms of COD reduction, whereas under 254 nm light, it showed an initial decrease to 532 mg.L^{-1}, and then it raised the COD level to 688 mg.L^{-1}. However, the biodegradability of STTE showed a decrease from 50 min of reaction time, suggesting lesser scope for further degradation. This may be attributed to the non-availability of compounds absorbing 254 nm light. The light absorption spectra of the STTE (Fig. 4) also showed light-absorbing compounds only in 200- 230 nm and 290-310 nm. This also supports the mode of light absorption. The photodegradation of STTE at pH 8 with UV light of 312 nm (Fig. 5) showed COD removal efficiency of 18%, and the biodegradability index was 0.16 after 60 min of degradation time. The BOD reached a maximum value of 97 mg.L^{-1} after degradation with light of 254 nm, showing the further increase in reaction time for photolysis of STTE of pH value 8 would positively

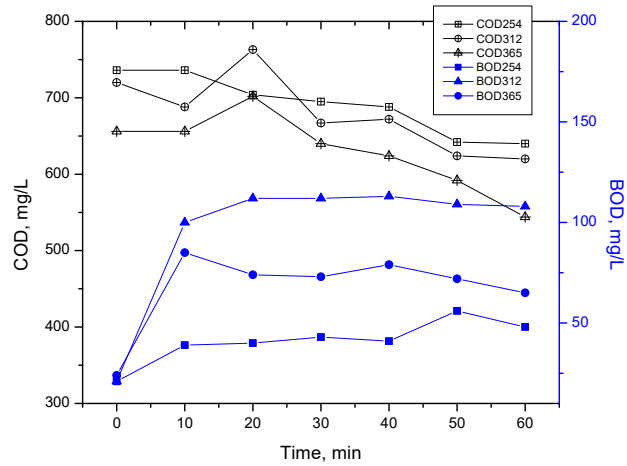

Fig. 3: Photodegradation of STTE: COD and BOD characteristics at pH 5.5.

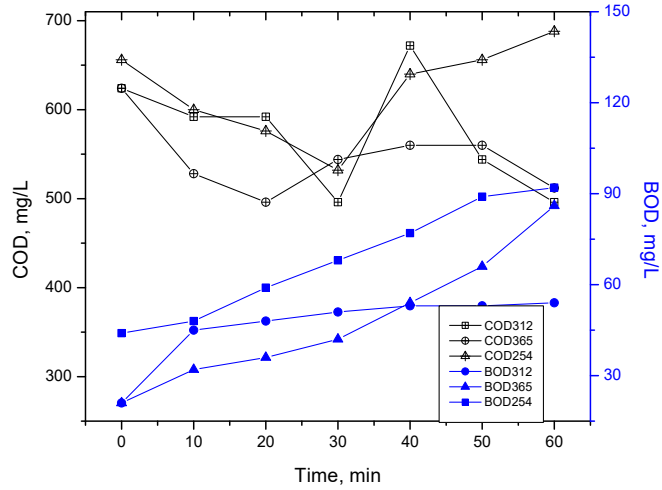

Fig. 4: Photodegradation of STTE: COD and BOD characteristics of STTE at pH 6.

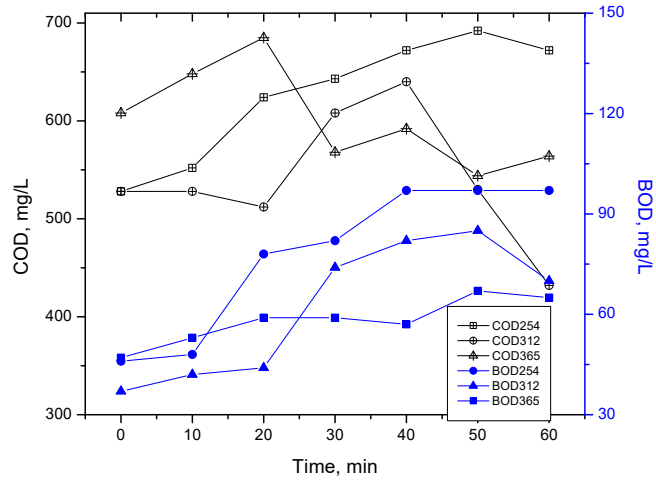

Fig. 5: Photodegradation of STTE: COD and BOD characteristics at pH 8.

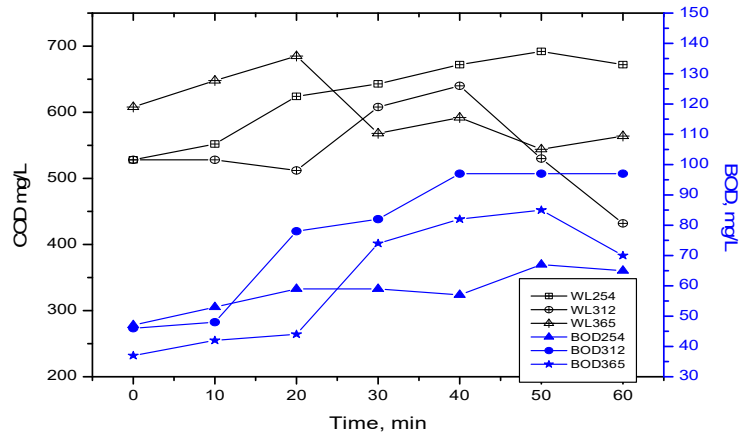

Fig. 6: Photodegradation of STTE: COD and BOD characteristics at pH 8.5.

influence in degradation of organics in wastewater (Fig. 6).

Numerous investigations have shown that the electrical excitation of the organic substrate takes place during the photolysis of organic materials (Giri et al. 2019, Hao et al. 2019, Sanzone et al. 2018, Xiong et al. 2016). The excitation causes the transfer of an electron from the excited state of the substrate to the ground state of molecular oxygen. It could also be caused by the hemolysis of substrate to form organic radicals, which later react with oxygen (Feng et al. 2013). Hence, the present study shows that the degradation of the contaminants by direct UV photolysis depends largely on the target compound under investigation (Kim & Tanaka 2009).

Effect of Catalyst in Photolysis of Organics

The key factors influencing UV photolysis are the medium's absorption properties, the photon flow rate at the excitation wavelength, and the amount of dissolved molecular oxygen present (De la Cruz et al. 2012). The wastewater samples were found to be rich in dissolved organic matter, thus showing reduced possibilities for direct UV photolysis treatment since, after subsequent biological treatment, the remaining colloidal organics may cause the photo-chemically active light to be filtered and screened. It is clear that the UV/H_2O_2 process implies the cleavage of H_2O_2 molecules by UV light into hydroxyl radicals (Jain et al. 2018), and the OH radical has high oxidation potential. The NPAC produced from the rice husk is capable of OH radical generation in the presence of oxygen (Karthikeyana et al. 2014). Hence, a detailed investigation was performed for the photocatalytic oxidation of STTE with H_2O_2 and NPAC as catalysts, respectively.

The characteristics of NPAC samples are important for enumerating their ability to enhance the efficiency

of oxidation. In NPAC, which belongs to the extrinsic semiconductor range, the energy gap (Eg) of 1.55 eV correlates with the highest reflectance at a wavelength equivalent to 800 nm. Due to its ideal surface area, pore diameter volume, and free electron density properties, NPAC was chosen as a heterogeneous catalyst for the oxidation process (Table 2). The Photocatalytic oxidation of STTE with H_2O_2 as the catalyst, as a function of time (Fig. 7), shows a maximum efficiency of 30% COD removal. However, the biodegradability was not much enhanced, and the

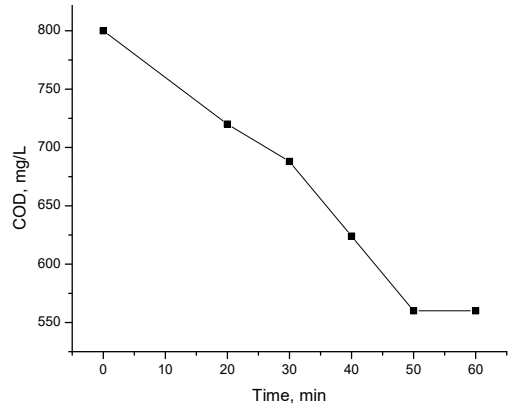

Fig. 7: Photocatalytic oxidation of STTE with H_2O_2 as catalyst.

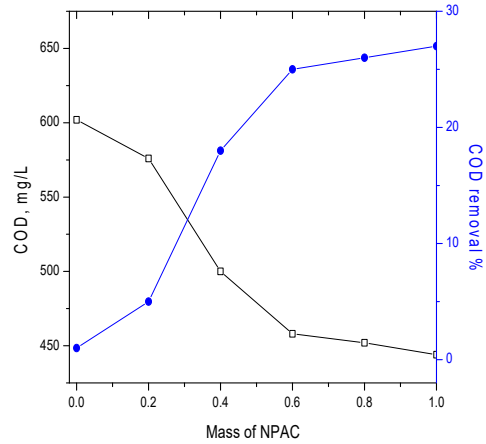

Fig. 8.a: Effect of mass of NPAC on degradation of organics-COD.

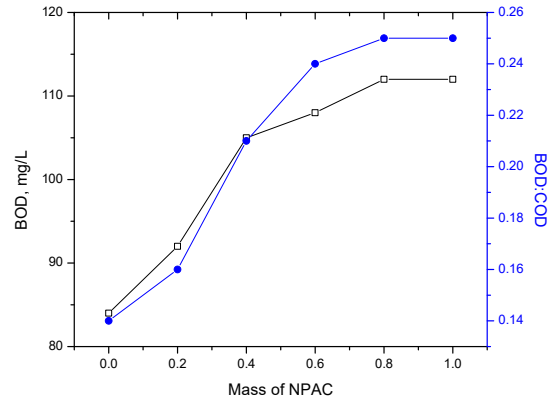

Fig. 8.b: Effect of mass of NPAC on degradation of organics – BOD, biodegradability.

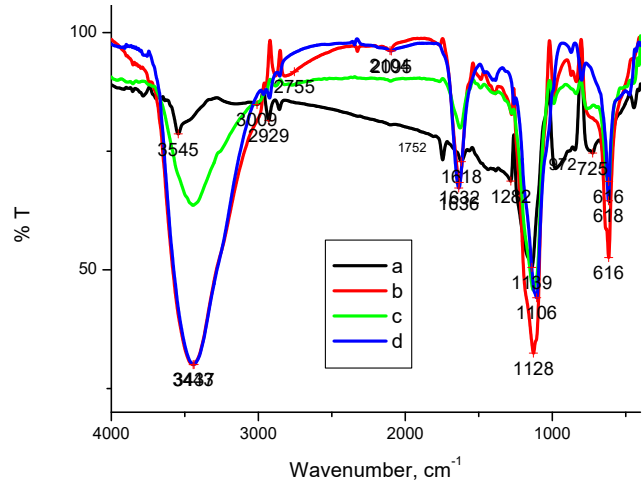

Fig. 9: FTIR spectrum of photodegradation of organics in secondary treated tannery effluent.
a. Initial, b. Photolysis at 312 nm for 60 min, c. Photocatalytic Oxidation with NPAC as Catalyst, and d. Photocatalytic Oxidation with Hydrogen Peroxide as Catalyst.

Table 2: Characteristics of nano-porous activated carbon.

S.No.	Parameters	Values
1.	Surface Area [$m^2.g^{-1}$]	379
2.	Average pore diameter [Å]	39.36
3.	Carbon [%]	41.58
4.	Hydrogen [%]	2.85
5.	Nitrogen [%]	0.75
6.	Free electron density [$spins.g^{-1}$]	16.05 x10^{21}
7.	Energy gap [eV]	1.55
8.	Ash Content [%]	41.60

biodegradable index of wastewater after the photocatalytic oxidation of STTE with H_2O_2 was found to be only 0.13. Fig. 8 (a & b) shows the effective treatability of refractory organics by photocatalytic oxidation with NPAC as the catalyst. Among all the oxidation reactions carried out, the photocatalytic oxidation of refractory organics with NPAC as a catalyst showed a maximum efficiency of 27%, and the biodegradability also increased to 0.25, further improving the content of biodegradable materials in the wastewater. This is also supported by the FTIR instrumental evidence, as shown in Fig. 9.

The variation in the region 1600-1200 cm^{-1} depicts the formation of a new compound after photon interaction in all the studies. There occurred elimination of many bands after the photocatalytic oxidation with NPAC as a catalyst. 2929 cm^{-1} in the raw wastewater sample may correspond to the presence of the methoxy group, which was then eliminated after degradation. The weak band at 3545 cm^{-1} may be attributed to p-CN or p-NO_2 group presence, which later got eliminated after the photo interaction of organics

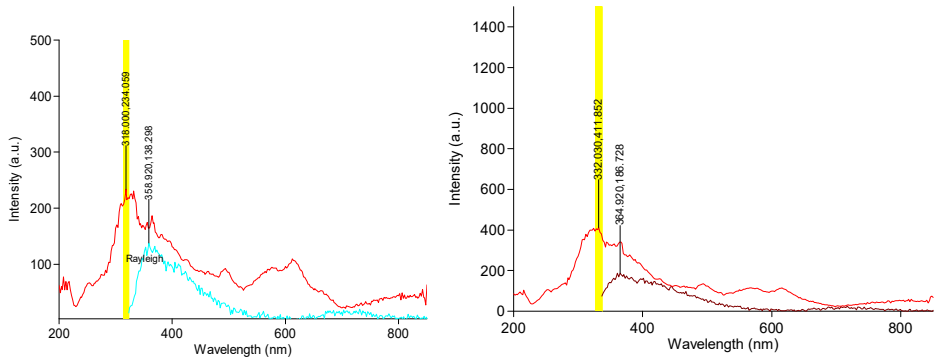

Fig. 10: Fluorescence spectrum of wastewater (a) before and (b) after degradation.

in wastewater. The medium band corresponds to hydroxyl absorption, and the elimination of the band at 1752 cm^{-1} may correspond to cyclic lactam. The UV visible spectrum and the fluorescence spectrum of wastewater before and after degradation also confirm the effective photocatalytic oxidation of refractory organics (Fig. 10). The shift in excitation wavelength from 318 to 332 nm and the increase in absorption intensity of the 332 nm peak suggests the formation of degraded products of less complexity after photocatalytic oxidation.

CONCLUSIONS

The Advanced Oxidation Process, especially UV photocatalysis, is a very useful method for the treatment of water consisting of aromatic compounds, which are highly bio-refractory and toxic. The optimum time, PH, dosage of H_2O_2, and mass of NPAC required for the effective treatment of the synthetic wastewater using photocatalysis were found to be 60 min, 8, 0.2 $mg.L^{-1}$, and 1g.100 mL^{-1}, respectively. It was found that the heterogeneous photocatalysis improves the biodegradability of refractory organics. The efficiencies of photolysis, homogeneous photocatalysis, and heterogeneous photocatalysis were found to be 18%, 30%, and 27%, respectively. Although the efficiency of homogeneous photocatalysis was found to be higher than that of heterogeneous photocatalysis, the biodegradability was higher in the latter, with a value of 0.26. The biodegradability of the former was found to be 0.13. Thus, heterogeneous photocatalysis assures a promising scope for future work. Further analysis in this study with prolonged time and higher mass of carbon may increase the efficiency of this process. Further research is needed to assess the suitability of photocatalysis in industrial sectors.

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