Photocatalytic Degradation of Aqueous VOCs Using N Doped TiO_2: Comparison of Photocatalytic Degradation under Visible and Sunlight Irradiation

V. S. Priya and Ligy Philip

***Abstract*—Present study deals with the photocatalytic degradation of low concentrations of VOCs like methanol, acetone, dichloromethane (DCM), benzene and toluene that are found in the pharmaceutical wastewater. The synthesized N doped TiO_2 were used. N doped TiO_2 with the reduced band gap energy of 2.91ev exhibited a complete degradation of the target VOCs in the presence of visible and sunlight. Kinetic study indicated that the rate of degradation of the target solvents was high in the presence of sunlight than when compared to visible irradiation. This may be due to the presence of the UV radiation in the natural sunlight. Irrespective of irradiation, the order of degradation of VOCs for the N doped TiO_2 was: benzene > toluene > DCM > methanol >acetone. For mixed pollutant condition, individual pollutants followed similar degradation pattern as single pollutant system. This study has proven the suitability of photocatalysis as an alternative polishing treatment for pharmaceutical wastewaters.**

***Index Terms*—Band gap energy, N doped TiO_2, pharmaceutical wastewater, visible light irradiation.**

I. Introduction

Volatile organic compounds (VOCs) are chemicals with a high vapour pressure. These pollutants arise in the environment from the industrial and household activities. There is a growing awareness for the elimination of VOCs from the wastewater because of their detrimental health impact on humans. Moreover, VOCs are volatile in nature and are responsible for formation of troposphere ozone and photochemical smog. Among various industries, pharmaceutical sector employs a wide variety of solvents belonging to various categories like aromatic, alcohol, halogenated, ketones for drug synthesis [1]. Presence of VOCs like methanol (75.61 million kg/year), dichloromethane (25.89 million kg/year), was reported in pharmaceutical industrial effluent by EPA [2]. Discharge of untreated effluents pollutes the surface and the ground water. In India, drug synthesis clusters are more predominant in Gujarat and hence there is an increase in contamination of ground and surface water in villages in around the industrial clusters. 15 μg/L of 1, 2-4 trichlorobenzene from an open well is reported in Sarangpur village (Gujarat, India). High concentration of trichloroethene (20 μg/L), chlorobenzene (85 μg/L), 1, 3-dichlorobenzene (10 μg/L), 1, 4-dichlorobenzene (5 μg/L) were detected from groundwater in Sarigam (Gujarat, India) [3]. Pharmaceutical industry adopts various physiochemical and biological treatment technologies for the treatment of VOCs. Most common method for aqueous VOC removal is by air stripping process, and the efficiency of this process depends on the air-water ratio. Adsorption of the dissolved aqueous VOC using activated carbon has limited efficiency and the inherent problem involved is the disposal/ decontamination of the spent carbon. The increase in aeration rate in the aeration tank tends to increase the off-gas VOC concentration from activated sludge process. Percentage removal of chlorinated compound like dichloromethane (23%), and tetra chloro ethylene (39%) by stripping was reported to be high when compared to non-chlorinated compounds like toluene (25%) and xylene (17%) [4]. Moreover, emission of VOCs from the aeration tank of the conventional activated sludge process and granule deterioration in the anaerobic systems due to solvent toxicity often result in an incomplete treatment of the volatile organic compounds. It is essential to degrade the low concentration aqueous VOCs from partially treated wastewater at a faster rate, because they get easily stripped off into the atmosphere when discharged either in a sewage treatment plant or in surface water body. Though air phase VOC removal is well established, not much work has been reported on the removal of low concentration aqueous VOCs. Heterogeneous photocatalysis, which involves photo excitation of the semiconductor results in the formation of electron donor and acceptor sites, provide scope for redox reactions. The advantage of shorter degradation time, the independence from bacterial activity extends its application in the treatment of recalcitrant compounds.

Among the various semiconductors, TiO_2 are the most widely used in environmental application because of its low cost, non-toxicity, and high oxidising power. Although TiO_2 have been widely investigated on the photocatalytic degradation of recalcitrant compounds like phenol [5] it cannot be effectively used because of the high band gap energy requirement.

Doping with non metals such as C [6], N [7], S [8] have been extensively studied and such doping exhibit a significant improvement in photo-sensitization in the visible region. The photo catalytic efficiency of N doped TiO_2 was higher in degrading the reactive red violet dye [9], phenol [10] in the visible light irradiation. The ability of such doped catalyst to perform well even in the presence of visible and sunlight can be extended from its application in degradation of single organic pollutant to a mixture of complex VOCs that is very commonly encountered in the industrial effluent.

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Photocatalytic degradation of mixtures of pollutant in the visible and in sunlight irradiation is an area to explore.

The objectives of the present study was to evaluate the efficiency of N doped TiO_2 in visible light and in solar irradiation to degrade low concentrations of target VOCs like methanol, acetone, dichloromethane, benzene and toluene, commonly found in pharmaceutical wastewaters, both as a individual pollutant as well as in mixed pollutant condition. An attempt was also made to compare the degradation rate of aqueous VOCs for N doped TiO_2 under visible and sunlight irradiation.

II. Materials and Methods

A. Chemicals

Solvents like methanol, acetone, benzene, dichloromethane and toluene used for this study were of HPLC grade (Rankem, India).

B. Preparation of N-Doped TiO_2

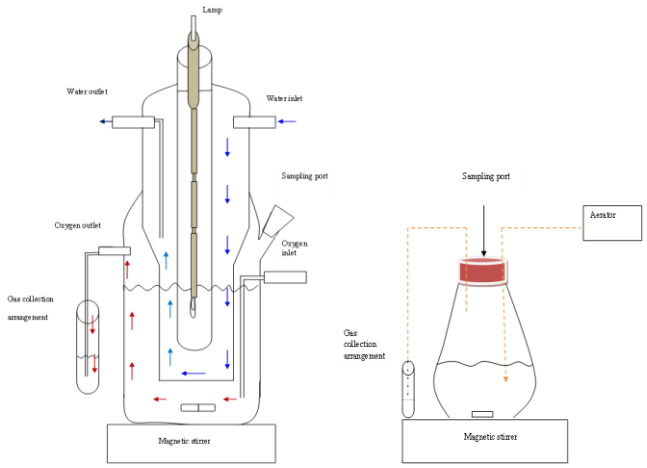
Preparation of N doped TiO_2 was carried out as per the procedure described by Senthilnathan and Philip [11]. 2.4 mL of titanium iso propoxide was dissolved in 20 mL in ethyl alcohol. Triethyl amine, which is the source of nitrogen, was added to it at a ratio of 1: 1.6 (Ti: N). 20 mL of 0.1 M HCl was added to the above solution and stirred to get a clear liquid. The solution was then autoclaved at 80 ˚C for 12 h followed by centrifuging the suspension at 8000 rpm. The residue was dried at 100 ˚C. Calcination of the dried sample was performed at 550 ˚C for 4 h. The resulting sample of N doped TiO_2 was used for further studies

C. Analytical Methods

Concentration of VOCs was analyzed using Gas chromatography (Perkin Elmer, USA) with Perkin Elmer Elite PE-624 column (30 m × 0.53 mm × 0.5 mm film thickness) equipped with flame ionisation detector (FID). The temperature of the injector and the detector were maintained at 150 ℃ and 300 ℃, respectively, and the column was maintained at 120 ℃ for a run time of 12 minutes. Nitrogen gas was used as the carrier gas and its flow was maintained at 2 mL/min. Standard graphs were prepared for each solvent using the peak area obtained when known concentrations were analysed and these standard graphs were used to analyse the unknown concentrations. TOC Analyzer (Shimadzu, Japan) was used to analyse the TOC of liquid samples obtained after the centrifugation at 10,000 ×g for 5 minutes.

D. Photocatalytic Reactor

A cylindrical photocatalytic reactor of 400 mL volume was provided with a water circulation arrangement to maintain the temperature in the range of 25–30 ◦C. A 500 mL conical flask with Teflon cap was used as the reactor for studies under sunlight irradiance. 150 W high pressure tungsten visible lamp, supplied by Haber Scientific, India, were used for the source of irradiation for visible light. An oxygen flow rate of 300 mL/min and a stirring rate of 150 rpm were maintained in all the experiments. The lamps were switched on 15 min before the start-up of the experiment to attain the required energy. The samples were collected at regular intervals of 5 minutes for the first 30 min. duration and then at an interval of 15 minutes till the completion of the experiment, for the analyses of various parameters. A schematic diagram of the photo catalytic reactor employed in the study is given in Fig. 1a and the reactor used for the studies under the sunlight is shown in Fig. 1b.



1.a 1.b

Fig. 1. Experimental setup: (a) photoreactor used under UV Lamp and Visible lamp, (b) photoreactor used under sunlight.

E. Batch Degradation Study

Batch degradation study was performed with the synthesized N doped TiO_2 for the solvents like methanol, acetone, dichloromethane, benzene and toluene. Single substrate degradation study was carried out for three different initial concentrations of 10 mg/L, 30 mg/L and 50 mg/L (in Millipore water). Mixed pollutants studies were also carried out with all the pollutants consisting of 50 mg/L of each of the pollutants. 200 mg/L of photocatalyst dosage was used in all the experiment. Photocatalytic efficiency of N doped TiO_2 was evaluated in visible and sunlight irradiance. Reaction rates, k (min−1) and R^2 were determined by plots of $\ln(C_o/C)$ versus time for all the irradiations. The purpose and description of each study is given in Table I.

TABLE I: Purpose and Description of Various Batch Photocatalytic Degradation Studies

Pollutant	Concentration (mg/L)	catalyst	Irradiance
Single substrate degradation study			
Methanol	10,30,50	N doped TiO_2	Visible and Sunlight
Acetone	10,30,50		
Dichloromethane	10,30,50		
Benzene	10,30,50		
Toluene	10,30,50		
Multiple pollutant degradation study			
Mixture of Methanol, Acetone, Dichloromethane, Benzene and Toluene	50mg/L of each solvent	N doped TiO_2	Visible and Sunlight

III. Result and Discussion

A. Properties of N-Doped TiO_2

The optical property of the N- doped TiO_2 is extensively studied by Senthilnathan and Philip [11]. It is reported that the average crystalline size of the sample was 22 nm. The band gap energy of the prepared sample was calculated by using the equation E_g=1239.8/λ where E_g is the band gap energy (ev) and λ is the wavelength (nm) of the absorption edge in the spectrum and it was reported as 2.9 ev [11]. The reduction in the band gap energy will enable the transfer of electrons from the valance band to conduction band in the presence of visible light.

B. Photocatalytic Degradation of Target VOCs with Nitrogen Doped TiO_2 under Visible Light

Selected VOCs were subjected to photocatalysis with N doped TiO_2 in presence of visible lamp. An initial concentration of 50 mg/L was employed for all the VOCs. The degradation was slower and the results are presented in Fig. 2.

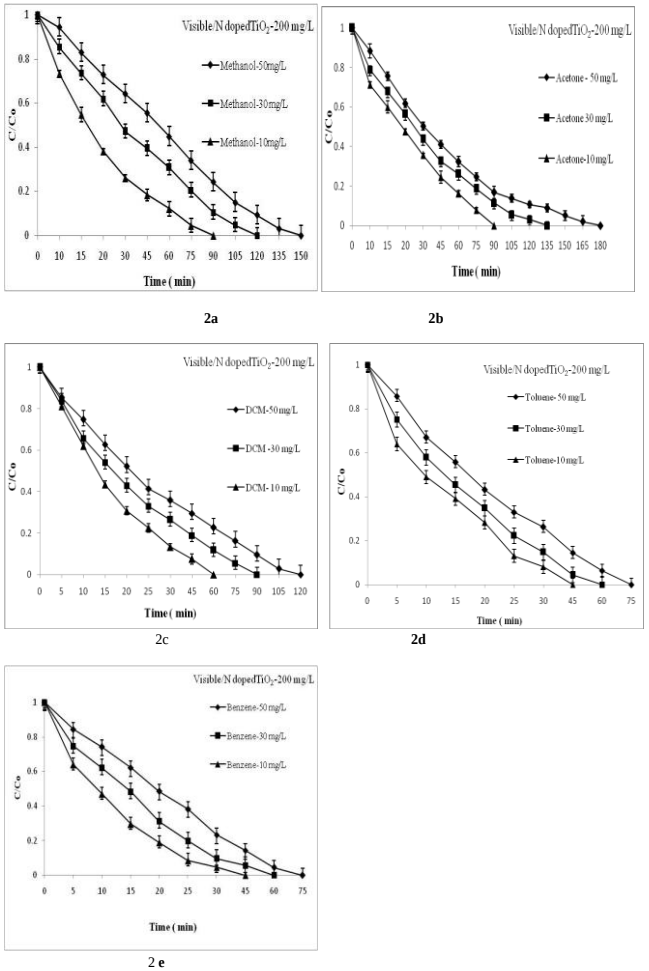


Fig. 2. Kinetic plots for different concentrations of various VOC degradation by Nitrogen doped TiO_2 (200 mg/L) under visible light : (a) Methanol, (b) Acetone, (c) Dichloromethane (DCM), (d) Toluene, (e) Benzene.

Nitrogen incorporated in to the lattice of the TiO_2 replaces oxygen (interstitial doping) without altering the average unit cell. Mixing of nitrogen 2p state with oxygen 2p state in the valence band or the N induced mid gap level attribute to a decrease in band gap [11]. The band gap energy of the synthesised catalyst was 2.91 ev and hence, it can be effectively used in the presence of the visible lamp. Oxygen vacancy is created due to the replacement of nitrogen atoms with oxygen in the TiO_2 lattice and the photocatalytic oxidation of pollutants depend on the oxygen vacancies (Ti^{3+}) because it serve as active site for degradation [12]. Molecular oxygen adsorbed on the active site are activated as oxygen species by charge transfer of electrons and they serve as a potential oxidising agent. Hence, adsorption of pollutants in the active site and its interaction with oxygen species determine the rate of degradation. 50 mg/L of benzene and toluene were degraded within 75 minutes. GC –MS analyses of the aqueous sample at an intermediate duration of the experimental study revealed that phenol was one of the by products formed during the degradation of benzene GC- MS analyses results of intermediate is presented in Fig. 3.

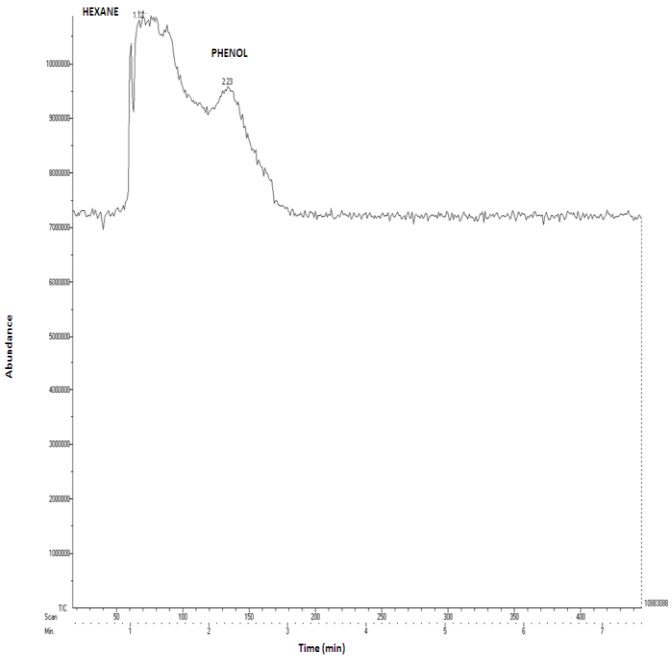


Fig. 3. Analysis of intermediate during benzene degradation with N doped TiO_2 in visible irradiance.

The possible degradation pathway of benzene may be the addition of hydroxyl radical to benzene and subsequent addition of oxygen which results in product like phenol [13]. Higher degradation rates observed for benzene and toluene may be attributed to its adsorption in the oxygen vacancies. It has been reported that the ethylene glycol chemisorbed on active site were degraded at a faster rate [14]. Chlorinated compounds also get degraded by the oxygen species. CO, HCl were some of the intermediate reported during the degradation of methyl chloride at the oxygen vacancies and the photoxidation due to OH groups occur only after the initiation of active species [15].

Complete degradation of DCM, took place within 120 min., but methanol and acetone took 150 min., and 180 min., respectively to degrade. Increased alcohol adsorption on the catalyst surface also and subsequent degradation of ethanol due to direct binding into the oxygen vacancy site have been reported in N doped catalyst [16]. Pollutants like methanol exhibit a tendency to block the surface active site (Ti^{3+}). This results in a prolonged time for the complete degradation of

methanol. The photo degradation rate of the VOCs by N-doped TiO_2 under visible irradiation followed the order; benzene > toluene > DCM > methanol > acetone. The lower rate of degradation in visible light may because the photocatalytic oxidation in N doped TiO_2 mainly occur through the O_2 reduction (superoxide radical) and not because of the hole generated at the N induced mid gap level [14]. Removal of 2, 4-dichlorophenol with Degussa P-25 and the nitrogen-doped TiO_2 using triethylamine was compared and it was reported that the N-doped exhibited a higher efficiency in the removal of 2, 4-dichlorophenol, in the visible light [17]. Irokawa *et al.* [18] have reported that in the presence of visible light, toluene, which was weakly adsorbed on the surface of the catalyst, was initially photo-oxidised to products like benzaldehyde, which then strongly adsorbed on to the surface of the catalyst and resulted in products like aldehydes. These products are then photo degraded to CO_2 and H_2O under visible light irradiation. The complete degradation of solvents like toluene, acetone and methanol were ascertained by measuring TOC. TOC values were below the detection limit at the end of the degradation time. The rate of degradation of target compounds using N-doped TiO_2 under visible light is presented in Table II.

C. Photocatalytic Degradation of Target VOCs with Nitrogen Doped TiO_2 under Sunlight

Photo degradation of target compounds was evaluated using N-doped TiO_2 under solar irradiation and the results are presented in Fig. 4. Even though the potential of sunlight to activate the TiO_2 catalyst has been extensively used for the destruction of various recalcitrant pollutants its potential to activate the N doped TiO_2 and to degrade the target solvent is evident from this study. The degradation of 50 mg/L of benzene and toluene was achieved within 60 min of irradiation under sunlight.

The compounds like DCM, methanol and acetone was degraded in 105 min, 135 min and 165 min, respectively. The degradation pattern under solar irradiation was similar to that of visible light. followed The affinity of the substrate to get degraded in the presence of sunlight was observed to be almost the same when compared with the visible light, except that the time required for the complete degradation was reduced. The stable compound like benzene achieved a faster degradation followed by toluene and DCM. In the N doped TiO_2, electrons are excited from the valance band as well from the N- induced mid gap level.

The excited electrons get adsorbed by the oxygen vacancy available in N doped lattice. Under the visible irradiation, the holes generated in the N induced mid gap level are less mobile and they get trapped by the photo induced electrons from the oxygen vacancies. Presence of UV in sunlight, excites more photo induced electrons directly to the conduction band of the N doped TiO_2, rather to the oxygen deficit sites [19]. This reduces the surface recombination of holes and the photocatalytic oxidation of pollutant can occur both due to superoxide radical at the active site as well as due to holes generated in the N-induced level.

Presence of UV radiation (5%) in the solar spectrum can enhance an easy transfer of electrons to the conduction band of the catalyst in the natural light. As a result, the rate of photo degradation obtained under solar irradiation using N doped TiO_2 was comparatively higher than that under visible light. The reaction rates are given in Table II. The stripping of VOCs in this process is inevitable because of the constant supply of oxygen. The loss of VOCs by stripping was determined by measuring the amount of the solvent that was collected in the impingers connected to the reactor. The percentage loss due to stripping was 1.3% and 2.3% for highly water soluble compounds like methanol and acetone, respectively and it was 3% for chlorinated solvent like dichloromethane.

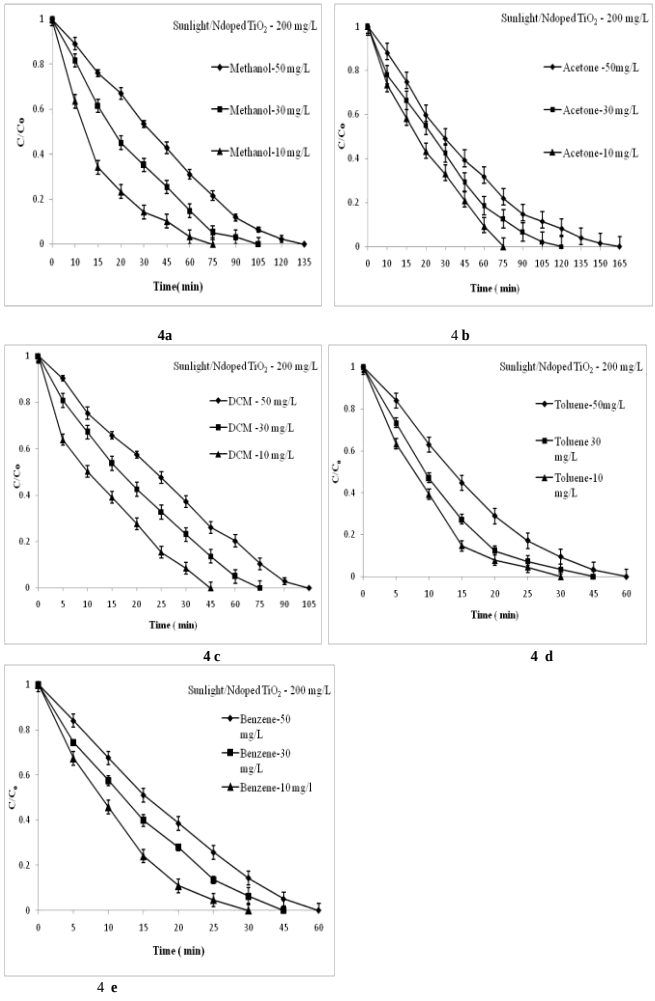


Fig. 4. Kinetic plots for different concentrations of various VOC degradation by Nitrogen doped TiO_2 (200 mg/L) under sunlight : (a) Methanol, (b) Acetone, (c) Dichloromethane (DCM), (d) Toluene, (e) Benzene.

D. Photocatalytic Degradation of Mixture of VOCs

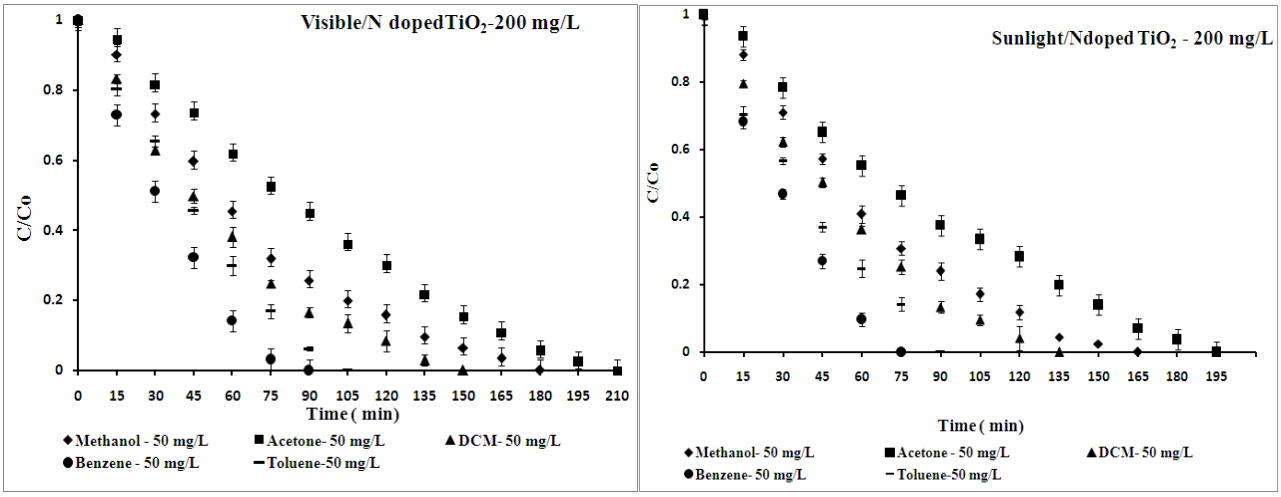
Mixture of equal concentration (50 mg/L of each) of methanol, acetone, dichloromethane, benzene and toluene were subjected to photo-catalysis under two conditions namely, N doped TiO_2/visible lamp and N doped TiO_2/sunlight and the results are presented in Fig. 5. In a mixed pollutant condition, (in all the above mentioned conditions) the degradation rate of the VOCs was different from the degradation rate when they were present as individual pollutants (Table II).

The presence of methanol and acetone did not interfere with the degradation of the toluene and benzene. Similar effect has been reported that when phenol and methanol were

present at equal concentration (1mmol/ L), the rate of phenol disappearance was independent on methanol [20]. There was no difference in degradation trend was observed during multiple pollutant condition in the present study when compared to the individual pollutant condition.

This indicates the dominance of the hydroxyl species in the degradation process. There was not much difference in the affinities of benzene and toluene to the hydroxyl radical. The photo degradation rate of the VOCs in all the cases followed an order of benzene > toluene > DCM > methanol > acetone. The degradation of pollutants were much faster in sunlight followed by visible light. Moreover, the time required for complete degradation was prolonged because of overloading of pollutants. It is reported that due to the competitive adsorption of benzene and toluene, the reaction coefficient of the photocatalytic oxidation of each of the pollutant was different from that in a mixed condition [21].

The complete mineralisation of the compounds was ensured with the complete removal of TOC from the samples after the photo catalysis. The degradation of the target solvents seem to be independent of each other in the mixed condition and the variation in the rate of the degradation seem to differ with the type of the excitation used. Degradation of such complex mixture of solvents in the presence of sunlight using N- doped TiO_2 can be employed as a polishing treatment for the removal of aqueous VOCs from secondary treated effluents of pharmaceutical industries.



5a 5b

Fig. 5. Kinetic plots for mixed pollutant degradation for a catalyst dosage of 200 mg/L under various light sources: (a). Ndoped TiO_2 under visible irradiation, (b). N doped TiO_2 under sunlight.

TABLE II: REACTION RATE CONSTANT FOR AN INITIAL CONCENTRATION OF 50 MG/L OF POLLUTANTS FOR DIFFERENT IRRADIATIONS

Catalyst/ irradiation	Individual pollutant condition									
	methanol		Acetone		DCM		Toluene		Benzene	
	k (min^{-1})	R^2	k (min^{-1})	R^2	k (min^{-1})	R^2	k (min^{-1})	R^2	k (min^{-1})	R^2
N doped TiO_2 /visible	0.019	0.899	0.019	0.993	0.026	0.969	0.044	0.995	0.046	0.962
N doped TiO_2 /sunlight	0.023	0.966	0.020	0.994	0.029	0.990	0.063	0.990	0.049	0.985
	Mixed pollutant condition									
N doped TiO_2/visible	0.011	0.979	0.011	0.922	0.017	0.986	0.020	0.945	0.028	0.945
N doped TiO_2 /sunlight	0.012	0.915	0.012	0.915	0.019	0.945	0.024	0.978	0.033	0.983

IV. CONCLUSION

The potential of Nitrogen doped TiO_2 to degrade VOCs present in aqueous environment was studied. Results indicate that complete degradation of VOCs at a lower concentration is achievable with the N doped TiO_2 in the presence of visible light. The efficiency of the N doped catalyst is higher in the presence of sunlight due to the presence of UV radiation in the natural solar spectrum. The reaction rate of each pollutant was different in individual condition when compared to the mixed condition due to the competitive adsorption in the mixed substrate condition.

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Dr. Philip has several publications in reputed journals and co-authored the following chapters such as i)Removal of heavy metals from wastewaters ii) Treatment of Phenolic wastewaters iii)Biotechnology for wastewater treatment in the book Wastewater treatment technology Ed. R.K. Trivedy, 1998 by Enviromedia.