

Synthesis and Photocatalytic Degradation of Dimer Model Compounds of Lignin Over UV Irradiated TiO₂

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The effluents of paper mill contain lignin as major constituent. Lignin is a bio polymer and it has less affinity to undergo biodegradation, therefore it persists in water. Model dimer compounds synthesized from coniferyl alcohol, *p*-coumrylic acid and sinapyl alcohol. The epoxides of respective alcohols are obtained by *m*-chloroperbenzoic acid oxidation on double bonds. In presence of sodium hydroxide the epoxides are refluxed with coniferyl alcohol, *p*-coumrylic acid and sinapyl alcohol, respectively so that dimers are formed. The reaction products are isolated by flash chromatography. Here an attempt has been made to subject photocatalysis of model compounds using aqueous TiO₂ suspension. For the experimental setup fabricated, the optimum mass of catalyst and pH of aqueous media suitable for optimum possible degradation was investigated. Initially, the absorption characteristics of model compounds prepared are determined. The influence of the nature of phenolic units on photo degradation has been investigated by comparing the initial degradation rates. The Langmuir-Hinshelwood kinetics constant parameters evaluated. Kinetics of chemical oxygen demand and phenolic contents studied and based on the results path of degradation proposed.

Key Words: Photocatalytic degradation, Dimer, Lignin, TiO₂.

INTRODUCTION

Water pollution is becoming a serious global problem. Lignins are present in effluent. They are not decomposed by biodegradation. Therefore they get accumulated in nature and persist for a long time¹. Conventional biological treatment processes are non-destructive for these compounds.

The traditional physico-chemical treatments such as nanofiltration, ozonization and combustion are some efficient, but have inherent limitations in applicability², such as effectiveness and cost. Advanced oxidation processes have been considered as promising for the remediation of contaminated water³.

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It is possible to decompose various organic materials using an irradiated TiO₂ semiconductor⁴. Herein, an attempt is made to degrade model dimers of lignin by photocatalysis.

EXPERIMENTAL

The laboratory system consists of a recirculating batch photo reactor fabricated with pyrex and teflon components. Fig. 1 is the schematic diagram of the laboratory system. Water, chemical substance under investigation and titanium dioxide are continuously recirculated through 500 mL reactor until the substance concentration is below the detection limit. A constant flow rate of *ca.* 1 L/min is always used. Small aliquots were taken from the reservoir at reaction time intervals during each run and analyzed for COD required. UV absorbance at predetermined λ of concern compound and phenolic contents using Lovibond Multidirect at specified wavelength is prescribed by standard methods for examination of water and wastewater. In this way no significant loss of volume occurred during the experiment. Oxygen is introduced through a flow meter and distributed *via* a sintered glass tip to maintain a constant oxygen concentration (20-30 mL min⁻¹). In these experiments the pH of the solution was adjusted by adding an appropriate amount of buffer solution to suspension⁵. Irradiation was carried out with a UV tube light of UV A range, 36 W, having λ range 310-400 nm and λ_{max} is 365 nm.

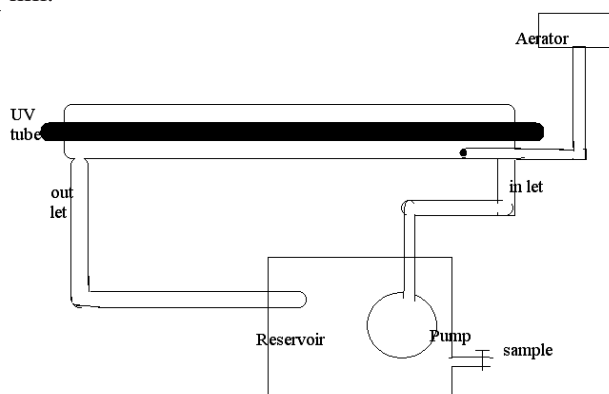


Fig. 1. Schematic diagram of the laboratory system

The powered semiconductor catalyst used as supplied, without any pre treatment. The titanium dioxide used was Degussa P-25 (particle size 20 to 30 nm by transmission electron microscopy, surface area 53 m² g⁻¹ by BET methods, crystal structure 83 % anatase and 17 % rutile as determined by X-ray diffraction analysis. Catalyst loading in all kinetic runs was 1 g L⁻¹ to 3.5 g/L, this value has been found to be large enough to make the reactor opaque to UVA radiation.

Coniferyl alcohol, *p*-coumrylic acid, sinapyl alcohol, *m*-chloroperbenzoic acid, NaOH, LiAlH₄ and dichloromethane are of AR grade procured from Aldrich.

Synthesis of dimers: The chemicals coniferyl alcohol, *p*-coumrylic acid, sinapyl alcohol, *m*-chloro perbenzoic acid, NaOH, LiAlH₄ used without any further purification. The first step of reaction conducted in three necked flask with sealed stirrer unit, a 250 mL dropping funnel and a double surface condenser are fitted to it. The guard tubes containing calcium chloride attached to the open ends of condenser and dropping funnel. A funnel with a very short wide stem placed by removing dropping funnel and introduced powder lithium aluminium hydride into flask through it. Dried ether used to transfer the last traces into the flask. Replace the dropping funnel and guard tube. While stirring a solution of 10 g of *p*-coumrylic acid in dry ether is added to an ethereal solution of lithium aluminium hydride at such a rate that the reaction mixture boils gently. When the reaction mixture becomes viscous, then 50 mL of anhydrous ether added to facilitate stirring. Continued the stirring for 15 min after all solution from dropping funnel is added. The excess lithium aluminium hydride is decomposed by the addition of 75 mL of water. The reaction product is filtered through sintered glass funnel and dried ethereal solution with magnesium sulphate and distilled off ether with rotary evaporator. Colourless viscous residue is solidifies on cooling. Dissolved the sludge that left in the filter funnel in 20 % sulphuric acid, extracted the resulting solution with six 10 mL portions of ether, again ether is removed by means of rotary evaporator, on cooling alcohol get crystallized. In the second step 5 g of *p*-coumryl alcohol prepared is dissolved in 5 mL of dichloromethane and 5.6 g of *m*-chloro perbenzoic acid is dissolved in 5 mL of dichloromethane and stir the mixture at 0 °C for 24 h, filtered, washed with 10 % NaHCO₃ and dried over anhydrous sodium sulphate and then distilled the dried dichloromethane solution through an efficient fractionating column. 2.5 g of epoxide of *p*-coumryl alcohol is added to 2.5 g of *p*-coumryl alcohol dissolved in 10 M NaOH. The mixture is refluxed for 4 h. The contents are cooled and crystallized then subjected to flash chromatography described⁶. Silica gel 60 (40-230 mesh) was used as column, solvent mixture of acetate/light petroleum (b.p. 30 to 60 °C) used as eluant. The flow rate maintained as 2 ± 0.1 in/min. Same procedure adopted as above to prepare dimer model compounds of coniferyl alcohol or sinapyl alcohol.

For all the prepared compound IR, UV, ¹H NMR (200 Hz), ¹³C NMR spectra taken for conformation of structure. All batch equilibrium experiments were conducted in the dark. Measurements were made on suspensions prepared by mixing 20 mL solution of various initial concentrations of model compounds at pH 5-6 and fixed amount of catalyst. A TiO₂ concentration of 1-5 g L⁻¹

was used in adsorption dark experiments. Preliminary experiments suggest that the adsorption gets saturation in 40 to 50 min, so before irradiation the reactants mixture kept in dark for 50 min before subjecting to photocatalysis. The equilibrium concentrations are determined using HPLC after centrifugation and filtration, through Millipore filters (0.45 mm diameter), of the suspension. The number of organic compound molecules adsorbed per gram of TiO₂ (n^s_2 in mol g⁻¹) is calculated as follows⁷:

$$N^s_2 = V \times \Delta C / W \quad (1)$$

where ΔC is difference between the initial concentration, (C_i) and equilibrium concentration, (C_{eq}), V the volume (0.02 L) and W is the weight of TiO₂ (0.02 g).

RESULTS AND DISCUSSION

Epoxide given by *p*-coumryl alcohol (III), 4-[3-(hydroxymethyl)-oxiran-2-yl]phenol: ¹H NMR signals: 7.35 d, 6.84 d, 3.15 d, 3.7 m, 5.36 s. ¹³C NMR environments: c_1 157.97, c_2 and c_6 116.34, c_3 and c_5 125.1, c_4 129.4, c_2' 64.2, c_3' 59, c_4' 60. λ_{max} is 281 nm.

Epoxide given by *p*-coniferly alcohol (III), 4-[3-(hydroxyl methyl)-oxiran-2-yl]-2-methoxyphenol: ¹H NMR signal: 7.1 d, 6.9 d, 6.96 s, 3.79 s, 3.07 s, 3.68 m, 4.47 s, 3.79 s. ¹³C NMR: c_1 145.9, c_2 148, c_3 107, c_4 129.4, c_5 117.3, c_6 115.7 c_2' 62.8, c_3' 58.4, c_4' 60.4. λ_{max} is 287 nm.

Epoxide given by *p*-sinaply alcohol (III), 4-[3-(Hydroxyl methyl)-oxiran-2-yl]-2,6-dimethoxy phenol: ¹H NMR signals: 6.64 s, 3.8 t, 3.0 m, 3.67 m, 4.09 s, 3.85 s. ¹³C NMR environments: c_1 134.6, c_2 and c_6 148.4, c_3 and c_5 101.8, c_4 136.7, c_2' 61.3, c_3' 57.8, c_4' 60.5. λ_{max} is 288 nm.

Dimer formed *p*-coumryl alcohol (IV), 4-{3-(Hydroxyl methyl)-5-[(1E)-3-hydroxyprop-1-en-1-yl]-2,3-dihydro-1-benzofuran-2-yl}phenol (Model dimer1): ¹H NMR signals: 7.33 d, 6.84 d, 5.52 d, 3.52 m, 3.9 m, 4.3 s, 6.8 d, 7.2 d, 7.49 s, 6.54 d, 6.12 m, 4.37 d. ¹³C NMR environments: C_2 87.4, C_3 54.6, C_4 130, C_5 128.1, C_6 129.4, C_7 123.7, C_8 109.8, C_9 160.6, C_1' 130.9, C_2' 128, C_3' 63.3, C_1'' 134.5, C_2'' and C_6'' 127.3, C_3'' and C_5'' 116, C_4'' 158.6, C_1''' 64.4. λ_{max} is 305 nm.

Dimer formed *p*-coniferly alcohol (IV), 4-{3-(Hydroxyl methyl)-5-[(1E)-3-hydroxyl prop-1-en-1-yl]-7-methoxy-2,3-dihydro-1-benzo furan-2-yl}-2-methoxy phenol. (Model dimer2): ¹H NMR signal: 3.55 m, 3.8 s, A₂B type 3.97 m, 4.19 d, 5.16 s, 5.56 d, 6.23 m, 6.5 d, 6.8 d, 6.87 d, 6.94 s, 6.97 s, 7.03 s. ¹³C NMR: C_3 53.7, C of methoxyl 55.9, C_3'' 62.8, C of hydroxyl methyl 63.7, C_2 87.8, C_3' 110.2, C_3 111, C_5 and C_6' 115.5, C_5' 119, C_2'' 127.8, C_1'' and C_4 130, C_4' 133, C_8 144.5, C_1' 146.7, C_9 and C_2' 147.8-148. λ_{max} is 312 nm.

Dimer formed *p*-coumryl alcohol (V), 4,4'-Tetrahydro-1*H*,3*H*-furo[3,4-*c*]furan-1,4-diyl diphenol. (Model dimer3): ¹H NMR signal: 3.09

m, 3.92 d, 4.22 d, 4.66 d, 6.64 d, 7.22 d, 8.11 s. ^{13}C NMR: C_3 and C_7 55, C_2 and C_6 64.5, C_4 and C_8 86.7, C_{11} C_{13} C_{17} C_{19} 116.1, C_{10} C_{14} C_{16} C_{20} 128.3, C_9 C_{15} 133.8, C_{12} and C_{18} 157.7. λ_{max} is 282 nm.

Dimer formed *p*-coniferly alcohol (V), 4,4'-Tetrahydro-1*H*,3*H*-furo[3,4-*c*]furan-1,4-diylbis(2-methoxyphenol), (Model dimer4): ^1H NMR: 3.08 m, 3.8 m, 3.83 s, 4.31 d, 6.78 d, 6.83 d, 6.98 s, 7.48 s. ^{13}C NMR: C_3 and C_7 49.5, C of methoxyl 55.8, C_2 and C_6 68.4, C_4 81.4, C_3' and C_3'' 109.8, C_6' and C_6'' 114.9, C_5' and C_5'' 118.8, C_4' and C_4'' 129.7, C_1' and C_1'' 145.8, C_2' and C_2'' 147.3. λ_{max} is 287 nm.

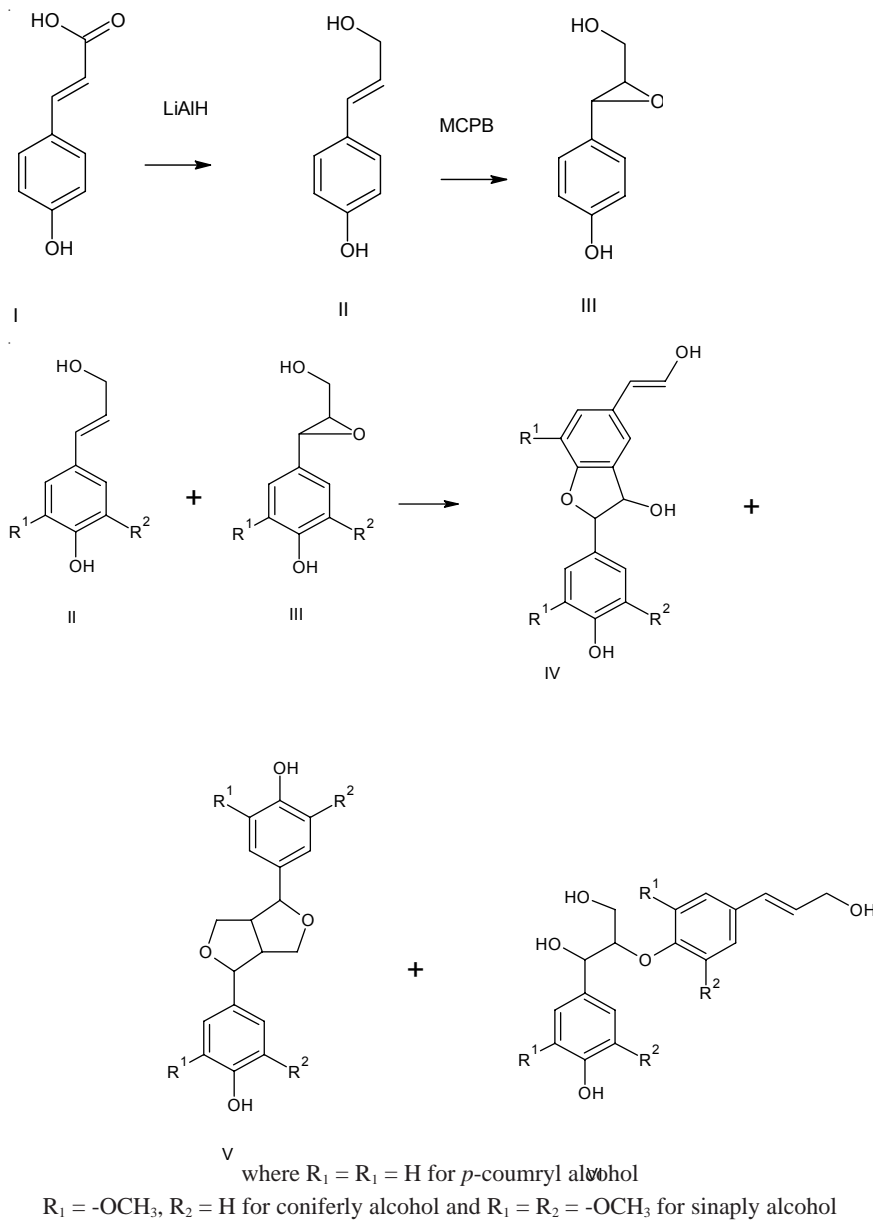
Dimer formed *p*-sinaply alcohol (V), 4,4'-Tetrahydro-1*H*,3*H*-furo[3,4-*c*]furan-1,4-diylbis (2,6-dimethoxyphenol). (Model dimer5): ^1H NMR: 3.08, 3.87 s, A_2B type 3.96 m, 4.32 and 4.32, 5.52 s, 6.59 s. ^{13}C NMR: C_3 and C_7 54.4, C of methoxyl 56.3, C_2 and C_6 70.4, C_4 and C_6 86, C_3' C_5' C_3'' C_5'' 103.6, C_4' C_4'' 132.2, C_1' C_1'' 135.1, C_2' C_6' C_2'' C_6'' 147.9. λ_{max} is 288 nm.

Dimer formed *p*-sinaply alcohol (IV) is not isolated. Only (V) is appears to be forming under the reaction condition adopted. Further VI type of compound too not isolated in HPLC analysis of products formed after 2nd stage of reaction scheme when *p*-coumryl alcohol or coniferly alcohol.

Non-cyclic dimer of *p*-coumryl alcohol (VI) formed by β -O-ether bond (Model dimer 6): ^1H NMR signals: 3.55 m, 4.17 d, 4.4 t, 4.94 d, 5.29 s, A_2B type signal 6.26 m, 6.5 d, 6.77 d, 6.9 d, 7.31 m. ^{13}C NMR signals: C_γ 61, C_3'' 63.9, C_α 72.49, C_β 83.39, C_2 and C_6 113, C_2' and C_6' 115.99, C_2'' 126.7, C_3 and C_5 127.68, C_3' and C_5' 129.27, C_1'' 130.8, C_4 130.7, C_4' 133, C_1' 157.16, C_1 158.77. λ_{max} is 302 nm.

Non-cyclic dimer of *p*-coniferly alcohol (VI) formed by β -O-ether bond (Model dimer 7): ^1H NMR signals: A_2B type at 3.57 m, 3.8 s, 4.18 d, 4.28 m, 4.89 d, 5.08 s, A_2B type at 6.25 m, 6.49 d, 6.74 d, 6.84 d, 6.99 d, 7.09 s. ^{13}C NMR signals: C of methoxyl groups 56, C_γ 61.2, C_3'' 62.82, C_α 72.89, C_β 86.65, C_3 and C_3' 109.89, C_6' 114.8, C_6 118.21, C_5 and C_5' 120.2, C_2'' 128.78, C_1'' 129.88, C_4 131.58, C_4' 133.42, C_1' 145.53, C_2' 146.83, C_2 147.9, C_1 151. λ_{max} is 308 nm. On signeryl alcohol only IV compound is formed probably due to methoxy groups at 2 and 6 positions attacking of phenoxide group of phenol on epoxide at neither α or β positions is seems to be hindered. The dimer formed by attacking at α position of epoxide seemed to have favourable conformation to give cyclization product III. Though dimer formation through β -O- bond is possible the corresponding benzo furan derivative VI is not forming.

4-{2-(Hydroxyl methyl)-5-[(1*E*)-3-hydroxyl prop-1-en-1-yl]-2,3-dihydro-1-benzofuran-3-yl}phenol (VI) is not formed during second stage of scheme of synthesis probably attacking of phenol functional group at C_2 of epoxide that favours and stabilized by phenyl group. On analyzing ^{13}C



Scheme-I

NMR data obtained for dimer peak at 87 ppm suggests that **IV** structure may assign to dimer. As methyl proton of biphenyl group would give its peak in the range of 44-47. Further if **VII** is the structure of dimer formed it might have given 12 kinds of proton signals, where 13 signals are showing in ^1H NMR of dimer. Therefore doublet at 5.52 δ and multiplet at 3.49 δ are assigned to proton of α (C_7) *i.e.* benzyl alcohol part of structure and β (C_{12}), respectively.

The important consideration in photocatalytic reactions is the adsorption of organic compounds on the surface of the catalyst particles. It has been often reported that adsorption of the organic compound is a prerequisite in photo degradation of organic compounds⁷. In order to evaluate the role of adsorption the dark adsorption isotherm for the model compounds was measured at different initial concentrations and mass of catalysts⁸. After allowing complete adsorption the equilibrium concentration was measured. On plotting $\log C_{eq}$ against $\log x/m$ where x is the mass of adsorbed species and m is the mass of catalyst. All are found to be following Freundlich adsorption isotherms⁹ at a concentration of below 10^{-4} mol/L at pH of 12. The intercept ($\log K$) and slope ($1/n$) values obtained from graph are given in the Table-1.

TABLE-1

	Apparent rate constant	Apparent equilibrium constant
Model dimer 1	0.58	1.29
Model dimer 2	0.76	1.62
Model dimer 3	0.52	1.36
Model dimer 4	0.66	1.49
Model dimer 5	0.79	1.58
Model dimer 6	0.82	1.74
Model dimer 7	0.90	1.83

Effect of UV radiation without catalyst: Aqueous and ethanolic solution of the compound was exposed to UV radiation for 3 h in the absence of TiO_2 . It was noticed that the compound is quite stable under UV radiation.

Effect of TiO_2 with out UV radiation: Aqueous and ethanolic solution of the compound was kept in darkness for 3 h in the presence of TiO_2 . It was noticed that the compound is quite stable under this conditions.

Effect of initial concentration: Experiments carried out with varying initial concentrations of compound. At the natural pH of the solution the rate of degradation increased with an increase in $[compound]_0$ and remain constant for beyond certain level, indicative of Langmuir kinetics. This is confirmed by the linear plots of $1/rate$ vs. $1/[compound]_0$ with an intercept on the ordinate (Figs. 2-8), in agreement with the equation¹⁰.

$$Rate = k'K[compound]_0 / 1 + K[compound]_0$$

Hence

$$1/rate = 1/k'K[compound]_0 + 1/k'$$

where k' = a proportionality constant and K = equilibrium constant. The values of k' and K obtained from the intercept and slope of this plot are k' mol dm^{-3} s^{-1} and K dm^3 mol⁻¹ respectively¹¹.

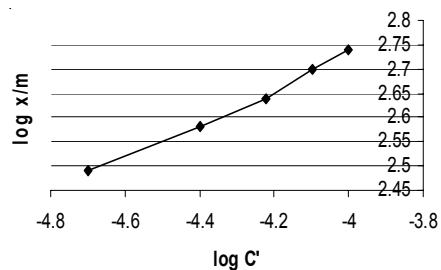


Fig. 2. Freundlich adsorption isotherm of model dimer 1

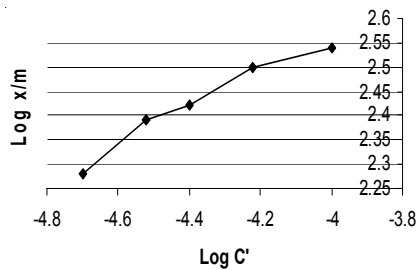


Fig. 3. Freundlich adsorption isotherm of model dimer 2

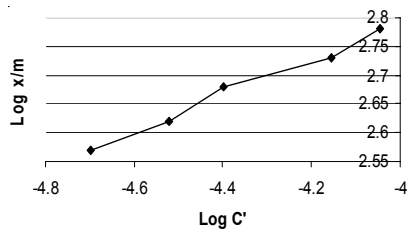


Fig. 4. Freundlich adsorption isotherm model dimer 3

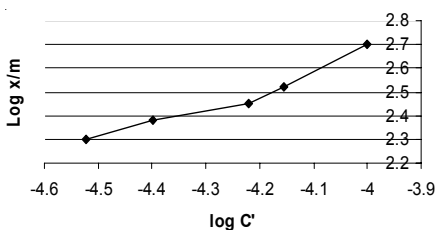


Fig. 5. Freundlich adsorption isotherm model dimer 4

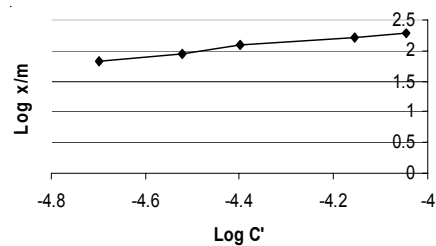


Fig. 6. Freundlich adsorption isotherm model dimer 5

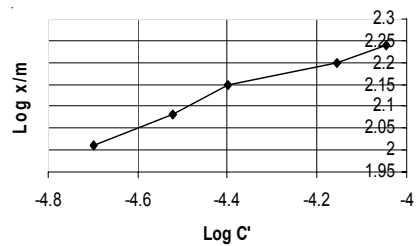


Fig. 7. Freundlich adsorption isotherm model dimer 6

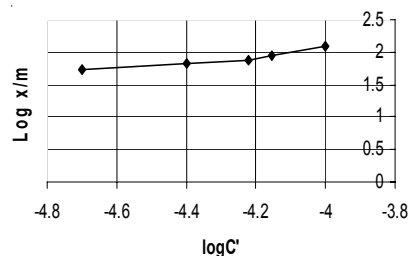


Fig. 8. Freundlich adsorption isotherm model dimer 7

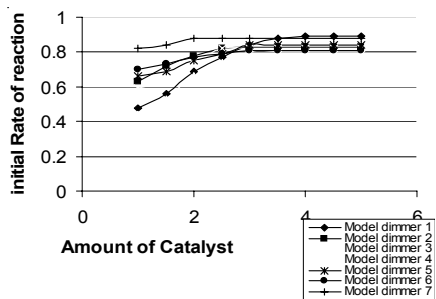
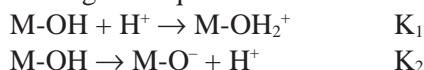


Fig 9. Plot of amount of catalyst against initial reaction rate

Effect of amount of catalyst: The effect of varying the amount of catalyst is studied (Fig. 9). Experiments performed with various amount of catalyst powder (1-5 g/L) and constant compound concentration ($0.0001 \text{ mol dm}^{-3}$) showed the rate increased with an increase in the amount of catalyst and remained almost constant above a certain level.

Effect of pH: The role of the pH on the rate of photolytic degradation was studied in the pH range. This is indicative of the significant role of the surface property of the photocatalyst (Figs. 10-12). The acid base property of the metal oxide surface has a considerable influence on the photocatalytic activity¹² with varying pH. The effect of pH can be explained on the basis of zero point of charge of TiO_2 . The adsorption of H_2O molecules at the surface metal sites is followed by the dissociation of OH groups leading to chemically equivalent metal hydroxyl groups. Due to the amphoteric behavior of most metal hydroxides, the following two equilibria must be considered¹³



The zero point of charge (pH_{zpc}) of metal oxide particle is defined as the pH at which the concentrations of protonated and deprotonated surface groups are equal¹⁴.

$$\text{pH}_{\text{zpc}} = \frac{1}{2} (\text{p}K_1 + \text{p}K_2)$$

The metal oxide surface is predominantly positively charged below pH_{zpc} and negatively charged above, the pH_{zpc} of TiO_2 is 3.5-6.7.

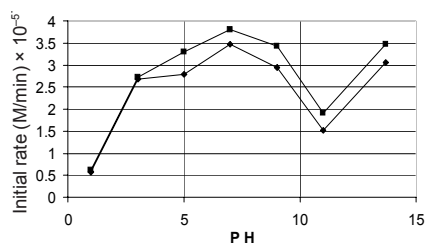


Fig. 10. Influence of pH on initial rate for model 1 and 2 compounds

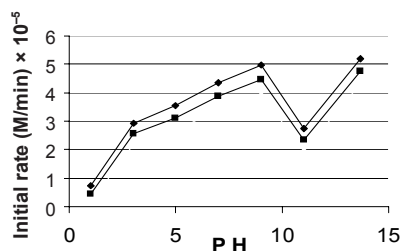


Fig. 11. Influence of pH on initial rate for model 3, 4 and 5 compounds

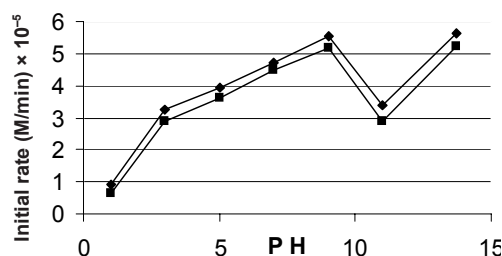
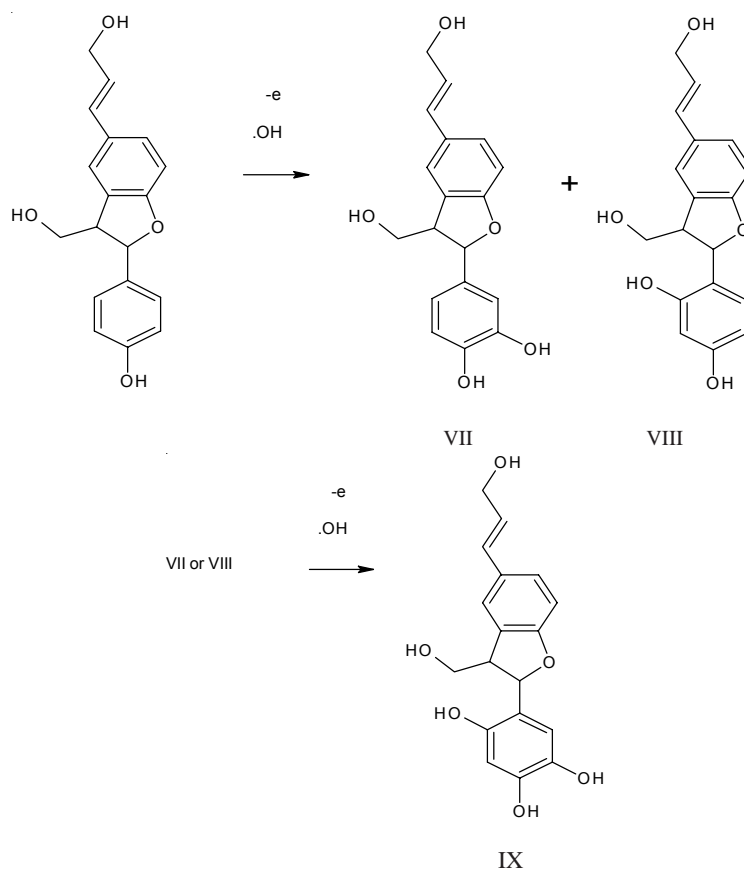
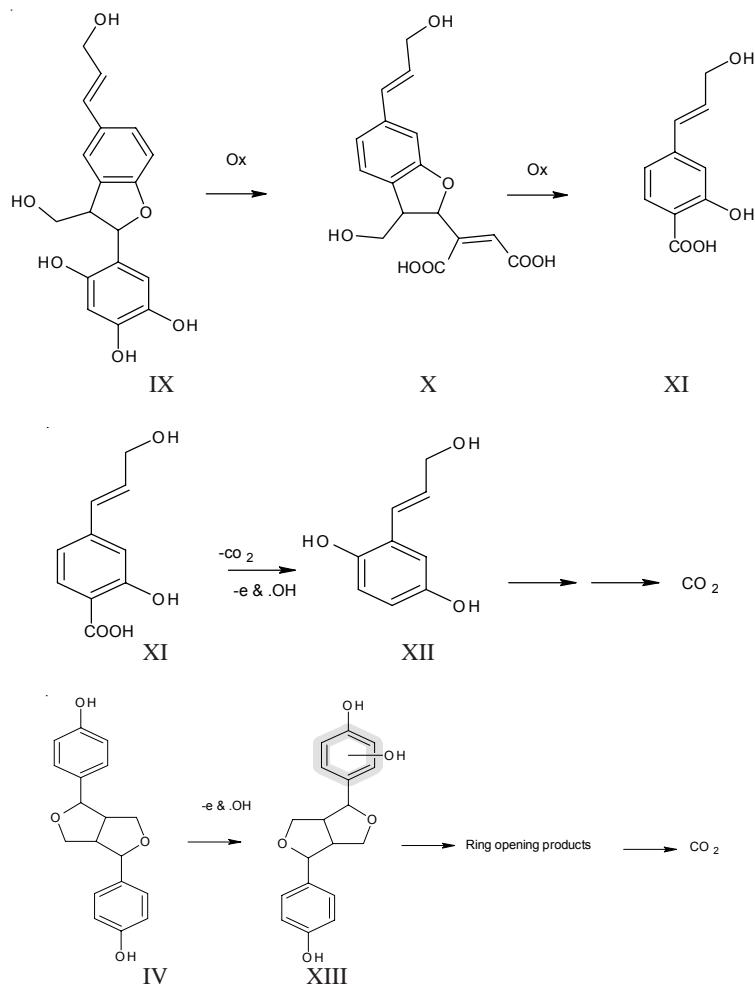


Fig. 12. Influence of pH on initial rate for model 6 and 7 compounds

Mechanism of photodegradation: In these experiments aqueous dispersion of the above substrates 0.0001 M, of 1000 mL of each model compound subjected to photocatalysis at pH 7 and 2.5 g of catalyst. After irradiation of UV light for 2 h UV spectrum of sample taken here below the superimposed spectra of model dimer 1 and spectra taken after 2 h subjection of catalysis is showing drastic change in absorption peak appearing. Similar shift of spectrum changes observed in the case of model dimer 3 too. This is an indication of degradation products, further to propose the probable path of photocatalysis variation of COD and phenolic contents with respective to time during photocatalysis is studied.

There is a gradual decrease in chemical oxygen required in mg/L for all model dimers. Whereas phenolic contents of all model dimer showed a gradual decrease initially, the unexpected increase found in case of model dimer 1, 2, 6 and 7 but for model dimer 3, 4 and 5 the behaviour is as expected. To explain the results obtained the following degradation path is proposed (**Scheme-II**).



**Scheme-II:** Degradation mechanism**Conclusion**

Without under going polymerization that gives a high molecular weight molecules by epoxidation followed by reaction with parent alcohol (*i.e.* from which the epoxide is prepared) gives dimers which are building blocks of lignin. So this initial study of degradation of model lignin dimers may be helpful in applications of advanced oxidation processes to treat effluents of paper mills, cover board manufacturers, textile mills and tanneries.

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