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Solar Radiation Catalyzed Aerobic Photooxidation of 1-Naphthol on Some Semiconductors

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Abstract - **Phototransformation of 1-naphthol into 1,2-naphthoquinone (1,2-NQ) using photocatalysts; TiO_2, V_2O_5, PbO_2, ZnO, Fe_2O_3, ZnS and Al_2O_3 has been studied in ethanol in the presence of air, under sunlight. The photooxidation exhibits saturation type kinetics with respect to [1-naphthol] and air. The rate of formation of 1,2-naphthoquinone increases linearly with respect to illumination area. The photooxidation rate is not suppressed by Singlet oxygen quencher, azide ion. Vinyl monomers and sacrificial electron donors do not interfere in photooxidation. The mechanisms of solar photocatalysis on semiconductor and non-semiconductor surfaces have been discussed and a kinetic law deduced. The solar photocatalytic efficiencies of the catalysts follow the order: $Al_2O_3 > Fe_2O_3 > V_2O_5 > TiO_2 > ZnO > PbO_2 > ZnS$. UV-visible, FT-IR and NMR spectroscopy spectral data clearly indicate that the photo product is 1,2-NQ.**

I. INTRODUCTION

Photochemical processes in heterogeneous systems have gained popularity in recent years because of their wide applications in degradation and mineralization of pollutants, bactericidal activity, chemical synthesis and conversion and storage of solar light energy. Semiconductor mediated photocatlysis involves photo-excitation that causes charge separation in semiconductor particles followed by simultaneous oxidation and reduction of adsorbed substrates.

Reports on photocatalyzed oxidation of a variety of substrates on semiconductor surfaces are numerous; titania is the most extensively used photocatalyst.[1-3] Photosensitization of the titania nanoparticles with carotenoids leads to the formation of superoxide anion and singlet oxygen on red light irradiation.[4] The dye photosensitization of zinc oxide occurs by injection of electron by the directly adsorbed dye.[5] Doping of titania with iron(III) shifts the adsorption to longer wavelength and the absorbance increases with increasing dopant concentration.[6] An increase in dopant ion content favours electron-hole separation and therefore enhances the photoactivity. Although doping of titania with chromium(III) shifts the absorbance to visible region the photocatalytic activity is nil in the visible region and diminished by 25-1000 times under UV light.[7] This is attributed to an increase in electron-hole recombination at the chromium(III) ion site. On the other hand, doping of titania with lithium increases the photocatalytic efficiency, as seen from the degradation of malachite green.[8]

The photo-oxidation of iodide on 60% and 80% of aqueous ethanol was studied as a function of iodide ion concentration, amount of catalyst suspended, airflow rate, light-intensity and solvent composition and wavelength of illumination, conform to Langmuir-Hinshelwood kinetic model. Iron(II) undergoes photooxidation generating iron(III)[12-14] which is simultaneously photoreduced on the metal oxides surfaces.[15]

The photooxidation of 4-chloroaniline in aqueous solution has been reported.[16] A photo-oxidation of aniline to azobenzene on titania in ethanol medium has been studied.[17] The catalytic efficiency of titania has been increased for the photooxidation of aniline by doping with transition metal (or) noblemetals.[18] The bimetallic catalyst of Au-Cu/Tio_2 has employed for the partial oxidation of methanol to produce hydrogen.[19]

The photooxidation of cyclohexane shows that the photocatalytic efficiency depends on the particle size of the photocatalyst.[20] The rate of photocatalytic production of hydrogen peroxide is 100 to 1000 times faster with Q-sized particles than with bulk zinc oxide particles.[21] The photocatalytic oxidation of ethanol on small and large zinc sulfide particles reveals selective oxidation,[22] ethanol is selectively oxidized at micrometer particles to acetaldehyde without side products by a “two hole” process. In the case of nanometer particles the primarily formed α-hydroxyethyl radicals in a “one hole” process undergo a secondary reaction, *i.e.,* the dimerization and disproportionation of the free radicals. A two hole process on nanometer particles becomes impossible because the time interval between two successive photon absorption incidents which lead to a successful hole transfer process in a nanometer particle is much longer than the maximum life time of the α-Hydroxyethyl radicals formed in the first step. Photooxidation of 1- naphthol to 1,2-naphthoquinone has been studied extensively. The product of photooxidation of 1-naphthol affords significant amount of 1,2-naphthoquinone. [23-25] Oxidation of 1-naphthol using a sunlight on heterogeneous photocatalyzed oxidation of a variety of organic substrates are numerous literature lacks reports using solar radiation and hence this work.

II. EXPERIMENTAL

A. Materials

1-naphthol AR (merck) was used as received. TiO_2, Al_2O_3, ZnO, Fe_2O_3, PbO_2, ZnS and V_2O_5 were used as received from merck. Commercially available ethanol was distilled over calcium oxide and used.

B. Solar Photocatalysis

Photooxidations of 1 – naphthol using sunlight in presence of catalysts TiO_2, V_2O_5, PbO_2, ZnO, Fe_2O_3, ZnS and Al_2O_3 were made from 11.30 am to 12.30 pm during summer, February to May on open terrace exposed to sunlight. The intensity of solar radiation was measured using Digital Illuminance Meter (LUX-METER), Model: TES-1332A, TES Electrical Electronic Corp., Taiwan. The reactions were carried out in glass case; solutions of 1-naphthol (1-NAPH), prepared afresh were taken in wide reaction vessels of uniform diameter and catalyst beds covering the entire bottom of the reaction vessels were maintained. Air was bubbled using micro pumps without disturbing the catalyst bed. The volume of the reaction solution was kept as 25 mL and the loss of solvent due to evaporation was compensated periodically (every 20 min). The area of cross section of the reaction vessel was determined from the measured height of known volume of deionized distilled water taken in the reaction vessel.

C. Product Analysis and Estimation

Solar photooxidation of 1-naphthol in ethanol on TiO_2, Al2O3, ZNO, Fe2O3, PbO2, ZnS yields a single product as 1, 2-naphthoquinone (λmax = 380nm)[26] which is confirmed by TLC. The reaction solution was evaporated and the product isolated using column chromatography using benzene as eluent. The brownish yellow solid was obtained (mp: 147°). The analysis of the product by FT-IR spectrum of a powder sample of 1,2-naphthoquinone was recorded using Shimadzu (8400S) spectrometer using KBr disc and the NMR Spectra were record on a 300 MHz Bruker spectrometer (using $CDCl_3$ as a solvent). The UV-vis spectra of the reaction solutions irradiated with sunlight, were identical with that of reported 1, 2-naphthoquinone (λmax = 380nm).[27] The absorbance of the product formed conforms to the Beer-Lambert law and the product was estimated using the reported value of the molar extinction coefficient (3.162×10^3 L mol^{-1} cm^{-1}).[28] The photo product identified by using UV, IR and ^{1}H-nuclear magnetic resonance (NMR) spectral datas are: IR : 3319, 3051, 1701, 1458, 1515, 1579 and 1625 and 1273 cm^{-1} ; ^{1}H NMR: 8.10, 7.74, 7.40, 7.30, 7.10 and 6.70 ppm; ^{13}C NMR: 180, 178,144, 135, 134.5, 132, 131, 130, 129 and 128 ppm.

III. RESULTS AND DISCUSSION

A. Solar Photooxidation of 1-Naphthol in the Presence of Semiconductors

The measurement of solar radiation shows fluctuation of sunlight intensity during the course of the photooxidation even under clear sky (Fig.1, Table I). Also, the intensity of solar radiation is different on different days. Now, for solar photooxidation experiments of different reaction conditions carried out in a set, the quantum of sunlight incident on unit area was made the same, by carrying out the experiments simultaneously, side-by-side, thus making it possible to compare the solar results. The solar photooxidation results are consistent. Solar photooxidation of 1-naphthol in ethanol on Titania, vanadia, zinc oxide, ferric oxide, lead dioxide, zinc sulfide and alumina yields 1, 2-naphthoquinone as the product. The progress of the solar photooxidation on observed using TiO_2 catalysts at various time intervals is shown in Fig. 2. The spectra clearly show a maximum absorbance (λ_{max}) at 380 nm; however there is no such absorbance is observed at 0 min. It reveals that the maximum absorbance at 380 nm is corresponding to 1,2-naphthoquinone which was identical with that of reported in literature.[101] A couple of solar experiments of identical reaction conditions carried out simultaneously, side by side, yield results within ± 5% and this is so on different days. This reproducibility is not surprising as the quantum of solar radiation absorbed by the test is the same as that of the control (standard) and the ratio becomes independent of fluctuation.

B. Factors Influencing Solar Photocatalysis

The various factors such as concentration of 1-naphthol, air flow rate and bed area influencing the solar photocatalyzed reactions in ethanol was examined by carrying out a set of experiments simultaneously under identical illumination; also an experiment under standard conditions was carried out side by side with the entire rate measuring experiments. The rate of formation of 1,2-naphthoquinone (1,2-NQ) at different concentration of 1-naphthol at various semiconductor catalysts show that the oxidation rate increases with increase in the concentration and the variation conforms to Langmuir-Hinshelwood model (Fig. 3, Table II). The double reciprocal plot of rate *versus* [1-NAPHTHOL] affords straight line with positive *y*-intercept in all cases (Table III). The effect of airflow rate on the rate of formation of 1,2-NQ was also carried out. Study of the photo catalyzed oxidation 1-naphthol as a function of airflow rate reveals that the enhancement of photo catalysis with oxygen concentration in the formation of 1,2-NQ (Fig 4, Table IV). The variation of rate with airflow rate suggests Langmuir-Hinshelwood kinetic model. The double reciprocal plots of rate versus airflow rate confirm the same (Table V). The reaction was also studied without bubbling air but the solutions were not deaerated. The dissolved oxygen itself brings out the reaction but the photo oxidation is weak. The reaction does not occur in dark. The photo catalysts used do not lose their catalytic activities on irradiation. Study of the photocatalyzed oxidation of 1-naphthol as a function of illumination area on the rate of formation of 1,2-NQ was also carried out. The reaction rate increases linearly with the apparent area of the catalytic bed (Fig. 5, Table VI). The Photoformation of 1,2-NQ have been studied in the presence of surfactants, vinyl monomer and azide ion (Table VII). The surfactants, singlet oxygen quencher and azide ion does not interfere the photochemical reaction. The vinyl monomers neither retard and nor polymerize during the course of photocatalysis.

C. Mechanism

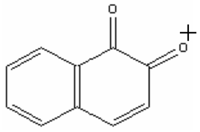
Of the seven photocatalyst employed in this study alumina is an insulator providing non-reactive surface while others are semiconductors with finite band gap energies. The bandgap excitation of semiconductors results in creation of electron-hole pairs such as holes in the valence band and electrons in the conductance band. Illumination of the semiconductors with light of an energy greater than the bandgap results in electron-hole pair generation. Since the recombination of a photogenerated electron-hole pair in a semiconductor is so rapid (occurring in a picoseconds time scale) (Eq. 3.1), for an effective photocatalysis the reactants are to be adsorbed on the catalysts[7]. The hole reacts with the adsorbed 1-naphthol molecule to form 1-naphthol radical-cation ($1-\mathrm{NAPH}^{+\bullet}$) (Eq. 3.2), while the electron is effectively removed by transfer to the adsorbed oxygen resulting in highly active superoxide radical-anion, $O_2^{-\bullet}$ (Eq. 3.3).[27] The 1-naphthol radical-cation may react with superoxide radical-anion yielding 1,2-naphthoquinone and water molecules (Eq. 3.4). And this mechanism is similar to the ZnO photocatalyzed oxidation of aniline in ethanol.[27]

$$Semiconductor + hv \rightarrow h^{+}_{(vb)} + e^{-}_{(cb)} \qquad (3.1)$$

$$1-NAPH_{(ads)} + h^{+}_{(vb)} \rightarrow 1-NAPH^{+\bullet} \qquad (3.2)$$

$$O_{2(ads)} + e^{-}_{(cb)} \rightarrow O_2^{-\bullet} \qquad (3.3)$$

$$1-NAPH^{+\bullet} + O_2^{-\bullet} \rightarrow$$



$$+ H_2O \qquad (3.4)$$

The donor and acceptor adsorbed on the photocatalyst surface undergoes photoexcitation followed by electron transfer. The donor excitation results in the transfer of excited electron whereas the acceptor excitation leads to an electron jump from the donor level to the vacant acceptor level.[28]

D. Kinetic Law

The photocatalysis on reactive as well as non-reactive surfaces requires adsorption of 1-naphthol and oxygen molecules on the catalyst surface. The rate of formation of 1,2-naphthoquinone is a function of

(i) The fraction of the surface adsorbed by the 1-naphthol molecule,
(ii) The fraction of the surface on which oxygen molecule is adsorbed,
(iii) The surface area of the catalyst bed, and
(iv) The intensity of illumination.

Hence

$$\text{Rate} = \frac{kK_1K_2A[1\text{-NAPHTHOL}]\gamma\, I}{1 + K_1\,[1\text{-NAPHTHOL}] + K_2\gamma + K_1K_2\gamma\,[1\text{-NAPHTHOL}]}$$

where K_1 and K_2 are the adsorption coefficients of 1-naphthol and oxygen molecules on the catalyst surface, k is the specific rate of oxidation of 1-naphthol, γ is the airflow rate, A is the surface area of the catalyst bed and I is the intensity of light. The fitment of the experimental data (Fig. 2 and 3) to the Langmuir-Hinshelwood curve, drawn using a computer program based on saturation kinetics with respect to 1-naphthol and airflow rate, confirms the kinetic equation. The kinetic expression explains satisfactorily the product formation as a function of 1-naphthol concentration, the airflow rate and the apparent surface area of the catalytic bed. The experimentally determined photocatalytic efficiencies of the catalysts reveal that they are not determined solely by the bandgap energies.

IV. CONCLUSION

Solar radiations catalysed photooxidation of 1-naphthol in ethanol in the presence of air on semiconductors yields 1-naphthoquinone and the conversion follows satutation kinetic with respect to 1-naphthol and airflow rate. The kinetic expression explains satisfactorily the product formation as a function of 1-naphthol concentration, the airflow rate and the apparent surface area of the catalytic bed.

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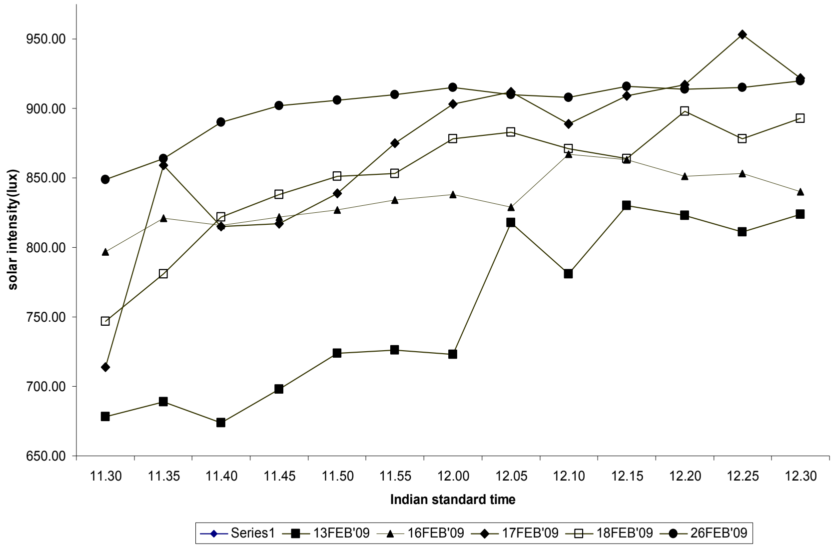

Fig. 1 Solar Intensity measured using LUX-meter

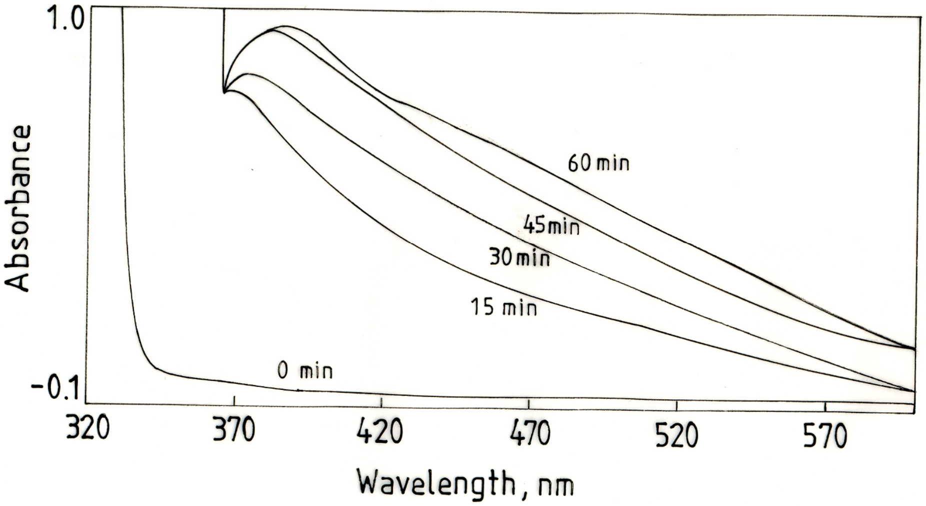

Fig. 2 Photoformation of o- naphthaquinone in ethanol in the presence of TiO_2 at various time intervals. The UV-visible spectrum of the irradiated solutions. [1-NAPH] = 5.0 mM; catalyst bed area = 11.36 weight of catalyst = 1.0 g; airflow rate = 8.74 mL s^{-1}; volume of reaction solution = 25 mL.

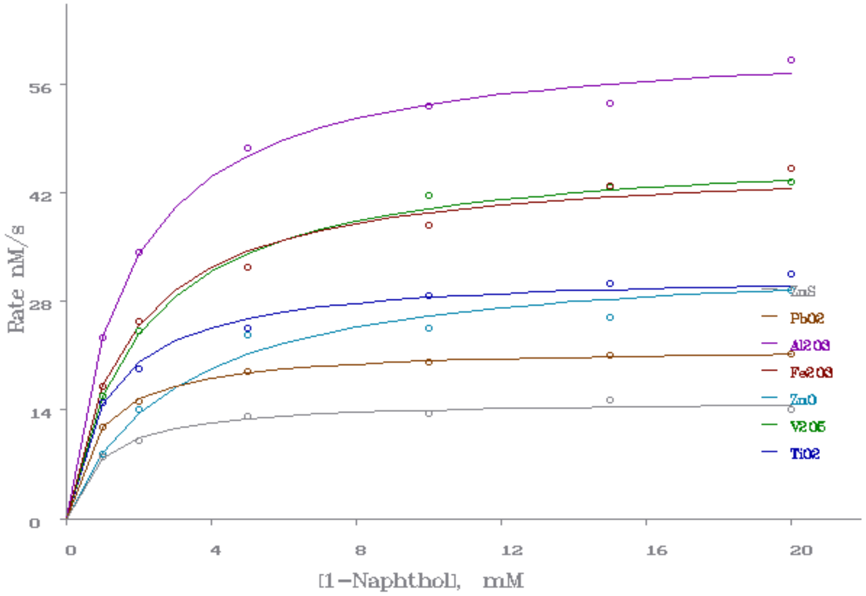

Fig. 3. Heterogeneous solar photooxidation of 1-Naphthol at different [1-NAPH]. Weight of Catalyst = 1.0 g; airflow rate = 8.74 mL s^{-1}; bed area = 11.36 cm^2; solvent: ethanol; volume of reaction solution = 25 mL.

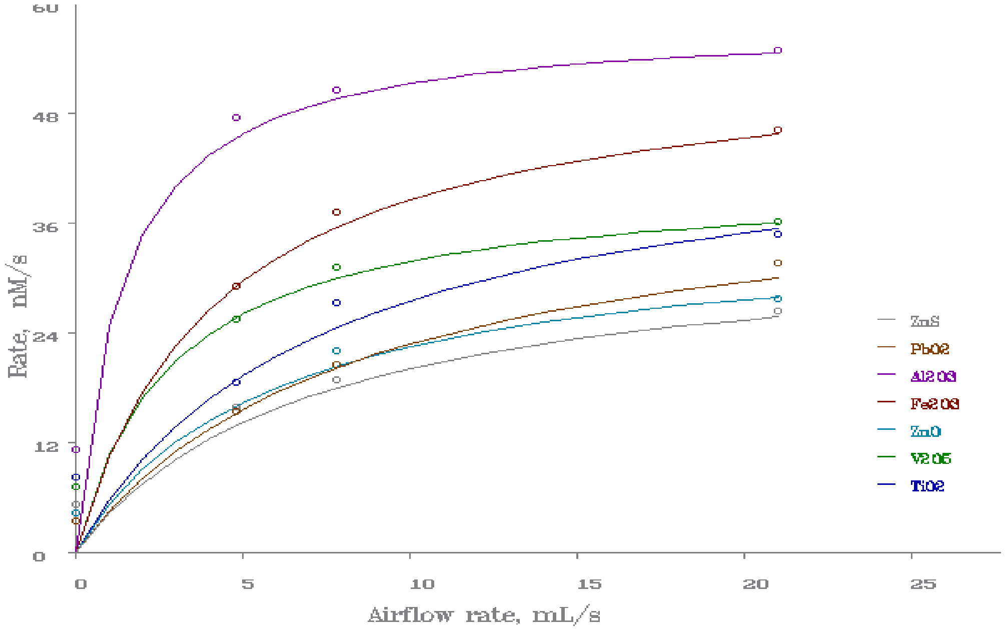

Fig. 4 Dependence of solar photooxidation rate of 1-naphthol on airflow rate . [1-NAPH] = 5.0 mM; weight of catalyst = 1.0 g; bed area = 11.36 cm^2; solvent: ethanol; volume of reaction solution = 25 mL.

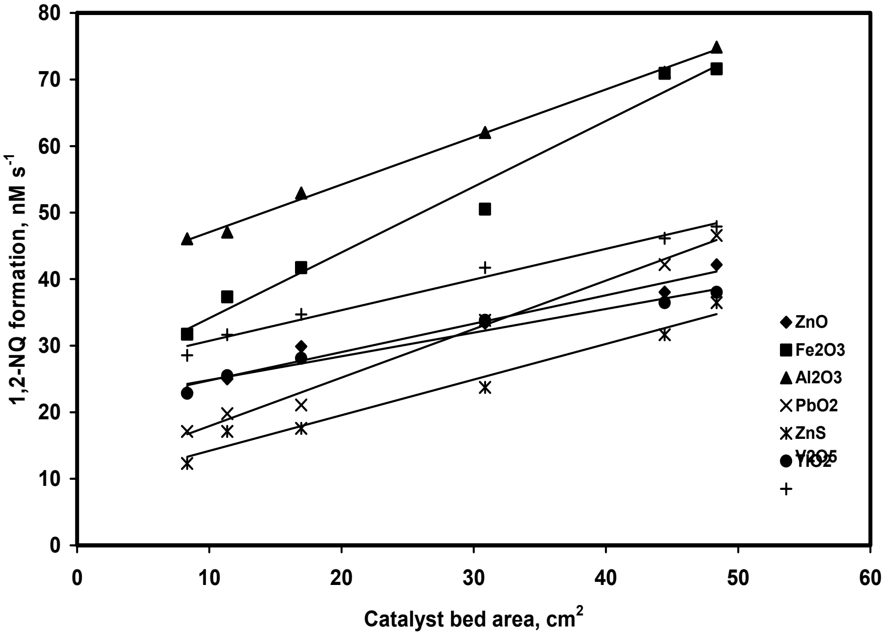

Fig. 5 Solar photooxidation of 1-Naphthol in ethanol as a function of illumination area. [1-NAPH] = 5.0 mM; weight of catalyst = 1.0 g; airflow rate = 8.74 mL s^{-1}; Solvent: ethanol; volume of reaction solution = 25 mL.

TABLE I SOLAR INTENSITY MEASURED USING LUX-METER

IST	**Solar intensity × 100 LUX**				
	13 Feb 2009	**16 Feb 2009**	**17 Feb 2009**	**18 Feb 2009**	**26 Feb 2009**
11.30	678	797	714	747	849
11.35	689	821	859	781	864
11.40	674	816	815	822	890
11.45	698	822	817	838	902
11.50	724	827	839	851	906
11.55	726	834	875	853	910
12.00	723	838	903	878	915
12.05	818	829	912	883	910
12.10	781	867	889	871	908
12.15	830	863	909	864	916
12.20	823	851	917	898	914
12.25	811	853	953	878	915
12.30	824	840	922	893	920

TABLE II SOLAR PHOTOOXIDATION OF 1-NAPHTHOL: 1,2-NQ FORMATION AT DIFFERENT [1-NAPH]

[1-NAPH], mM	**1,2-NQ formation, nM s^{-1}**						
	ZnO	**Fe_2O_3**	**Al_2O_3**	**PbO_2**	**ZnS**	**TiO_2**	**V_2O_5**
1.0	8.34	17.13	23.28	11.88	7.9	15.00	15.81
2.0	14.06	25.47	34.26	15.21	10.10	19.32	24.16
5.0	23.72	32.50	47.87	18.89	13.18	24.59	32.42
10.0	25.91	37.78	53.15	20.28	13.61	28.81	41.72
15.0	24.60	43.00	53.58	21.04	15.37	30.30	42.78
20.0	29.43	45.24	59.29	21.23	14.05	31.62	43.48

Weight of catalyst = 1.0 g; catalyst bed = 11.36 cm^2; airflow rate = 8.74 mL s^{-1}; solvent: ethanol; volume of reaction solution = 25 mL.

TABLE III THE DOUBLE RECIPROCAL PLOT OF RATE (NM S^{-1}) *VERSUS* [1-NAPH]

(1/[1-NAPH]), mM	**1/(Rate of 1,2-NQ formation), (1/ nM s^{-1}) × 10^8**						
	ZnO	**Fe_2O_3**	**Al_2O_3**	**PbO_2**	**ZnS**	**TiO_2**	**V_2O_5**
1000	1.199	0.5830	0.4255	0.8418	1.2659	0.6665	0.6325
500	0.7112	0.3926	0.2918	0.6575	0.9900	0.5175	0.4139
200	0.4210	0.3076	0.2088	0.5294	0.7587	0.4066	0.3085
100	0.3850	0.2646	0.1881	0.4931	0.7347	0.3471	0.2397
66.66	0.4065	0.2325	0.1866	0.4753	0.6506	0.3300	0.2337
50	0.3398	0.2210	0.1686	0.4710	0.7117	0.3162	0.2299
r	0.995	0.996	0.999	0.999	0.993	0.993	0.998

TABLE IV SOLAR PHOTOOXIDATION OF 1-NAPHTHOL: 1, 2-NQ FORMATION AT DIFFERENT AIRFLOW RATES

Airflow rate mL s^{-1}	**1,2-NQ formation, nM s^{-1}**						
	ZnO	**Fe_2O_3**	**Al_2O_3**	**PbO_2**	**ZnS**	**TiO_2**	**V_2O_5**
0	4.33	5.14	11.10	3.35	5.23	8.25	7.19
4.8	15.81	28.99	47.44	15.37	15.81	18.45	25.48
8.7	22.05	37.07	50.51	20.50	18.80	27.23	31.18
23.0	27.67	46.17	54.90	27.67	26.35	34.70	36.01

[1-NAPH] = 5.0 mM; weight of catalyst = 1.0 g; catalyst bed = 11.36 cm^2; solvent: ethanol; volume of reaction solution = 25 mL.

TABLE V THE DOUBLE RECIPROCAL PLOT OF RATE (NM S^{-1}) *VERSUS* (FLOW RATE)

(1/flow rate) mL s^{-1}	**1/ (Rate of 1,2-NQ formation), (1/ nM s^{-1}) × 10^8**						
	ZnO	**Fe_2O_3**	**Al_2O_3**	**PbO_2**	**ZnS**	**TiO_2**	**V_2O_5**
0	2.309	1.9455	0.9009	2.9850	1.9120	1.2120	1.3908
0.2083	0.6325	0.3449	0.2210	0.6506	0.7241	0.5420	0.3925
0.1144	0.4535	0.2697	0.1979	0.4878	0.5319	0.3672	0.3207
0.0434	0.3614	0.2168	0.1821	0.3162	0.3795	0.2881	0.2777
r	0.995	0.999	0.999	0.996	0.999	0.991	0.998

TABLE VI SOLAR PHOTOOXIDATION OF 1-NAPHTHOL: 1, 2-NQ FORMATION AT DIFFERENT AREAS OF CATALYST BED

Catalyst bed, cm^2	**1,2-NQ formation, nM s^{-1}**						
	ZnO	**Fe_2O_3**	**Al_2O_3**	**PbO_2**	**ZnS**	**TiO_2**	**V_2O_5**
8.33	23.01	31.71	46.05	17.13	12.30	22.84	28.55
11.36	25.01	37.34	47.26	19.77	17.13	25.48	31.63
16.95	29.87	41.72	52.93	21.08	17.57	28.12	34.70
30.86	33.38	50.51	62.05	33.82	23.72	33.82	41.73
44.44	38.05	70.93	71.08	42.17	31.63	36.46	46.12
48.37	42.15	71.59	74.85	46.56	36.46	38.02	47.88

[1-NAPH] = 5.0 mM; weight of catalyst = 1.0 g; airflow rate = 8.74mL s^{-1}; solvent: ethanol; volume of reaction solution = 25 mL.

TABLE VI PHOTOOXIDATION OF 1-NAPHTHOL WITH SOLAR RADIATION IN THE PRESENCE OF SEMICONDUCTORS: 1,2-NQ FORMATION WITH SURFACTANTS, VINYL MONOMER AND AZIDE ION.

Surfactant	**1,2-NQ formation, nM s^{-1}**						
	ZnO	**Fe_2O_3**	**Al_2O_3**	**PbO_2**	**ZnS**	**TiO_2**	**V_2O_5**
-	24.16	36.90	47.73	19.50	17.85	24.99	31.63
SLS	39.97	47.88	51.83	28.11	42.60	25.48	40.41
CTAB	29.87	43.04	53.58	27.23	38.65	29.43	35.14
Acrylamide	25.12	37.09	48.15	21.96	19.05	26.35	33.38
Azide ion	26.23	37.97	48.32	20.25	19.76	23.95	32.85

[1-NAPH] = 5.0 mM; [reagents] = 5.0 mM; weight of catalyst = 1.0 g;
airflow rate = 8.74mL s^{-1}; solvent: ethanol; volume of reaction solution = 25 mL.