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Dear Dr. Aravamudhan

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Abstract Ref. No. **AOP039**

Paper Title: **UV/TiO₂/H₂O₂ Photo Catalysis: A Case for Investigating the Magnetic field Dependence.**

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UV/TiO₂/H₂O₂ Photo Catalysis: A Case for Investigating the Magnetic field Dependence

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Abstract

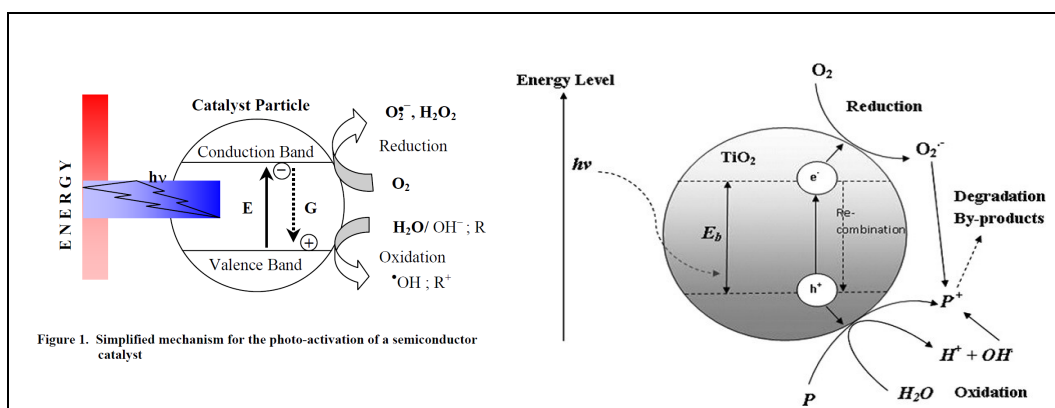
The decontamination of polluted water by the UV/TiO₂ has been well studied, and in particular, the effect of Dissolved Oxygen and the presence of Hydrogen Peroxide have been well documented in recent publications. The possible mechanisms involve generation of radicals and in certain cases recombination of the reactive radicals while the organic molecular pollutants are effectively mineralized. The radical pair mechanisms, which explain the occurrence of Dynamic Electron Polarizations and consequential Chemically Induced Dynamic Nuclear Polarizations, have also the consequence of affecting reaction dynamics involving free radicals.

To this context it may be worth noting that not only the radical pairs generated (by UV radiation) in singlet state, but also such of those generated radical pairs separating by diffusion and then again getting closer to distances enabling overlap of wave functions can influence the reaction dynamics. Until now, this author has not known any report in the literature on the effect of applied magnetic fields on the decontamination efficiencies. Hence, a consideration would be reported in this contribution, which can provide the nature of possible magnetic field effects on the UV/TiO₂/H₂O₂ photo catalysis

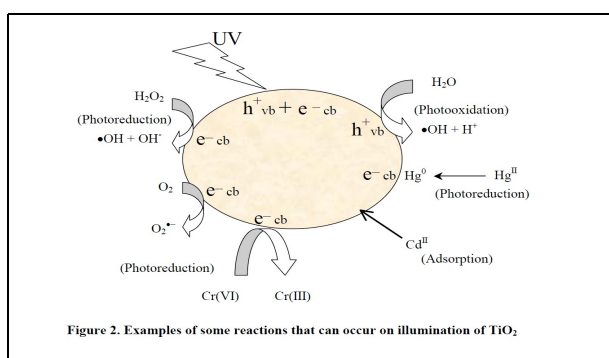
1. INTRODUCTION

The Photo catalytic reactions using a semiconductor like TiO₂ was found interesting in the context of production of Hydrogen from water and for the treatment of contaminated water and air particularly when the contaminant to be removed is an organic substrate. A simplified diagram (1) indicating the photo activation of semiconductor is given in Fig.1 It is mostly the hydroxide radical which plays an important role in oxidizing the substrate. The typical characteristics of heterogeneous surface catalysed reactions are described in most of

the Physical chemistry Text Books (1). The process consists of 5 steps. These are: “(i) diffusion of reactants to the surface, (ii) Adsorption of reactants onto the surface, (iii) reaction on the surface, (iv) Desorption of products from the surface, and (v) Diffusion of products from the surface” (3). Of these, the first step by which the substrates move to the surface is diffusion process that does not proceed through an activated state. Similarly the final step when the products formed on the surface move to the bulk is diffusion controlled and hence does not require any activation energy. When the radiation energy ‘ $h\nu$ ’ of



appropriate frequency (UV radiation using a lamp with λ_{max} at 365 nm is used) the valence band electrons are excited (3.2eV) to the conduction band, such an activated catalyst particle can be described as a hole-electron pair (h^+/e^-). And the h^+ is an electron acceptor and the e^- is an electron donor, by either of the two characteristics the substrate can be degraded. Further the presence of dissolved oxygen DO and H_2O_2 can facilitate the oxidation process



and make easier the mineralization of the organic substrate to carbon dioxide, elemental carbon, nitrogen and oxygen. Even though a direct photolysis has been tried out it is the photo catalysis in presence of TiO_2 and DO/ H_2O_2 which seems to be efficient way to oxidize the organic substrates.

In all these cases of the so many pathways it is the highly reactive $OH^\bullet$

radical which seem an important active species formed on the surface that causes the quick oxidation of the organic substrate. Then the efficiency of this process can be affected by the $OH^\bullet$ radical diffusing away and getting quenched by other $OH^\bullet$ radicals in the medium. The two radicals ($OH^\bullet$) when they get quenched what is the spin orientation of the two free electrons of the $OH^\bullet$ radicals is a question which can be considered from the point of view

of trying to reduce the quenching process and prolong the life of $\text{OH}\cdot$ radicals, so as to increase the availability for the organic solute and enhance the oxidative degradation. It is this aspect which is being addressed to in this paper.

When the two radicals combine to form a chemical bond, the two electrons must be in a singlet-state, ready to be a paired electron to be shared in the resulting bond of product molecule. In order to affect the reaction dynamics, this orientation of spins can be influenced by the presence of an external field provided the mechanisms operating when the two radical pairs by diffusion get to closer proximities when the wave-functions of the two electrons overlap and hence the spin-spin interaction becomes possible. When the photo catalysis is conducted in presence of an external magnetic field, the reaction dynamics would be affected and the monitored yield and reaction rates would provide an indication as to whether the oxidative degradation can be made more effective by altering the reaction conditions appropriately. When it is found that the effect of magnetic field is significant in the catalytic process, then the current day Photo-catalytic Membrane Reactors (PMRs) or the hybrid membrane distillation units must be designed(4) for appropriately applying the magnetic field where the photo catalysis at the slurry TiO_2 occurs.

At this stage, a consideration of the photochemical degradation organic molecules in aqueous media would be covered in the next section 2. A report of the main experimental results available in the publications on this subject is what is intended. The presence of hydroxyl radical $\text{OH}\cdot$ would be the emphasis.

In section 3, the processes involving hydroxyl radical which reduce the availability of the formed hydroxyl radicals would be discussed. As a step in the mechanisms the diffusion controlled separation of hydroxyl radicals and collision of two diffusing hydroxyl radicals would be considered with special reference to these hydroxyl radicals combining to result in diamagnetic products would be mentioned as possible way for the depletion of hydroxyl radicals, thus reducing the efficiency of degradation of organic molecules which are the solvent pollutants targeted in AOPs.

Further, in the next section 4 would contain a discussion of externally applied Magnetic Fields influencing the reaction dynamics of radical reactions and how in certain cases radical recombination is favored by Magnetic fields and in certain other cases the combination process gets reduced.

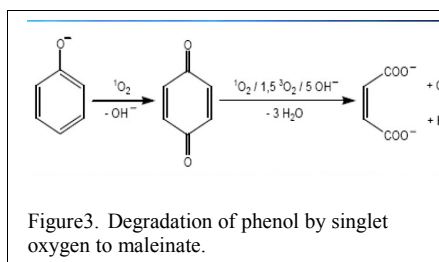
In section 5, the photo catalytic case of $\text{UV/TiO}_2/\text{H}_2\text{O}_2$ would be considered with the particular attention to $\text{OH}\cdot$ radical involved process in the mechanism and the possibility of uncorrelated spins after a diffusion process may still be responding to the magnetic field and influence the mechanism and yields. In the case of TiO_2 photo catalysis for the provision of magnetic field to be applied a special consideration may be required to design the reactor-apparatus which will be briefly described. Dissolve oxygen and particularly the presence of

H₂O₂ had considerable effect in the efficiency of photo degradation of organic pollutants and the H₂O₂ case is considered more specifically in this article.

2. PHOTOCHEMICAL OXIDATIVE DEGRADATION OF ORGANIC MOLECULES

Photodegradation and Photocatalysis (5) are usually oxidations of organic compounds in exothermic processes, in which the absorption of light energy is the driving reaction force. These photochemical methods, rather than the thermal decomposition, have been found to be advantageous in several ways for the removal of organic pollutants in water.

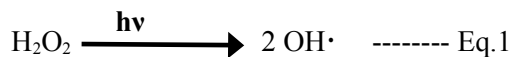
In *direct photochemical* degradation the electrons of an organic compound are excited. This can lead to decomposition of the organic molecule into two radicals, which are extremely reactive. More often the excited point of the molecule is attacked by oxygen molecules, which then oxidize the organic compound. As the reactivity of ground state triplet oxygen with the singlet ground state of the most compounds is very low, the excitation of oxygen to singlet is even more important for the oxidation of organic compounds. Thus is the importance of dissolved oxygen more generally the presence of reactive radical species in the photochemical degradation.



As the photo degradation of many microorganisms and compounds is very slow and lead to oxidation or degradation of organic compounds, the **advanced oxidation process (A.O.P.)** by combining UV illumination with ozone or H₂O₂ supply is much more effective often leading to complete mineralization.

This is achieved especially by the photolytic reaction of

H₂O₂. Thus this is the *indirect photochemical* degradation, where the electrons of the supplied H₂O₂ molecules are excited to form reactive radicals which react with organic molecules to decompose them. In the indirect photochemical oxidation radicals are formed by photolysis, which then attack the organic compounds. An example is the reaction of H₂O₂.



In the case of photo catalysis a free radical mechanism is responsible for the degradation process. Thus, the deactivation is likely to cause enhancement in the attack of OH• radicals. This may be the reason why chloro benzene (6) degrades rapidly. The formation of phenol as an intermediate also justifies this supposition. As hydroxide radicals need an electron to obtain an octet, they are very electrophilic and attack electron rich areas of any molecule. Thus the importance of OH• (hydroxyl radicals) becomes evident in the water treatment of polluted water by photochemical degradations. In photocatalysis a couple of semi conductive materials can be used for photocatalysis. Most popular is TiO₂ because of its

chemical stability and high efficiency. Refer to the schemes in Figure 1 and Figure 2 for the activity of this semiconductor catalyst also described in the section 1, introduction. These Ti^{+3} reaction centers act as electron holes some of which diffuse to the surface, where they can react with other molecules. Examples for possible reactions forming highly reactive radicals:

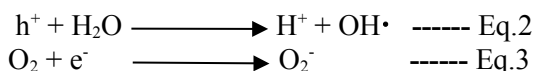
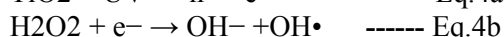
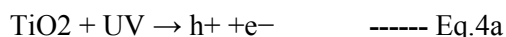


Photo catalysts can be used in the form of particles dispersed in the water. In this way the surface of the catalyst is used most efficiently but the dispersed particles disturb illumination. Additionally the particles have to be removed after the treatment. Another method is a fixed bed covered with TiO_2 . This elementary consideration of the photo degradation of organic molecules (organic pollutants in water) indicates the necessity to generate $OH\cdot$ radicals for efficient degradation and mineralization of the organic pollutants. The review article (7) discusses the generation of hydroxyl radical in section 3 and the subsection 3.2.3 deals with the radical-radical reactions. The section 3.5 deals with TiO_2 /UV related processes in connection with photo catalytic oxidation for water treatment. The study reported in the dissertation available in the repository (8) of Iowa State University has significant results pertaining to the $OH\cdot$ radical reaction with organic molecules containing benzene ring. These studies include experimental work and quantum chemical theoretical calculations. Also, the monitoring of the products by NMR spectroscopy is an indication of a significant viable tool in this context.

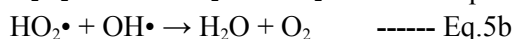
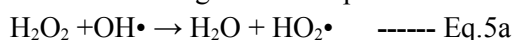
3. PROCESSES INVOLVING HYDROXYL RADICALS DURING PHOTOCATALYSIS: UV/ TiO_2 /H $2O_2$

It is known that H_2O_2 can enhance the reaction by providing additional hydroxyl radicals either through trapping of photo generated electrons and/or photolysis of H_2O_2 .



As mentioned in the previous section generation of $OH\cdot$ radical in the catalytic reactor and this reactive radical species are the factors that seemed to enhance the efficiency of photo catalytic oxidative degradation of organic pollutants in water. When the dosage was high, however, the degradation was suppressed due to the capture of $OH\cdot$ radicals and the competitive adsorption of H_2O_2 . In order to abate the disadvantages caused by using a higher H_2O_2 dosage, sequential replenishment of H_2O_2 into UV/ TiO_2 system was performed. Experimental results demonstrated that both degradation and mineralization efficiencies

were enhanced by the restraint of the capture of OH• radicals, the additional OH• radicals caused from the second stage addition of H₂O₂, and the participation of oxygen in photo catalytic degradation(9). When the oxidation is carried out in presence of H₂O₂, it was found that there was an increased availability of OH• radicals for the oxidative degradation of the organic pollutants (10). However beyond a certain concentration of H₂O₂ the presence of H₂O₂ decreased efficiency in the pollutant degradation was noted and the following process was envisaged for the removal of OH• radicals and hence the same was not available for the reaction with organic solute pollutant.

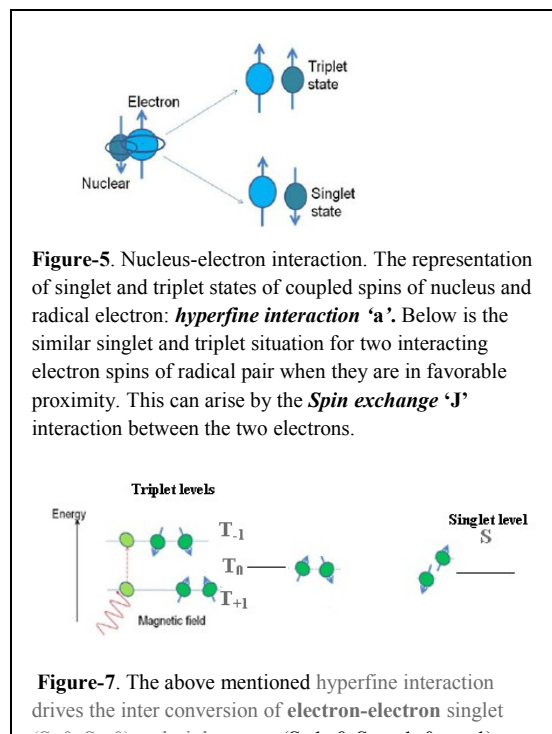
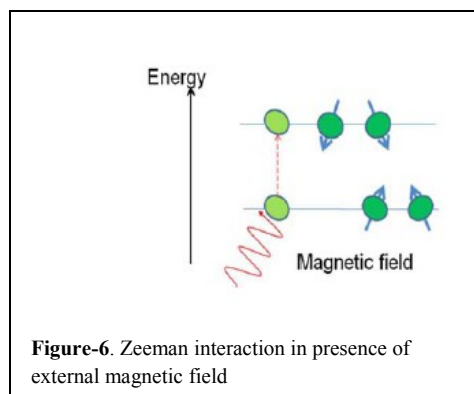
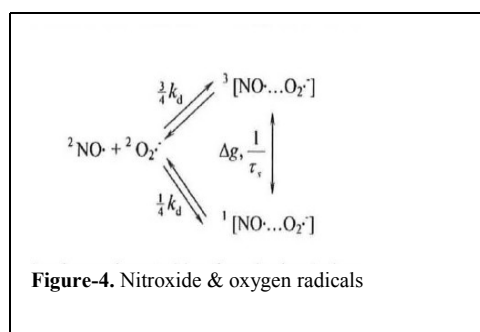


There are a series of chain oxidative-reductive reactions that occur at the photon activated surface was widely postulated. The possibility that the organic solute diffuses from the bulk to the catalytic surface in this case of heterogeneous catalysis (9) as can be envisaged from Figure-1, and get oxidized at the surface by reacting with the e⁻ of the photo generated h⁺/e⁻. But the most important factor at this juncture is the OH• radicals diffusing into the solvent medium (water) and reacting with the organic solute pollutant molecules. If the OH• radicals undergo recombination by the collision of two hydroxyl radicals, then the availability of the hydroxyl radicals is reduced and the oxidative degradation of organic pollutants would be reduced. Since the recombination of radicals is affected by the spin states of the radical pair, the magnetic field can influence the radical recombinations. Mostly it is the hydroxyl radicals which are diffusing in the system do not have much of spin state correlation for any given pair. It is to be remarked that the radical pairs generated by the photo excitation of a molecule would be a 'intimate radical pair' with the retention of a singlet state of the pair of electrons formed by the breaking of a bond.

4. EXTERNALLY APPLIED MAGNETIC FIELDS INFLUENCING REACTION DYNAMICS OF MECHANISMS INVOLVING FREE RADICALS

When the hydroxyl radicals, are freely diffusing in the medium, then there need not be any interaction between the two molecules to cause a spin-spin interaction of the electrons to correlate them and assign a coupled spin state (singlet or triplet) for the pair. When the molecules during diffusion approach each other, and get to such closer proximities, that the electron wave functions of the free electrons in the two molecules overlap (11) then the spin-spin interaction becomes possible. If the radical pair has to recombine by a bond formation then the two electron spins must get correlated to be in a singlet state so as to be spin paired for remaining "shared" in bond formation. It is this criterion that enables an intervention by external magnetic field. The spins which are interacting with an external magnetic field would follow the usual Boltzmann distribution for the non interacting spin

case. When the radicals get nearer to each other the spin-spin interactions (specifically of relevance is the spin-orbit coupling and the spin exchange) and depending upon the relative importance of these spin-spin interactions with respect to the Zeeman interaction with external magnetic field, the reaction dynamics can be affected. Can such influence of magnetic fields applied externally be of significance in the efficiency regulation of the



UV/TiO₂/H₂O₂ is the question raised and this seems to be a case to be investigated. That is, the magnetic field effects on this photocatalytic degradation due to the predominantly reactive hydroxyl radicals. To cite another context (12), for **Magnetic-Field Effect involving the small biologically relevant inorganic radicals NO and O₂⁻**, the scheme below was envisaged (Figure-4) More generally unpaired electron can interact with a nuclear magnetic moment when conditions are favorable, and they can be either in a singlet 'S' (spins antiparallel) state or three triplet state T₋₁, T₀, T₊₁ (spins parallel). This is an intra-radical coupling between the magnetic moment of an atomic nucleus and the magnetic moment of an unpaired electron. This is the hyperfine interaction and can be present even in the absence of an external field. This electron-nuclear coupling depends on the orientation of the molecule when an external magnetic field is present. And the hyperfine interaction drives the inter conversion of singlet and triplet states of the radical

pairs. As the diffusing Hydroxyl radicals approach each other, when the distance between the two radicals is much longer (11, 12 & 13) for the electron wave functions of the two electrons, one on each radical, to overlap, then only the intra-radical *hyperfine interaction* and *spin-orbit interactions* would prevail. That is the unpaired electron in the OH• radical with the proton of the radical and the orbital magnetic moment of the radical electron.

When the distance reduces, then at shorter distances the exchange interaction (between the two electrons) increases and might become even stronger than the hyperfine interaction. Thus of the two mechanisms the relative strengths can vary and as shown in figures 4-7, the coupling between the two electrons increases when the exchange interaction is strong. This exchange interaction results in distinct singlet (favorable for recombination by bond formation) and triplet spin states. If the recombination has to be reduced, the singlet states may not be formed, or there must be a way to influence the preferential formation of more of triplet. The formed singlet and triplet undergo singlet-triplet intersystem (spin system states) crossing by hyperfine interaction. When an external magnetic field is applied, the 3 energy levels of triplet states split. And the T_0 state of the triplet and the S_0 singlet would interconvert if the 'g' values of the radicals are different, which can be attributed to the different Larmor precession frequencies due to Zeeman interaction with external fields. Thus the inter conversion of the singlet and triplet (the intersystem crossing) can be affected by magnetic field. Thus this provides a possibility to influence the reaction dynamics if the mechanism involves free radical participation. Section-5 to follow contains descriptions more specific to the UV/TiO₂/H₂O₂ photo catalysis.

5. MAGNETIC FIELD EFFECTS FOR THE PHOTOCATALYTIC SYSTEM

UV/TiO₂/H₂O₂ The considerations for this photo catalytic system would be generally applicable where the OH• radicals are involved in the mechanism of reactions. The presence of H₂O₂ in photo catalytic reactions is known to generate the much required reactive OH• radicals. Earlier it has been observed that the magnetic fields can affect the reaction dynamics was known and the effect was noted to be not much significant but further studies and the theoretical models (14) did predict the possibility of significant weak magnetic field effects. Magnetic field effects by about a factor of 100 have been reported (15) in connection with enzyme kinetics and certain biological systems where the radical pairs are generated in the reaction media. If the photo catalytic reactors have the design considerations as mentioned (3 & 4) earlier in Section-1, then the fact that this magnetic field effect has to do with OH• radicals diffusing in the aqueous medium and not necessarily adsorbed to the surface of the TiO₂ is to be noted. First a laboratory scale experiment may be conducted where it is possible to include a flow system to let the water flow out with the

organic pollutant solute and the generated and diffusing $\text{OH}\cdot$ radicals, expose this to a magnetic field and monitor the yield of the CO_2 , H_2 etc., to infer the magnetic field effects. In the reactor it should be possible to provide for application of a magnetic field; in particular, if the field required is weak in the sense of a few Oertsteds, then an electromagnet can take care of the needs.

Since, the triplet spin states $T_{\pm}$ are split in a magnetic field (which may be degenerate in the absence of field) the magnetic field strength for the Zeeman interaction energy to compare with respect to the spin orbit coupling strength is a determining factor. If the splitting of $T_{\pm}$ in presence of the magnetic field is relatively large, then it is only by the difference in the 'g' factors of the electrons in the two radicals which might cause the triplet-singlet interconversion by the difference precession rates. Then with whatever probability the Singlet and Triplet states form as the radicals diffuse to closer distances would be retained without much change. Thus varying the magnetic field strengths from zero to a higher value gradually and monitoring the yields (degradation efficiencies) would be a certain way to ascertain the magnetic field effects and further construct models to regulate the mechanistic aspects related to the magnetic field effects. This would further enable a suitable water treatment reactor plant for the efficient photocatalytic oxidative reductions. Moreover, when the reactor plants are designed for a large turnover of treated water, even small increase in efficiency by application of magnetic field could be prove highly economical proposition.

In **conclusion**, in the present context, it seems worthwhile to consider an experimental study for the magnetic field effects on the efficiency of AOP using $\text{UV/TiO}_2/\text{H}_2\text{O}_2$.

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