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Photochemical reactions of *trans*-stilbene and 1,1-diphenylethylene on silica gel: mechanisms of oxidation and dimerization

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Abstract

The photochemical reactions of *trans*-stilbene and 1,1-diphenylethylene have been studied at a silica gel–air interface. Irradiation of *trans*-stilbene leads to the formation of a substantial amount of two dimers, along with the formation of *cis*-stilbene, phenanthrene and a small amount of benzaldehyde. Photochemical dimer formation from *trans*-stilbene on silica is promoted by inhomogeneous surface loading and slow surface diffusion. Benzaldehyde arises from a Type II oxidation mechanism. Irradiation of 1,1-diphenylethylene at a silica gel–air interface leads primarily to the formation of benzophenone. The oxidation of 1,1-diphenylethylene occurs by a Type I mechanism, as previously proposed [J. Org. Chem. 50 (1985) 149]. The differences in observed photochemistry for these two substituted phenylethylenes on silica is attributed to two factors. Isomerization of *trans*-stilbene competes efficiently with oxidation, whereas this pathway appears not to be important for 1,1-diphenylethylene. Singlet molecular oxygen is shown to be quenched in solution and at the silica gel–air interface at a faster rate by *cis*-stilbene than by *trans*-stilbene or 1,1-diphenylethylene, thus providing an enhanced Type II oxidation pathway for stilbene.

Keywords: Silica gel; Photochemical oxidation; Stilbene

1. Introduction

Photochemical oxidations of polycyclic aromatic hydrocarbons (PAH) at silica gel–air and alumina–air interfaces have been studied in our laboratory [2–14]. These studies have focused on understanding photochemical mechanisms through product studies and through the application of conventional physical organic methods originally developed for use in liquid phases. It is important to differentiate between the influence of surface–substrate interactions and inherent molecular properties of the substrate if we are to fully understand interfacial reactions. The PAH studied in our laboratory to date have been highly rigid aromatics with no significant conformational flexibility. In this paper we examine *trans*-stilbene and 1,1-diphenylethylene with an emphasis on the role of conformational interconversion of *trans*- to *cis*-stilbene and how that can influence photochemical oxidation mechanisms.

The emphasis of our silica gel-supported PAH studies has been to identify Type I (electron transfer) and Type II (energy transfer) oxidation mechanisms. These two mechanisms on silica gel are readily differentiated through the use of singlet oxygen sensitizers and traps, in much the same way that they are in solution. For example, the direct photolysis (300 nm) of 1-methoxynaphthalene on silica gel leads to phthalic acid, monomethylphthalate, 1,4-naphthoquinone and 4-methoxy-1,2-naphthoquinone [8]. Photolysis in the presence of 2,5-dimethylfuran, an efficient singlet molecular oxygen trap [15], eliminates the quinone products and decreases the photolysis rate proportional to their contribution to the product set. Photolysis (650 nm) of 1-methoxynaphthalene on silica gel in the presence of co-adsorbed methylene blue, a good singlet oxygen sensitizer, leads to the production of only the quinones. This stands in contrast to the photochemistry (300 nm) of naphthalene on silica gel and alumina where phthalic acid is the only observed product [14]. Singlet oxygen traps do not slow the naphthalene oxidation reactions on silica gel, and methylene blue sensitization does not lead to oxidation products. Direct photolysis of naphthalene on silica gel has been shown to lead to the formation of superoxide, as observed by EPR, through an electron transfer mechanism [14].

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We have generally observed that PAH which react readily with singlet molecular oxygen in solution will also react by this mechanism at the silica gel–air interface, while those that are not good singlet oxygen acceptors may react by an electron transfer mechanism. This general rule is violated in some cases. For example, phenanthrene is oxidized by addition of singlet molecular oxygen across the 9,10-position when adsorbed at a silica gel–air interface [10]. However, phenanthrene does not readily react with singlet oxygen in solution. Additionally, we have observed that 1-cyanonaphthalene is not photochemically oxidized at a silica gel–air interface, likely due to its high oxidation potential [14].

In related work, the photochemical oxidation of substituted phenylethylenes on inorganic oxides has been reported [1]. In that study, the photolysis of 1,1-diphenylethylene on silica gel was reported to produce a 98% yield of benzophenone. A Type I oxidation mechanism was proposed to proceed by direct excitation of a 1,1-diphenylethylene–oxygen charge-transfer contact pair on the surface. In related work, the photosensitized oxidation of alkenes adsorbed on pentasil zeolites (ZSM-5) has recently been reported [16]. The regular array of pores in ZSM-5 zeolites, having dimensions in the 5.2–5.8 Å range, allow the olefin and oxygen entry but restrict access of the sensitizer and solvent. The size limitations of the system permit singlet molecular oxygen mediated oxidation within the pores, while preventing electron-transfer oxidation processes. Among the olefins studied, *trans*-stilbene was reported to undergo a Type II oxidation to generate benzaldehyde as the sole product in dry zeolites. Stilbene *trans* to *cis* isomerization was not observed within the zeolite pores. Oxidation of *trans*-stilbene has also been observed to occur by a Type I mechanism in solution and in dye-exchanged X and Y zeolites [17].

This paper describes our study of the photochemical oxidation of *trans*-stilbene and 1,1-diphenylethylene at a silica gel–air interface. Singlet molecular oxygen sensitization and trapping studies have been used to verify the previously reported Type I mechanism for 1,1-diphenylethylene oxidation on silica gel [1]. The same techniques are used to determine the oxidation mechanism for *trans*-stilbene on silica gel. The study also extends the range of organic substrates investigated in our laboratory to include these two substituted phenylethylenes.

2. Experimental

The silica gel used in these studies has characteristics identical to those previously reported [8]. All solvents used were HPLC grade and used as received. 1,1-Diphenylmethane, *trans*-stilbene and *cis*-stilbene were purchased from Aldrich and used as received. The silica gel was loaded to the desired surface coverage by adsorbing the substituted phenylethylene onto the surface from a cyclohexane solution by removing the solvent under vacuum at ambient temperature. The samples were photolyzed in quartz tubes with 300 nm light from a Rayonet RPR-208 photoreactor (2.7×10^{15} photons $cm^2 s^{-1}$). Singlet molecular oxygen sensitized experiments used co-adsorbed methylene blue as a sensitizer. Sensitization experiments were done in Pyrex glass tubes in a Rayonet RPR-100 photoreactor fitted with 650 nm bulbs (2.6×10^{16} photons $cm^2 s^{-1}$). The free-flowing silica gel samples were mixed during photolysis by holding the tubes in a horizontal configuration while rotating on a merry-go-round apparatus.

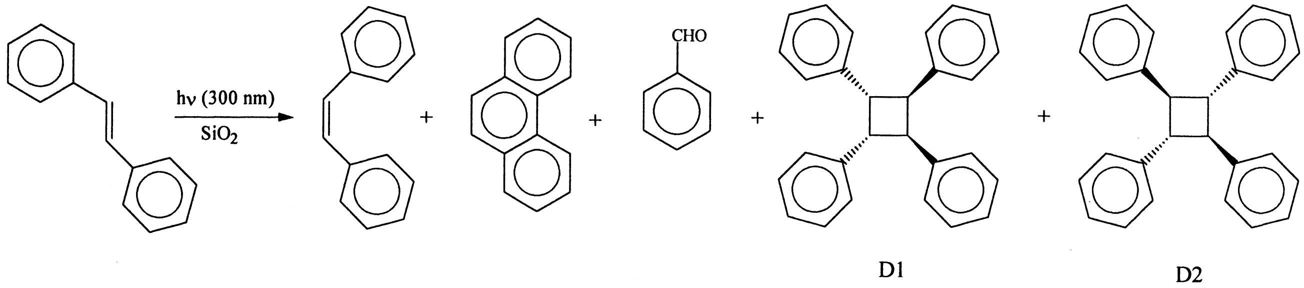
Products were analyzed quantitatively using a GC/FID with an internal standard method and known standards. Qualitative product determination was done by GC/MS(EI). *Trans*-stilbene dimer ratios were determined from the integrated ^{1}H-NMR peaks for the cyclobutyl protons as previously reported [18]. NMR spectra were measured in $CDCl_3$ with a Bruker 400 MHz spectrometer. Fluorescence spectra were recorded on a Spex Fluorolog fluorimeter and are reported to be fully corrected. Singlet molecular oxygen quenching rates were measured in our laboratory on an instrument patterned after a previously reported design [19].

3. Results and discussion

3.1. *Trans-stilbene*

Photolysis of *trans*-stilbene (300 nm, 4 h, 96% conversion) adsorbed at a silica gel–air interface (2.5×10^{-5} mol g^{-1}) led to isomerization to *cis*-stilbene (27 mol%), oxidation to benzaldehyde (2 mol%), and cyclization of *cis*-stilbene to give phenanthrene (42 mol%). The remainder of the material balance was composed of two tetraphenylcyclobutane dimers of stilbene, designated D1 and D2 in Scheme 1. Photolysis (4 h, 87% conversion) at a fourfold higher surface loading (1.0×10^{-4} mol g^{-1}) proceeded to give *cis*-stilbene (60 mol%), benzaldehyde (3 mol%), and phenanthrene (8 mol%). The remainder of the material balance was composed of dimers D1 and D2. Photolysis of *trans*-stilbene proceeds slower at the higher surface coverage and the main difference in product distribution appears to arise from a retardation of *cis*-stilbene to phenanthrene conversion. Phenanthrene formation, a secondary photochemical event, may be slowed at the higher coverage as the result of competitive light absorption by excess *trans*-stilbene, similar to an inner-filter effect in solution. Slowing of photochemical conversion with increasing surface coverage has previously been observed on silica gel in our laboratory [8].

Dimers D1 and D2 are formed on silica gel in a molar ratio of 1.3:1, respectively. The dimers are reportedly formed in a 1.2:1 ratio during the photolysis of *trans*-stilbene in water [18], whereas dimerization in benzene is only observed at concentrations above 0.5 M [20]. Dimerization of *trans*-stilbene on the silica gel surface is facilitated by the formation of ground-state pairs at the time when the silica



Scheme 1.

gel surface is loaded. The pairs exhibit an excimer-like emission as shown in Fig. 1. Fig. 1 shows the fluorescence of *trans*-stilbene monomer as curve C ($\lambda_{EX} = 295$ nm) and the corresponding excitation spectrum as curve A ($\lambda_{EM} = 360$ nm). When the sample was excited slightly to the red of the monomer, curve D ($\lambda_{EX} = 350$ nm), the excimer-like emission of the ground-state pairs is observed. Monitoring the emission further to the red generates excitation spectrum, curve B ($\lambda_{EM} = 400$ nm), having a greater contribution from the ground-state pairs. The emission attributed to the ground-state pair is red-shifted relative to the anticipated position of *cis*-stilbene emission based on previous measurements in solution [21]. *Cis*-stilbene emission on silica gel at room temperature appears to have a significant contribution from the *trans* isomer, as reported for solution spectra [21].

Oxidation is a minor pathway of decomposition from direct photolysis of *trans*-stilbene on silica gel. The oxidation product, benzaldheyde, was not reported from the photolysis of stilbene in water [18]. Photolysis of *trans*-stilbene on silica gel in the presence of co-adsorbed singlet oxygen trap, 2,5-dimethylfuran, does not result in the formation of benzaldehyde, thus suggesting a Type II oxidation mechanism. Sensitized production of singlet molecular oxygen on silica gel by excitation of methylene blue (650 nm, 28 h) in the presence of co-adsorbed *trans*-stilbene (2.5×10^{-5} mol g^{-1}) resulted in the formation of benzaldehyde (18 mol%) and a trace of phenanthrene (<1 mol%). The stilbene was recovered as both *trans* (31 mol%) and *cis* (50 mol%) isomers. Only a small amount of *trans* to *cis* isomerization was observed in the dark over a 72 h period.

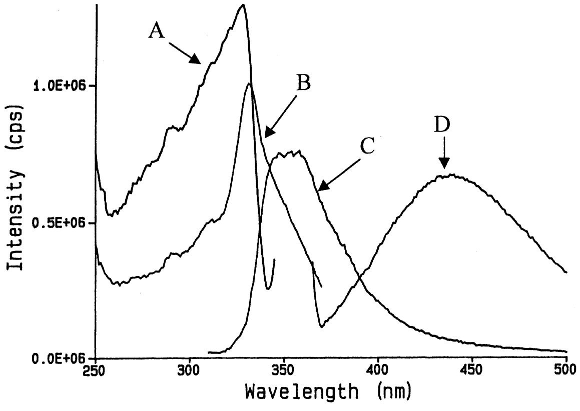
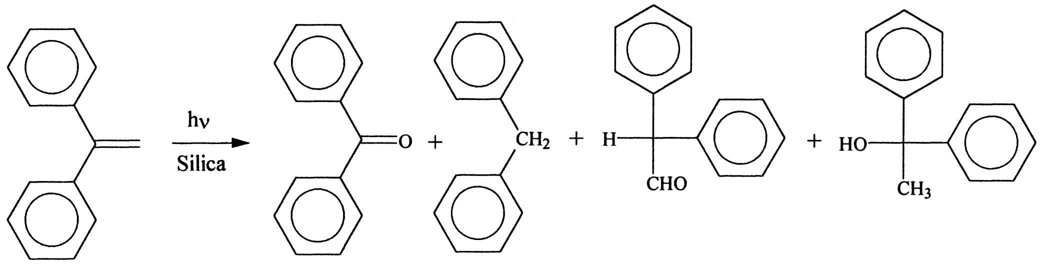


Fig. 1. Fluorescence of *trans*-stilbene on silica gel under He. Excitation and emission wavelenghts are: curve A ($\lambda_{EM} = 360$ nm), curve B ($\lambda_{EM} = 400$ nm), curve C ($\lambda_{EX} = 295$ nm) and curve D ($\lambda_{EX} = 350$ nm).

Photolysis (650 nm) of co-adsorbed methylene blue and either *cis*- or *trans*-stilbene, in the absence of oxygen, led to stilbene isomerization yielding a photostationary isomeric mix containing 55 ± 1% *cis*-stilbene. The photostationary ratio determined here is not significantly different from the photostationary 60% *cis*-stilbene reported for direct photolysis in a silica gel–cyclohexane slurry [22]. The mechanism of stilbene isomerization at the silica gel–air interface probably does not occur through triplet sensitization of *trans*-stilbene ($E_T = 60.9$ kcal mol^{-1}) by methylene blue ($E_T = 40.4$ kcal mol^{-1}) [23]. Although stilbene isomerization in solution has been demonstrated for other low-energy triplet sensitizers [24], in our laboratory, *trans*-stilbene is not isomerized in solution by methylene blue. We observe a hypsochromic shift in the methylene blue groundstate absorption by 3.7 kcal mol^{-1} on silica; however, a similar shift in the lowest triplet state of methylene blue would not be sufficient to lead to sensitized stilbene isomerization on the silica surface. Additionally, *trans*-stilbene isomerizes on silica upon excitation of methylene blue in the presence of molecular oxygen, a competitive quencher of the triplet state of methylene blue. An electron transfer mechanism is proposed to account for the isomerization. It has been demonstrated that the radical cation of *cis*-stilbene is thermally stable to isomerization in solution and on silica surfaces [25,26]; however, the radical cation ($\lambda_{max} = 510$ nm) does undergo photochemical isomerization in solution [27]. Light from our broad-band 650 nm source may be responsible for the observed isomerization in our studies.

3.2. *1,1-Diphenylethylene*

Photolysis of 1,1-diphenylethylene (300 nm, 4 h, 100% conversion) adsorbed at a silica gel–air interface (2.5×10^{-5} mol g^{-1}) led to the formation of benzophenone (85 mol%), diphenylmethane (11.4 mol%), diphenylacetaldehyde (<2 mol%), and 1,1-diphenylethanol (<2 mol%). The product structures are shown in Scheme 2. The yields of benzophenone, diphenylacetaldehyde and 1,1-diphenylethanol



Scheme 2.

are in good agreement with those reported earlier; however, diphenylmethane formation was not previously reported [1]. An identical sample of 1,1-diphenylethylene on silica gel was kept in the dark for 63 h and gave no diphenylmethane formation. Otherwise, the dark sample gave lesser yields of the same products observed under 300 nm photolysis. The major product in the dark reaction was 1,1-diphenylethanol.

Photolysis (300 nm, 3 h) of 1,1-diphenylethylene on silica gel in the presence of 2,5-dimethylfuran gave the same products as observed in the absence of the singlet oxygen trap. Additionally, the photochemical degradation rate was not decreased by the presence of the furan. Photolysis (650 nm, 7 h) of 1,1-diphenylethylene in the presence of co-adsorbed methylene blue gave minimal conversion to produce three oxidation products. Benzophenone was the major oxidation product formed, along with traces of 1,1-diphenylethanol and diphenylacetaldehyde. These results indicate that Type II oxidation of 1,1-diphenylethylene on silica gel is possible; however, the principal oxidation mechanism of direct photolysis on silica gel is Type I, as previously proposed [1].

The results reported here demonstrate different mechanisms of oxidation for *trans*-stilbene (primarily Type II) and 1,1-diphenylethylene (primarily Type I) on silica gel. These two substituted ethylenes have nearly identical ground-state oxidation potentials, 1.52 eV for 1,1-diphenylethylene and 1.51 eV for *trans*-stilbene (versus SCE, 0.5 M NaOAc in both cases) [28]. Insight into the origin of the mechanistic difference comes from the relative reactivity with singlet molecular oxygen. The singlet oxygen bimolecular quenching rates for 1,1-diphenylethylene, *trans*-stilbene and *cis*-stilbene in methylene chloride and at a silica gel–air interface are given in Table 1. The bimolecular rate constant measured for quenching singlet oxygen by *cis*-stilbene in methylene chloride ($3.66 \times 10^5\,M^{-1}\,s^{-1}$) is considerably larger than previously reported ($3.0 \times 10^3\,M^{-1}\,s^{-1}$ in CCl_4) [29]. In the absence of quencher, we obtain a singlet molecular oxygen lifetime of 73 μs in methylene chloride, in good agreement with reported values [30]. The pseudo first order rates for quenching of singlet oxygen at the silica gel–air interface are plotted in Fig. 2. The observed rates of singlet oxygen quenching on silica gel are not linear functions of quencher loading. We attribute the non-linear kinetics to molecular pairing of the quencher on the silica gel surface. One member of the quencher pair may be screening the other from interaction with singlet oxygen, or both members of the pair may fail to quench singlet oxygen. A simple model of the process is given by the following reactions, where M is the quencher monomer and GSP is the quencher ground-state pairs or aggregates on the surface.

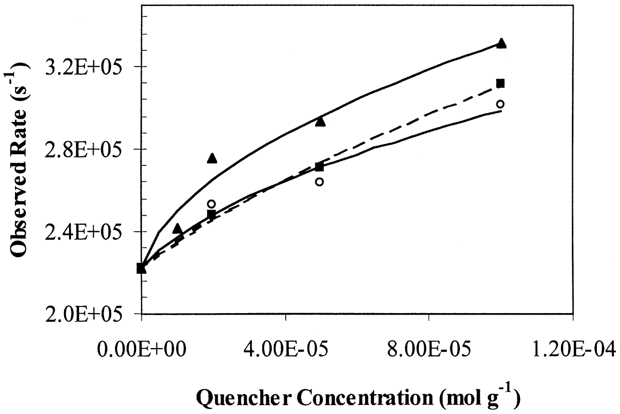


Fig. 2. Singlet molecular oxygen lifetimes at a silica gel–air interface as a function of 1,1-diphenylethylene, *trans*-stilbene and *cis*-stilbene surface loadings.

$$^1O_2 \rightarrow {}^3O_2 \qquad (k_1) \tag{1}$$

$$^1O_2 + M \rightarrow \text{Product} \qquad (k_2) \tag{2}$$

Table 1
Rates of singlet molecular oxygen quenching in solution and at a silica gel–air interface by 1,1-diphenylethylene *trans*-stilbene and *cis*-stilbene. The ground-state pair association constants, *K*, are given in parentheses for the silica gel–air data. See text for an explanation

Solvent/reaction medium	1,1-Diphenylethylene	*Trans*-stilbene	*Cis*-stilbene
CH_2Cl_2 ($M^{-1}\,s^{-1}$)	1.3×10^4	2.5×10^4	3.7×10^5
SiO_2/air ($g\,mol^{-1}\,s^{-1}$)	1.3×10^9 (7.3×10^3)	2.0×10^9 (4.2×10^4)	5.8×10^9 (2.3×10^5)

$$2\mathrm{M} \leftrightharpoons \mathrm{GSP} \qquad (K) \tag{3}$$

Eq. (1) represents the decay of singlet oxygen in the absence of the quencher, and Eq. (2) represents the sum of physical and chemical quenching by M on the surface.

Nonlinearity in Fig. 2 results from the equilibrium in Eq. (3). The equilibrium leading to ground-state pairs or aggregates is established when the quencher is loaded onto silica gel, as previously observed for multiple PAH on silica gel [2–14]. Restricted surface diffusion of the quencher will result in very little shift in the equilibrium on the time scale of singlet oxygen decay. Solving the simple set of Eqs. (1)–(3) gives the observed singlet oxygen decay rate, k_{obs}, as a function of the total quencher loaded onto the surface, M_{T}, as shown in Eq. (4).

$$k_{\mathrm{obs}} = \frac{k_1 + k_2}{2K}[(1 + 4KM_{\mathrm{T}})^{1/2} - 1] \tag{4}$$

The value of k_1 ($2.2 \times 10^5\,\mathrm{s}^{-1}$) was determined for singlet oxygen on silica gel in the absence of quencher. Values of k_2 and K (given in parentheses) in Table 1 were determined from fitting Eq. (4) to the observed rate constants. The lines shown in Fig. 2 are the best fit of Eq. (4) through the data.

The rate constants in Table 1 show that quenching of singlet molecular oxygen in solution is roughly 15–30 times faster for *cis*-stilbene than for *trans*-stilbene or 1,1-diphenylethylene. The Z isomers of aliphatic olefins are known to quench singlet molecular oxygen more efficiently than the E isomers [29]. Aliphatic olefins react with singlet molecular oxygen by an ene reaction and the rate difference is generally smaller than observed here. The data in Table 1 shows that the relative difference in rates of singlet oxygen quenching by *cis*- and *trans*-stilbene is mitigated somewhat at the silica–air interface; however, quenching by *cis*-stilbene remains faster by a factor of 3–4. The quenching data offers a possible explanation to the observed difference in oxidation mechanisms for 1,1-diphenylethylene and *trans*-stilbene on silica gel. *Trans*-stilbene isomerizes to *cis*-stilbene on silica gel, either by direct photolysis or sensitization with methylene blue, and the *cis* isomer reacts more readily with singlet oxygen. The structure of 1,1-diphenylethylene is invariant upon positional interchange of the two ethylenic hydrogens and reactivity with singlet oxygen is slower than *cis*-stilbene, allowing for a slower electron transfer oxidation pathway to compete. In addition, once *trans*-stilbene has converted to the *cis* isomer, intramolecular cyclization to form phenanthrene becomes the dominant reaction pathway at low surface coverage.

From the value of K for *trans*-stilbene (4.2×10^4, Table 1), the simple model allows us to calculate the percent of M_{T} forming ground-state pairs or aggregates (GSP, Eq. (3)). At a surface loading of $2.5\times10^{-5}\,\mathrm{mol\,g^{-1}}$, 39% of the molecules would form ground-state pairs. Assuming that photochemical dimer comes from a subset of those ground-state pairs, only 74% of the pairs lead to dimer formation. At a surface loading of $1.0 \times 10^{-4}\,\mathrm{mol\,g^{-1}}$, 62% of the molecules would form ground-state pairs; however, only 37% of those pairs lead to photochemical dimer formation. The decrease in the fraction of “pairs” leading to dimer at higher coverage is interpreted as resulting from the formation of crystals on the surface. Crystalline *trans*-stilbene is reported not to undergo photochemical dimerization [18]. We observe the same result under our reaction conditions.

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