

THE USE OF DIFFUSE REFLECTANCE LASER FLASH PHOTOLYSIS
TO STUDY PRIMARY PHOTOPROCESSES IN ANISOTROPIC MEDIA

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Summary. The technique of Diffuse Reflectance Laser Flash Photolysis is outlined in terms of the general principles for detecting transient absorption within an optically-dense medium, and for interpreting from diffusely reflected analysing light the kinetic and spectral properties of the transient species. The application of data obtained by the technique to further the understanding of photoprocesses in anisotropic media is discussed using examples of dyed cotton fabrics, functionalised polymers, molecules adsorbed as fractions of a monolayer on oxide substrates, and ketones intercalated in the channels of 'silicalite'.

Introduction

Flash photolysis is frequently applied to organic molecules to investigate primary photoprocesses from which a greater understanding of photochemical reactions can be obtained. Numerous publications deal with the general methodology of flash photolysis¹⁻⁴, in which a pulse of exciting light is employed to generate transient species which are detected by absorption of light from an analysing source. The decay of the transient absorption at one particular wavelength can be followed ('kinetic spectrophotometry') or the absorption spectrum of the transient is recorded at given times after the exciting pulse ('flash spectroscopy'). Hitherto a basic requirement was that the sample must transmit a portion of the analysing light. This requirement is met when a given molecule is dissolved in a fluid solution or exists in the gas phase, but is not met if, for example, it is adsorbed on an opaque or highly scattering substrate. It is clear that transmission flash photolysis is ideal for studying a transparent isotropic medium, but is not directly applicable where the optical density is so high that no detectable light is transmitted. We have developed and extended nanosecond laser flash photolysis for direct application to highly-scattering substances, and in this article we will discuss both the technique and its application to optically dense anisotropic media.

A given opaque sample has a certain reflectance R_A^0 at a wavelength λ . R_A^0 is defined as J_A^0/I_A , where J_A^0 and I_A are the levels of reflected and incident light, respectively, at the sample surface. If excitation produces a transient that absorbs a fraction of light of wavelength λ , J_A^0 will be decreased to some value J_A^1 , and the reflectance is reduced to R_A^1 . As the transient decays J_A^1 will increase, and if complete decay takes place and no permanent colour change or other damage has occurred, the original level of J_A^0 will be attained. Thus the decay of the transient can be followed by measuring the change in the level of reflected analysing light as a function of time. Note that two kinds of reflection can take place from the sample; (i) the specular, regular surface, or mirror reflection, and (ii) the diffuse reflection. The essential difference between these two types is that in the former the incident light is almost entirely reflected by the surface of the sample and the reflected beam emerges with the same angle to the normal as the incident beam, whereas for the latter reflection the incident beam penetrates into the sample before emerging. The diffuse reflection occurs randomly and is not directional. In "Diffuse Reflectance Laser Flash Photolysis" diffusely reflected monitoring light interrogates transient absorption in an analogous fashion to transmitted monitoring light in transmission flash photolysis. The interpretation of kinetic and spectral parameters contained in diffusely reflected monitoring light, and a selection of examples of the photophysics and photochemistry of some organic systems will now be discussed.

One approach to considering light diffusely reflected from a medium containing randomly distributed, uniformly absorbing and scattering particles involves two differential equations which describe the light flux in a thin slice of thickness dx at a depth x beneath the irradiated surface⁵:

$$dI = -(K+S)I dx + SJ dx \quad (1)$$

$$dJ = (K+S)J dx - SI dx \quad (2)$$

where I and J are the light fluxes away from and towards the surface, respectively. K and S are the absorption and scattering coefficients, respectively, of the sample. When the sample is so thick that the layer containing the light flux is backed by material into which no light penetrates, equations (1) and (2) can be solved to give the Kubelka-Munk equation which relates the observed reflectance R to K and S : viz.

$$\frac{(1-R)^2}{2R} = \frac{K}{S} \quad (3)$$

For a single substance $K = 2\epsilon C$, where $\epsilon =$ extinction coefficient and $C =$ concentration. If there is background absorption by the substrate then $K = K_B + 2\epsilon C$, where K_B is the absorption coefficient of the substrate. For a homogeneous conversion of ground state species to a transient, at any time after flashing $K = K_B + 2\epsilon_G(C_G - C_T) + 2\epsilon_T C_T$, where G represents the ground state absorber and T the transient. If S is constant then

$$\frac{(1-R')^2}{2R'} - \frac{(1-R)^2}{2R} = \frac{2(\epsilon_T - \epsilon_G)C_T}{S} \quad (4)$$

where R and R' are the reflectances observed before and after lasing, respectively. If the Kubelka-Munk approach is used for considering diffusely reflected light, equation (4) is a general one for relating the observed transient reflectance to the initial reflectance of the sample and the extinction coefficients of the ground state absorber and the transient species.

Generation of a uniform transient distribution by an exciting pulse occurs when a high percentage conversion of ground state to transient occurs, which would be expected in a dilute sample with a high ground state extinction coefficient at the exciting wavelength. It has been shown theoretically that increasing the intensity of excitation in such a sample causes an increase in the thickness of the layer containing a uniform transient distribution⁶. This is depicted in Fig. 1. In samples where a low percentage conversion of ground state to transient is expected, an exponentially falling-off

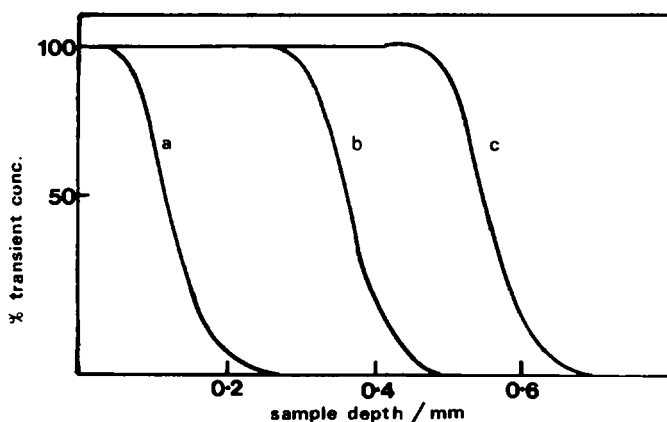


Fig.1. The migration of the transient concentration profile into a sample as a function of photon density of the exciting pulse - (a) 10^{-7} (b) 5×10^{-7} (c) 10^{-6} Einstein cm^{-2} . The curves are calculated for a concentration of ground state absorbers of 5×10^{-3} mol dm^{-3} , ϵ is 10^4 mol $^{-1}$ dm^3 at the exciting wavelength, and $S=100$ cm^{-1} .

concentration of transients is obtained and equation (4) does not apply. Calculations have shown that when less than 10 % of available monitoring light is absorbed, the change in reflectance is directly proportional to the transient concentration⁷. If the entire depth of sample is totally converted from the ground state, for example where a thin absorbing layer is adsorbed on to a scattering substrate, equation (4) is inappropriate and the observed reflectance is related to the absorption and scattering coefficients by:

$$R' = \frac{(1-\beta^2) \sinh(\alpha d)}{(1+\beta^2) \sinh(\alpha d) + 2\beta \cosh(\alpha d)} \quad (5)$$

where $\alpha = \sqrt{K'(K'+2S)}$, $\beta = \sqrt{K/(K'+2S)}$, and d is the sample thickness.

Kinetic analysis and spectral interpretation of transient absorption data obtained from diffuse reflectance laser flash photolysis are therefore dependent on the distribution of transients within the scattering medium. Samples such as organic microcrystals which have very high ground state concentrations are likely to possess an exponentially falling-off transient concentration and the change in transient reflectance (" ΔR ") can be taken as proportional to the transient concentration. First order kinetic analysis is performed by plotting $\ln(\Delta R)$ against time; second order processes would not normally be expected where a concentration gradient exists. The transient extinction coefficient will be proportional to ΔR .

In an optically-thick sample such as a monolayer of absorbing material adsorbed on an inert substrate, a uniform transient population may be produced. To obtain the first order rate constant $\ln\{[(1-R')^2/2R'] - [(1-R)^2/2R]\}$ should be plotted against time, and for second order analysis a plot of $1/\{[(1-R')^2/2R'] - [(1-R)^2/2R]\}$ versus time will yield $k_2 C_0$, where k_2 is the second order rate constant, and C_0 is the initial transient concentration. Provided that S is independent of wavelength, a plot of $[(1-R')^2/2R'] - [(1-R)^2/2R]$ against wavelength at several times after excitation will give the time-resolved transient difference spectrum. For all the spectra shown in this paper S is assumed to be independent of wavelength.

The geometric disposition of sample, exciting and analysing beams, and detector (e.g. a photomultiplier) is naturally different for a scattering sample to that employed in transmission flash photolysis. It is crucial that the area of sample receiving analysing

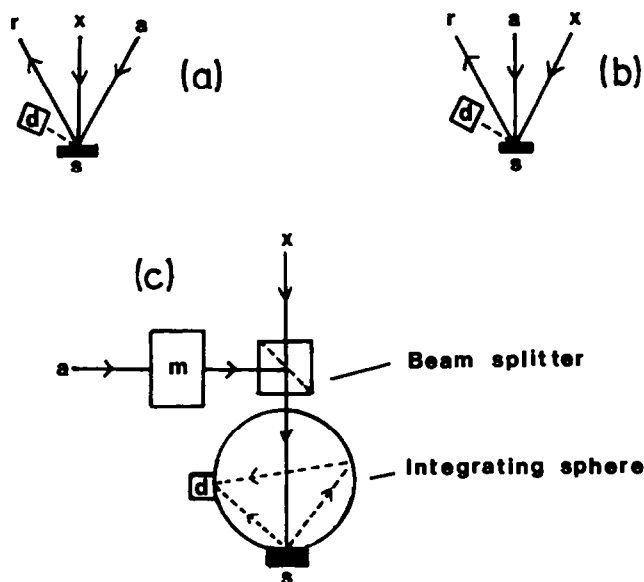


Fig.2. Three geometric dispositions of sample 's', exciting 'x' and analysing 'a' beam and detector 'd'. (a): excitation normal to the sample, with analysing light incident at an angle of about 45° . (b): as for (a) but exciting and analysing beams reverse positions. In (a) and (b) 'r' is the specular reflection of the analysing and exciting beams, respectively, and the detector 'd' includes a monochromator. (c): an arrangement including an integrating sphere where excitation and analysis is performed coincidentally. Here the detector 'd' is a photomultiplier alone and monochromator 'm' is placed before the sample.

light has been wholly excited, and that no specular reflection of the analysing beam is detected since it contains little or no transient information. It is equally important that neither the diffuse nor the specular reflection of the exciting laser beam enters the detector, since spurious signals due to photomultiplier fatigue and recovery can result. Fig.2 shows geometric arrangements for production and detection of a transient absorption in an opaque sample, including a system which contains an integrating sphere. However, when the sphere was employed, no improvement in signal to noise ratio was obtained when the monochromator-photomultiplier was placed at the sphere exit port, since only a very small amount of light from the sphere can be directed through the monochromator to the photomultiplier. If the monochromator is positioned before the sphere entrance and an 'end-on' photomultiplier placed at the exit, as in Fig. 2(c), this problem can be overcome; but any luminescence of the sample will usually overload the photomultiplier, and this arrangement is therefore only viable for non-emitting samples. Our experience has shown that little or no improvement can be gained from using an integrating sphere, and our preferred geometry is the simpler arrangement of Fig.2(a) or 2(b).

Results

The use of nanosecond diffuse reflectance laser flash photolysis was first reported for microcrystalline benzil⁸ and benzophenone⁹, where the triplet-triplet absorption was detected for each material. We later demonstrated the versatility of the technique by applying it to inorganic phosphor powders¹⁰, doped semiconductors¹¹, as well as to the heterogeneous anisotropic substances which are to be discussed below. It is gratifying to note that other workers have used the diffuse reflectance method to study, for example, the photophysical behaviour of pyrene on alumina¹² and of ketone molecules adsorbed on silica^{13,14}.

(i) Dyestuffs incorporated into woven cotton fabric

Plain weave, non-fluorescent cotton fabric provides a medium into which dyestuffs can be readily incorporated from a fluid solution. Hitherto the photophysical properties of a dyed fabric could only be directly obtained from luminescence measurements and model studies or transient absorption by the dye would need to be carried out in a dilute fluid solution by transmission flash photolysis. By means of the diffuse reflectance technique, transient absorption with cotton fabric dyed with aluminium sulphonated phthalocyanine (ALPCS)¹⁵ and halogenated derivatives of xanthene dyes (eosin and erythrosin) have been studied directly. Fig.3 shows the time-resolved transient difference spectrum for ALPCS dyed cotton fabric. From the spectral features the transient can be assigned as the triplet-triplet absorption of ALPCS¹⁶, and its decay follows an exponential rate law for which k_1 is in the region of 10^3 s^{-1} . This

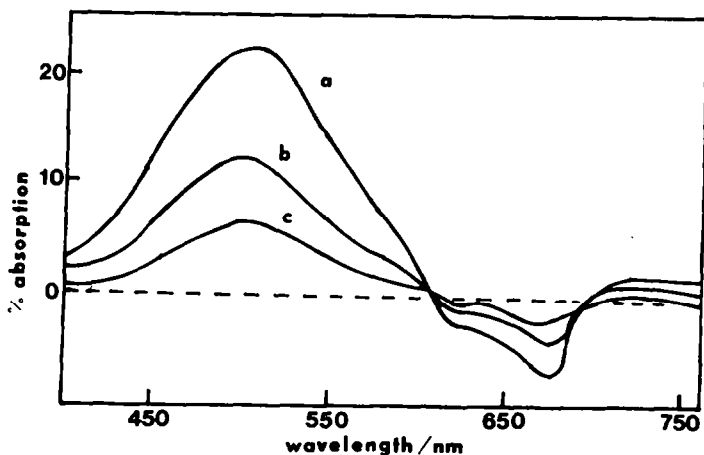


Fig.3. Time-resolved transient difference spectrum of nitrogen-purged, dry cotton fabric dyed with ALPCS following excitation at 354 nm, (pulse duration = 15 ns, pulse intensity = 40 mJ/pulse). (a) 0 μs (b) 50 μs (c) 100 μs after laser flash.

constant holds whether the fabric is dry or water-saturated in the presence of a positive pressure of nitrogen, and in dry fabric under a positive pressure of oxygen hardly any quenching of the transient is noted. However, when both water and oxygen are present, quenching does take place. The decay in the presence of water and oxygen does not obey pseudo-first order kinetics and there is evidence for the formation of a permanent product. Analogous behaviour occurs for cotton dyed with eosin and erythrosin; for these fabrics the major transient component is the triplet state, assigned on the basis of spectra reported for a dilute fluid solution^{17,18}, although under certain conditions for eosin there is also spectral evidence for the presence of the semi-reduced form of the dye.

(ii) Polymer-bound Photosensitisers

The successful immobilisation of rose bengal to a polymer backbone by Schaap et al^{19,20} has considerably extended the important role of this compound as a photosensitiser. However, a full investigation of the primary photophysical processes within polymer-bound rose bengal is not possible by transmission flash photolysis, due to the opaque nature of the sensitizer. We have carried out a diffuse reflectance laser flash photolysis study of "Sensitox I" (rose bengal bound to chloromethylated styrene-divinyl copolymer beads¹⁹) and "Sensitox II" (rose bengal bound to the polymer formed from copolymerisation of chloromethylstyrene and the monomethylacrylate ester of ethylene glycol²⁰), and compared and contrasted the photophysical behaviour of rose bengal in a variety of other heterogeneous environments²¹. Fig.4 gives the time-resolved transient absorption spectra for Sensitox I and Sensitox II. The structure of these two spectra are not dissimilar, but the shoulder at 680 nm for Sensitox I becomes a peak in the case of Sensitox II. Note also that the different polymer base results in different decay times, with the transient absorption in Sensitox I decaying approximately ten times more quickly than the transient from Sensitox II. For both materials the transient is assigned as the triplet-triplet absorption, and laser-induced emission maximising at 765 nm was also observed. The emission decay in each sample has the same first and second half life as the corresponding absorption decay, within experimental error; from both this observation and spectral information the emission is assigned as phosphorescence.

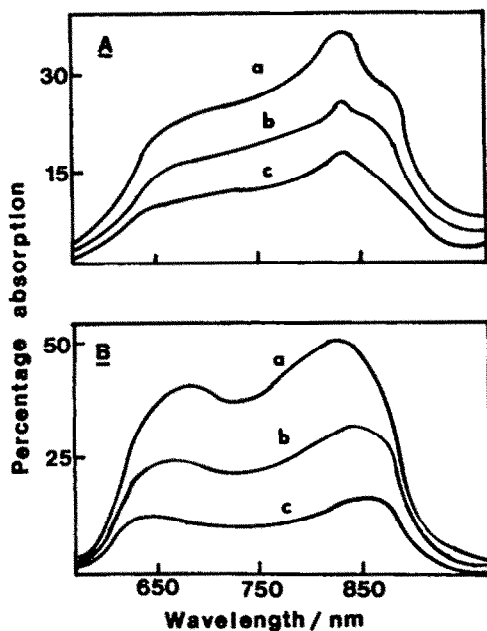


Fig.4A. Transient absorption spectrum of Sensitox I (a) 0 μ s (b) 1 μ s (c) 4 μ s after laser flash.

Fig.4B. Transient absorption spectrum of Sensitox II (a) 0 μ s (b) 25 μ s (c) 100 μ s after laser flash. For both samples, excitation wavelength = 354 nm, pulse width = 15 ns, intensity = 40 mJ/pulse.

Decay by a mixture of first and second order processes can be expressed as:-

$$-\frac{dC}{dt} = k_1C + k_2C^2 \quad (6)$$

The integrated form of this equation may be written as:-

$$\ln \left[\frac{C_0 + \frac{k_2 C_0}{k_1}}{C} \right] = k_1 t + \ln \left[\frac{1 + \frac{k_2 C_0}{k_1}}{1} \right] \quad (7)$$

From analysis of absorption and phosphorescence decays according to equation (7) a fit to the data was found²¹. Decay traces obtained from rose bengal in other heterogeneous environments can also be fitted by equation (7), and for samples where a faster triplet decay obtains, an additional laser-induced emission is observed which centres on 610 nm and which decays more rapidly than the phosphorescence. In the 600 nm region rose bengal is expected to exhibit fluorescence, but the decay observed at 610 nm is too slow to be prompt fluorescence. It is more likely that delayed fluorescence is occurring, and this implies that triplet-triplet interaction to re-populate the singlet is more predominant in some heterogeneous environments than in others. This kind of information may help in evaluating the efficiency of a triplet sensitizer, since reversal of the triplet to the singlet can compete with the triplet sensitisation process.

Benzophenone is a well-established triplet sensitizer in organic

photochemistry, and has recently been synthesized and used in polymeric form (as benzoylated polystyrene) to bring about photochemical reactions such as the (2+2) photocycloaddition of cyclohexene to maleic anhydride, the E-Z isomerisation of (E)-1,3-pentadiene and methyl(E)-2,2-dimethyl-3,5,-hexadienoate, and the photooxidation of secondary alcohols to ketones²². Polymeric benzophenone exists in the form of insoluble beads in which we have

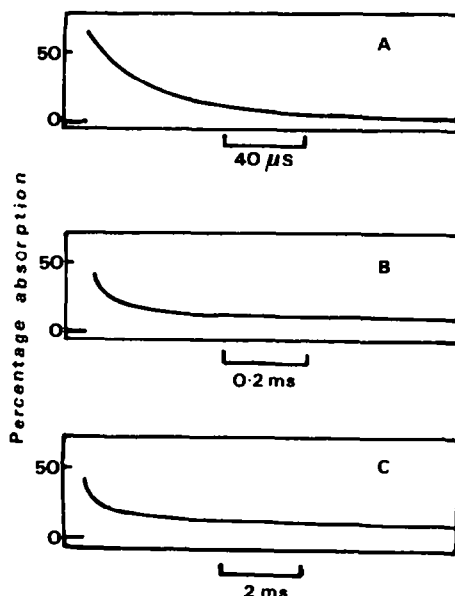


Fig.5 Decay of the transient absorption in polymeric benzophenone (benzoylated polystyrene) monitored at 530 nm following excitation at 354 nm (pulse width = 15 ns, pulse intensity = 20 mJ/pulse).

A "P-1", where 1% of available phenyl groups in polystyrene-2% divinyl benzene co-polymer is benzoylated.

B "P-29" - 29% benzoylation.

C "P-50" - 50% benzoylation.

studied transient absorption using diffuse reflectance laser flash photolysis. Fig.5 shows that the absorption decay occurs more rapidly as the percentage benzoylation of available phenyl groups in polystyrene-2% divinylbenzene copolymer beads is increased. Studies of laser-induced emission show hardly any change in luminescence decay time for different percentage benzoylation of the polymer base. The emission is assigned as phosphorescence from its spectral distribution, but the transient absorption arises from a species - perhaps a radical formed from the triplet state - which appears to be quenched when a higher concentration of phenyl groups and/or abstractable hydrogens surround the benzophenone moiety. The experiments with benzoylated polystyrene are still in their initial

stages, but from the data obtained so far it is evident that interactions between an immobilised chromophore and the substrate or an anchored quencher can be investigated in optically dense materials by diffuse reflectance laser flash photolysis.

(iii) Hydrocarbons adsorbed on to alumina

Samples of naphthalene, biphenyl, triphenylene, pyrene and phenanthrene chemisorbed on γ -alumina were the first materials to be subjected to diffuse reflectance flash photolysis²³. From the spectrum obtained for each sample the transient was assigned as the triplet state, although the use of a millisecond flashlamp as the excitation source precluded any study of shorter-lived transients. It was from these results that the impetus was derived to fully develop and exploit nanosecond diffuse reflectance laser flash photolysis.

(iv) Acridine adsorbed on alumina and silica

The interaction between a π -electron system and the electron-deficient sites at the alumina or silica surface can be probed by studying the properties of transients using time-resolved diffuse reflectance spectroscopy. For acridine on alumina the transient absorption maximises at 485 nm and is assigned as arising from complexes between acridine and Lewis acid sites in alumina. Evidence for insignificant interaction between adjacent acridine molecules

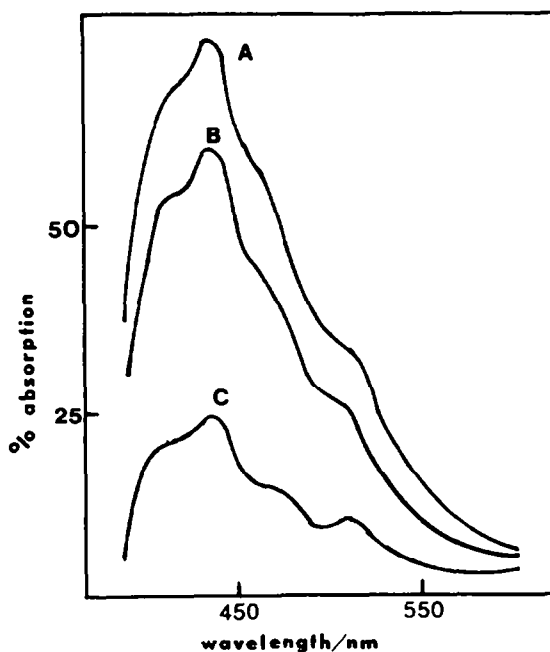


Fig.6 Triplet-triplet absorption spectrum of acridine adsorbed on silica (A) 0.5 ms (B) 1.0 ms (C) 4.0 ms after excitation at 354 nm (pulse duration = 15 ns, pulse intensity = 40 mJ/pulse).

comes from the observation that the decay of the transient absorption is virtually independent of surface coverage. This is in sharp contrast to where silica is the adsorbent. In this case the transient absorption maximises at 440 nm - see Fig.6. - and is assigned as a triplet-triplet absorption. At low coverages of the silica surface the triplet has a lifetime of 30 ms and its decay follows an exponential rate law. When the surface coverage is raised the order changes from first to second; this is explained by triplet-triplet annihilation which results from the mobility of adsorbed acridine on the silica surface or from the formation of aggregates even at low surface coverages²⁴.

(v) Ketones intercalated in the channels of 'silicalite'

Silicalite is a hydrophobic zeolite with a zig-zag system of circular and elliptical channels which have a free cross section of about 5 Å. Molecules which can be included in the channels should be expected to exhibit different properties to when existing in an isotropic medium, e.g. by virtue of restricted molecular motions, and by having a variety of potential adsorption sites. These properties have been probed for different ketones intercalated in the channels of silicalite²⁵. Fig.7 depicts the time resolved spectrum for xanthone on silicalite. This spectrum arises from the triplet state of xanthone²⁶, for which λ_{\max} is known to be sensitive to the polarity of the environment. We observe λ_{\max} at 605 nm, which implies that the environment offered by silicalite is rather polar. Restricted molecular motion within the silicalite channels is demonstrated with acetophenone, butyrophenone, and valerophenone. In each of these

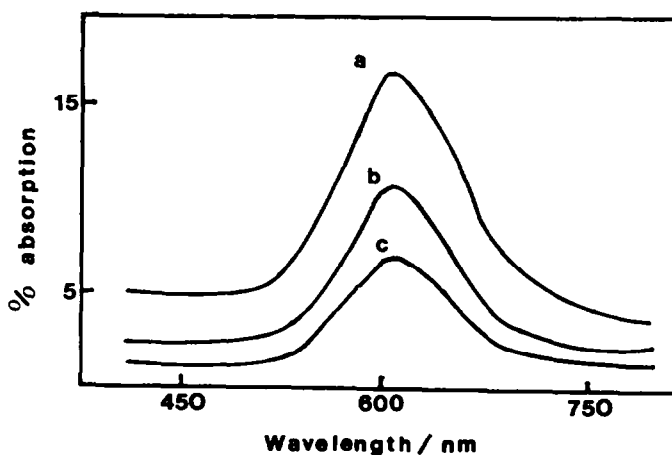


Fig.7. Triplet-triplet absorption spectrum of xanthone on silicalite (a) 0 μ s (b) 25 μ s (c) 100 μ s after laser flash. λ_{exc} = 354 nm, pulse duration = 15 ns, pulse intensity = 30 mJ/pulse.

samples a transient absorption assignable to the triplet benzoyl chromophore is obtained following excitation at 266 nm. The lifetime of the triplet increases in the order acetophenone > butyrophenone > valerophenone, but it is substantially longer than when the ketones are in a dilute solution, indicating restricted molecular mobility within the zeolite channel structure. The trend of the lifetimes does parallel solution behaviour which suggests that the Norrish Type II hydrogen abstraction can occur in the cases of butyrophenone and valerophenone, but at a considerably reduced rate.

Experimental

The apparatus to carry out diffuse reflectance laser flash photolysis is shown schematically in Fig.8. A harmonic of an Nd-YAG pulsed laser (J.K. Lasers Ltd) acts as excitation source - 3 harmonics are available: 532 nm, with a maximum intensity of 200 mJ/pulse; 354 nm (50 mJ/pulse); and 266 nm (10 mJ/pulse). The analysing source is

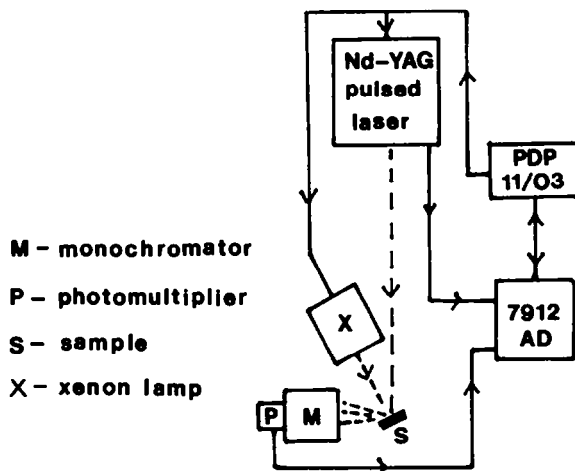


Fig.8 Schematic diagram of apparatus for diffuse reflectance laser flash photolysis.

the pulsed output of a 250 W Xenon lamp (pulse width = 0.5 ms) (Applied Photophysics Ltd), and an R928 Multiplier (Hamamatsu Ltd) is the detector. The photomultiplier signal is led to a Tektronix 7912AD programmable digitizer for transfer to a PDP 11/03 minicomputer (Digital Equipment Ltd) for analysis. The minicomputer is in overall control of the timing sequences and the operation of the apparatus, namely the laser firing, pulsing of the arc lamp, shutter control, reading of digitized data and resetting instruments for the next run. The digitizer time-base is triggered from the Q-switching unit of the YAG laser.

Sample preparation is as follows:

(1) Dyed cotton fabrics

A piece of cotton fabric is left soaking in an aqueous solution of the dye for 15 hours followed by drying in an oven at 80°C for 2 hours. The loading of the cotton is in the order of a few micrograms of dye per gram of fabric.

(2) Functionalised polymers

Sensitox I and Sensitox II (Hydron Laboratories Inc) were a gift from Dr A.P. Schaap, and were used as received. Benzoylated

polystyrene is made by Friedal-Crafts benzoylation of 2 β -divinylbenzene using benzoyl chloride in carbon disulphide in the presence of AlCl₃ (22).

(3) Materials adsorbed on alumina and silica

The adsorbents were outgassed under a pressure of 10⁻⁶ mbar at 100°C or 300°C for 2 to 5 days to remove oxygen and physisorbed water, and the adsorbates introduced to the adsorbent from high-vacuum and left for 2-5 days to complete the adsorption process.

(4) Ketones on silicalite

Silicalite (Union Carbide) was heated at 500°C for 24 hours prior to use. A solution of the ketone in 2,2,4-trimethylpentane (isooctane) was stirred with silicalite for several hours, followed by washing and drying²⁷. The size of the solvent molecule ensures that it cannot be included in the channels of silicalite.

Samples of (1), (2) and (4) were investigated in a quartz cuvette, and deoxygenated by flowing N₂ in/around the sample, if necessary, and samples of (3) were held in a sealed quartz ampoule.

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