Femtoscience: Exploring the Ultrafast Dynamics of Molecular Processes

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Abstract

In the course of photo and radiation chemical processes, reactants are converted into products with the possibility of various transition states and transient intermediates on the reaction pathway. The properties of these transient intermediate species are central to the reaction and determine its rate, selectivity and the course of the reaction. Lifetimes of these intermediates vary from a few tens of a femtosecond to a few picoseconds. Development of high power femtosecond lasers has made possible investigation of these processes in real time. Ultrfast transient absorption spectroscopic techniques developed in our laboratory have enabled us to investigate the intra and intermolecular processes undergone by the excited states and the other intermediate species produced following optical excitation of a solute molecule in condensed phase with femtosecond time resolution. In this talk, we discuss briefly the results of our studies on the excited state dynamics and photophysical and photochemical processes in some model molecules in solution.

Introduction

Femtoscience is the branch of science, which uses the femtosecond lasers as tools. This branch of science has been developed only recently after the invention of the high power femtosecond laser systems. Now, the first question is: what is a femtosecond? Technically, 1 femtosecond (fs) = 10^{-15} s. However, for those, who have the difficulties to visualize the shortness of this time scale, to say non-technically, if light can travel from here to the moon in 1 s, in 50 fs it can’t even travel the width of a human hair. The next question is: What femtosecond lasers can deliver? Combination of ‘mode-locking’ pulse generation and ‘Chirped Pulse Amplification’ techniques have produced the shortest pulse of 0.65 fs duration, and the biggest pulse of 10^{15} W or 10^{22} W/cm^2 of energy. These short duration lasers have made possible applications of femtosecond lasers in physics (for example, ultrafast spectroscopy of materials, non-linear optics, plasma physics and particle accelerators, are among a few of many applications), biology, surgery, material processing, imaging (such as multiphoton confocal imaging, optical coherence tomography, T-ray imaging etc.) and in many other fields of science and engineering.

However, our interest in femtosecond lasers has been in investigation of molecular processes with femtosecond time resolution. Exploring phenomena on an ever-shorter time scale in the race against time, femtosecond time resolution is the ultimate achievement for studies of fundamental dynamics of the chemical bond. This field is known as ‘femtochemistry’. Femtochemistry has become a well established field of science and its importance has been recognized by awarding the 1999 Nobel Prize to Prof. Ahmed Zewail of the California Institute of Technology, USA, who has pioneered this area of research. The importance of femtochemistry is evident from his own recognition: “....knowledge of the mechanisms of chemical reactions is also important for our ability to control the reactions. A desired chemical reaction is often accompanied by a series of unwanted,
competing reactions that lead to a mixture of products and hence the need for separation and cleansing. If the reaction can be controlled by initiating reactivity in selected bonds, this could be avoided......”. We should also recognize the important fact that many of the natural processes take place with awesome rapidity and often complete within a few hundred femtoseconds. For example, in vision process, photoisomerization of retinal chromophore is complete within 200 fs. In photosynthetic reaction centre, electron moves about 20 angstrom distance in 3 ps.

Femtochemistry is concerned with the very act of the molecular motions that brings about chemistry. With femtosecond lasers, it is now possible to record the snapshots of chemical reactions with sub-angstrom resolution. A chemical reaction can be reduced to a number of elementary reactions – breaking and forming of chemical bonds, energy transfer within and between molecules, electron transfer, proton or hydrogen atom transfer etc.

Understanding a chemical reaction at the atomic level not only requires a description of the reactants and products but also a description of the medium or surroundings, in which the reaction takes place, as well as understanding of the interactions between the reacting system and the surroundings. Now it is possible to characterize the ‘transition state’, which determines the course of a reaction. It is not only possible to observe the molecular processes in real time, but also possible to control them at one’s will.

Why femtosecond lasers have been considered as attractive tools for studying dynamics of chemical reactions? Of course, for high peak power apart from short duration! Each laser pulse of 50 fs duration and 1 milijoule energy dumps about $2 \times 10^{12}$ W or 2 teraWatt power onto the material. This generates high nonlinear effects in the material and makes possible to generate laser beams of different colors from the single source and these laser beams are perfectly synchronized in time. Hence, it is suitable for pump-probe experiment. During the last few decades, the development of ultrafast lasers has out-spaced the ability to create detectors with comparable time resolution. This mismatch has necessitated the development of techniques based on two or more pulses with a

![Fig.1 Pump - probe spectroscopy](image)
time resolution that is limited only by the duration of the laser pulses themselves. The most common of these methods capable of initiating a dynamic processes and interrogating its time evolution is known as pump - probe. This technique has been schematically presented in Figure 1.

The pump pulse creates a large population of excited states within short time duration and the probe pulse monitors the evolution of excited states with time delay between the two pulses. Probing can be achieved by monitoring absorption, laser induced fluorescence, ionization, photoelectron detection, resonance Raman etc. Changing the length of the path of either of them by introducing a high precession linear motion translation stage controls the delay between the two pulses. Remembering the fact that light travels only 1 micron distance in 3.3 fs, the linear motion stage should have the resolution of about 0.1 micron to achieve the delay of a few femtosecond between the two pulses. Figure 2 displays schematics of the laser system used in our laboratory for tunable pump - tunable probe transient absorption spectroscopy.

**Dynamics of Chemical Bond-Breaking Process in Solution**

Two important and fundamental questions in chemistry are: what is the time required to break a chemical bond in a photo-dissociation process and how the energy supplied to the molecule is partitioned between the fragments and the different kinds of motions of the fragments, such as kinetic, rotational and vibrational? Here, we discuss an example of the laser induced bond-breaking process – the photodissociation dynamics of mercuric iodide (HgI₂) in ethanol solution (eq. 1).

$$HgI_2 \xrightarrow{h\nu, 330nm} HgI^* \rightarrow HgI^0 + I$$

When HgI₂ is excited by 330 nm light to one of its dissociative higher electronic excited state (HgI*), the molecule is dissociated into the fragments, vibrationally hot HgI (HgI#) and I. In this process, the HgI# fragment is produced in the ground electronic state (X 2+), but with a large amount of excess vibrational energy. The iodine atom is produced in the ground electronic state. The excess vibrational energy in HgI# is subsequently dissipated to the surrounding solvent bath by external vibrational relaxation (EVR) or vibrational cooling process. Using the transient absorption technique we have been able to follow-up the dynamics of the bond breaking and energy flow process in the photodissociation reaction of HgI₂ in ethanol solution. In our experiment, we have used 330 nm pump and probe pulses in the wavelength range 330 to 700 nm, both the laser pulses having 50 fs duration. However, to be brief, we will discuss only the results...
obtained from the experiments using 330 nm pump and 660 nm probe pulses, which we will designate as 330/660 experiments, respectively, to explain the photodissociation and energy flow dynamics in the said process.

Let us make a simple calculation to illustrate the fact that the photodissociation process is really very fast and have a rough estimate of the time taken to break the I-HgI bond in the photodissociation process. The excitation energy due to a single photon of 330 nm wavelength is 33,303 cm\(^{-1}\) and the energy required to photodissociate the I-HgI bond is 21,000 cm\(^{-1}\). Hence the energy of recoil with which the fragments are moving away from each other is 9303 cm\(^{-1}\). Hence the velocity of the fragments with which they are getting separated is, \(v = \left(\frac{2E_{\text{recoil}}}{\mu}\right)^{1/2}\) Km/sec, where \(\mu\) is the reduced mass of the separated fragments. If the bond is said to have broken, when the fragments travel away by about 5Å or 5 \(\times\) 10\(^{-13}\) Km apart from each other, the bond dissociation time is calculated to be about 250 fs.

The potential energy surface (PES) diagram for I—HgI bond dissociation dynamics has been depicted in Figure 3. HgI\(_2\) molecule is excited by a 330 nm photon onto a dissociative surface. While traveling on this surface, the molecule undergoes dissociation process completed within 250 fs. The temporal dynamics of the transient absorption recorded in the 330/660 experiment due to photolysis of HgI\(_2\) in ethanol is shown in Figure 4. One of the two most important and interesting features of the temporal dynamics of HgI\(^6\), as shown in Figure 3b, is that the absorption signal takes about 250 fs time to attain maximum. This confirms the prediction that the bond-dissociation takes about 250 fs to be completed following excitation. Another feature is an oscillatory component of the signal superimposed on the decay of HgI\(^6\). The oscillatory component of the signal persists for a few hundred fs. The total decay dynamics of
HgI\# has been modeled with the response function,

\[ \text{OD}(t) = A \exp\left(-t/\tau_A\right) \cos(\omega t + \pi) + B \exp\left(-t/\tau_B\right) \]  

(2)

A and B are amplitude factors, \(\tau_A\) the damping time constant for the oscillation amplitude, \(\omega\) and \(\pi\) are frequency and phase of the oscillatory component and \(\tau_B\) is the population decay time of the transient species. The best-fit parameters obtained are: \(A/B = 1.71; \tau_A = 330 \pm 33\) fs; \(\omega/2\pi c = 89.5 \pm 15\) cm\(^{-1}\); \(\tau_B = 2.75 \pm 0.27\) ps. The temporal dynamics as shown in figure 3a is fitted very well to a single exponential function with lifetime of 2.72 \(\pm\) 0.2 ps. The oscillation of the transient signal shown in Figure 3b arises due to the fact that following photodissociation of HgI\(_2\), HgI\# is created in a coherent superposition of vibrational states, which is popularly known as ‘wave packet’. The wave packet then oscillates between the classical turning points of the potential energy surface of the ground electronic state of the HgI\# species causing the 660 nm absorption to be tuned in and out of resonance between the ground electronic state and one of its excited state (Figure 5). Using the gas phase PES of HgI, an estimate of the energy content and distribution in the diatomic photoproduct may be ascertained. The fundamental frequency and anharmonicity constant of the stretching vibration, the only mode of Hg—I, are 126 and 1.3 cm\(^{-1}\), respectively. Calculation shows that the beats correspond to a vibrational frequency of ca \(v = 15\) and that the observed signal originates mainly from a fragment which is born with ca 1700 cm\(^{-1}\) of excess (mean) vibrational energy. The remaining photolysis energy (ca 7600 cm\(^{-1}\)) must then be distributed among the translational and rotational motions of the fragments as well as the associated solvent motions. The frequency of the wave packet oscillations represents the mean frequency of a distribution of vibrational states determined by the initial conditions. Finally, the overall signal decay with lifetime \(\tau_B\), which is 2.7 ps, should be due to solvent induced vibrational relaxation (EVR) process undergone by HgI\#.

The phase of the wave packet has been found to be \(\pi\) radian. This result suggests that when Hg—I bond is compressed, the molecule does not absorb at 660 nm. If the Hg—I bond in the nascent product HgI is compressed, such as would occur in a dissociation reaction producing I and HgI from HgI\(_2\) through the asymmetric stretching coordinate (Figure 2), the resulting wave packet would not be detected until it would move to the attractive turning point of the ground state PES. Hence this would yield a phase shift of \(\pi\) for the absorption signal, as observed in Figure 5.

From this study, the following conclusions have been drawn: In photolysis of HgI\(_2\) by 330 nm laser pulses of 50 fs duration, HgI is produced in a coherent superposition of vibrational levels on the ground electronic state. The mean excess vibrational energy corresponds to about 1700 cm\(^{-1}\). This excess vibrational energy is dissipated to the solvent with an average lifetime of about 2.7 ps.

Ultrafast Dynamics of Intramolecular Processes in the Excited States of Some Model Molecules in Condensed Phase

Recently, supramolecules (i.e. large size organic molecules) are being used for various applications, such as in photonics (e.g.
molecular switches, optical data storage devices, optoelectronic devices, sensors, light emitting devices etc.), as well as in solar energy conversion. The photophysical processes responsible for the said actions may be photoinduced electron transfer, proton transfer and/or photoisomerization processes. However, following optical excitation, such a large size molecule in condensed phase, undergoes a numerous number of intra and intermolecular processes, including the desired one important for the said action. Among them two of them are the most important ones, in which the dynamics of interaction of the solute with the surrounding medium play a significant role in determining the fate of the excited state of the solute and hence the rates of photophysical and photochemical processes undergone by it. These processes are: energy exchange between the solute and the molecules of the medium via vibrational relaxation or, more specifically, vibrational cooling and solvation of the excited state via reorganization of the solvent molecules following intramolecular charge separation in solution. All these processes take place in ultrafast time scale and affect the relaxation dynamics of the electronically excited molecules in condensed phase. We have chosen a few simple model molecules to study the dynamics of these processes, which control the course of the reactions of the excited states in condensed phase.

**Conformational relaxation dynamics in the excited state of Benzil**

α-Diketones are structurally flexible with respect to the dihedral angle, θ, between the two halves of the molecule, due to low energy barrier. The geometry of the molecule is the result of a balance between the steric effects and resonance stabilization. The later is maximized in all-planar configurations. On the other hand, the steric effects, which arise due to interactions between the carbonyl oxygens and the substituents on the ring atoms (in the case of benzil, these are hydrogen atoms), particularly in ortho positions with respect to the carbonyl groups, never allow the planar configurations to be the most stable ones. Hence, the molecule attains different stable conformations in different electronic states making a compromise between these two factors. As a result, the excited electronic states have very different geometries than that in the ground state (Figure 6).

The time resolved absorption spectra of the transients produced due to photoexcitation of benzil in acetonitrile solution by 310 nm laser pulses are shown in Figure 7. In each solvent the time resolved spectra show continuous evolution up to about 200 ps. The transient absorption in the region 540 – 580 nm decays with the rates comparatively faster than that in the bluer region and hence, the width of the spectrum recorded at 200 ps becomes narrower than the one recorded at 0.5 ps in the red region. As a result the peaks of the transient absorption spectra recorded at 200 ps appear at about 527 nm. The decay characteristics of the transient absorption spectra recorded at 200 ps appear at about 527 nm. The decay characteristics of the transient absorption in subpicosecond time domain have been monitored at 570 nm, since the change in absorbance with time has been observed to be maximum at this wavelength. The decay profile along with the fit function obtained by iterative deconvolution analysis using an instrument response function of sech² functional form having ~0.5 ps FWHM are presented in Figure 7. The inset of this figure, which represents the early time (up to about 15 ps after photo-excitation) dynamics of the
transient species, clearly reveals the non-single-exponential nature of the decay dynamics. The decay dynamics of the transient species have been seen to fit reasonably well with a decay function, which is a sum of at least three exponential terms. Among the three lifetimes, the longest-lived component (about a few hundred picosecond), which represents the residual absorption in this time domain, could be assigned due to the S₁ state having trans planar conformation. The component with the shortest lifetime, τ₁, is assigned to the S₁ state of the cis-skewed form of benzil. The second component with the intermediate lifetime, τ₂, could be assigned to the S₁ state of the meta-stable intermediate conformer. Hence, the change in conformation from the cis-skewed form to the more stable trans planar form in the S₁ state of benzil is associated with crossings of two consecutive energy barriers (Figure 7). The first of the two barriers is due to change of conformation from the cis-skewed to the meta-stable intermediate conformer and the second one is due to conversion of the later to the trans planar form. The two-step relaxation process, each one being associated with finite energy barriers, leads to non-exponential absorption decay kinetics. The rate of crossing of a particular energy barrier during transformation of one form to the other can be correlated with the disappearance of the precursor species. Since the conformational relaxation process should involve the movement of bulky groups in the molecule, say, the rotational motion of the phenyl and the dicarbonyl groups as well as the translational motion of the two phenyl groups, the rate of barrier crossing dynamics is expected to depend strongly on the viscosity of the medium. The present study in different kinds of solvents has clearly revealed two facts. One is
the existence of at least one other intermediate between the two well-known conformers, cis-skewed and trans-planar, and the other is that the rates of conformational changes are dependent not only on the viscosities but also polarities of the solvents. In polar solvents, the barrier height of the cis(int) to the trans form is reduced significantly. Hence, it becomes evident that in addition to solute - solvent frictional interactions, the solute-solvent dielectric interactions also play an important role in activated barrier crossing dynamics for conformational changes in the S1 state of benzil. The most obvious effect arising due to polarity is 'static' and involves the solvation of the reactants, products and transition states. Static interactions modify the intramolecular potential energy surfaces and influence the barrier crossing flux. The solvent induced modification of the barrier height overrides the viscous reduction of the flux over the effective barrier. The situation becomes more complicated by the likelihood that the lowering of the barrier in polar solvents is due largely to dynamic solvent polarization, as the conformational changes proceed such that the barrier crossing is actually a time dependent quantity.

In conclusion, conformational relaxation dynamics of benzil in the S1 state from the cis-skewed to the trans planar form has been established to take place in two steps via the formation of a meta-stable intermediate conformer, the configuration of which probably has the configuration very near to the cis-planar form. The energy barrier for the first step, i.e. conversion of cis-skewed to the meta-stable intermediate is relatively smaller and barrier crossing dynamics is controlled by 'dynamic' or 'frictional' interactions with the solvent and the effect of 'static' interaction is not very significant. The energy barrier for the second step i.e. conversion of the meta-stable conformer to that of trans planar one is relatively higher and the barrier crossing dynamics is mainly controlled by 'static' rather than 'dynamic' interactions with the solvent.

**Ultrafast relaxation dynamics in the excited states of dimethylaminobenzophenone (DMABP)**

For several decades, steady state and time resolved phosphorescence spectroscopy as well as flash photolysis techniques have been used to study the photophysical and photochemical properties of the excited singlet and triplet states of benzophenone (BP) and its numerous kinds of derivatives by different groups. Most of these studies have been devoted to unravel the intricacies of the mechanisms of photoreduction reactions undergone by these aromatic carbonyl compounds in presence of hydrogen atom donors. The rate and efficiency of this process have been shown to depend on the nature of hydrogen atom donor as well as the nature of the substituents on the aromatic rings. Here we present our results of our detailed studies on the photophysical properties of 4-dimethylaminobenzophenone (DMABP) using femtosecond transient absorption spectroscopic technique with a special attention to the solvation and conformational relaxation dynamics in the S1 state of DMABP.

Geometry optimization for the ground state by molecular mechanic calculation shows that the dihedral angle between the two halves of the DMABP molecule consisting of the phenyl and the dimethylamino substituted phenyl groups attached to the carbonyl group is about 54°. Although we could not perform the geometry optimization for the excited ICT state, the geometry optimization for the ICT state formed in the ground state indicate that, in this state, the dihedral angle between the two phenyl moieties of the molecule is about 90°, i.e. the two halves are orthogonal to each other (Figure 8).

Steady state absorption and fluorescence spectroscopic studies of DMABP in different kinds of solvents reveal significant number of informations regarding the dynamics of intramolecular motions in the excited state: 1. a large shift of absorption in polar solvents, indicating that the Franck-Condon state is an
intramolecular charge transfer (ICT) state, 2. large bathochromic shift of fluorescence, indicating that solvation of the ICT state, 3. exceptionally large Stokes shift indicates large change in geometry during deexcitation in the excited state, 4. non-fluorescent in rigid matrices, indicating that only the relaxed state is emissive. The difference in dipole moments between those of the fluorescing excited state and the ground state has been determined to be about 6.8 D. This indicates the unsymmetrically charge distributed ππ* (in non-polar solvents), or the ICT (in polar solvents) character of the S1 state.

We have studied the early time dynamics of the photophysical processes of DMABP using 400 nm laser pulses of 50 fs duration for excitation and monitoring the transient absorption profile in 470 - 1000 nm wavelength region with about 120 fs time resolution. The temporal profiles have been recorded due to photoexcitation of DMABP in acetonitrile at different wavelengths in 20 nm intervals. A few typical such temporal absorption profiles are shown in Figure 10. The time resolved transient absorption spectra have been constructed using these transient absorption profiles and the same in six time-windows have also been presented in

Fig. 8 Structure of DMABP

Fig. 9 Steady state absorption and fluorescence spectra of DMABP
Figure 10. The spectrum constructed for 0.2 ps time-window consists only one major absorption band in 600 – 800 nm wavelength region having maximum at ca 670 nm. The transient absorption monitored at different wavelengths in this region grows with instrument limited rise time and then decays in ultrafast time scale with lifetime of about 0.15 ± 0.05 ps. This is followed by a further growth of absorption with a growth lifetime of about 0.55 ± 0.05 ps. The time-resolved transient absorption spectra constructed for the later time-windows reveal the ultrafast decay of the band in 600 – 800 nm region with the concomitant development of the two absorption bands in 470 – 600 nm and 800 – 1000 nm wavelength regions. The maximum for the latter band is observed to appear at ca 950 nm at 0.4 ps. However, at later time-windows, as the absorbance in this band continues to increase up to 5 ps, the maximum shifts gradually towards the shorter wavelength region and the spectrum constructed at 5 ps show the absorption maximum at ca 850 nm. We also observe the growth of the band in 470 - 600 nm region. In 470 - 600 nm wavelength region, we observe only the growth of the transient absorption with growth lifetime of 0.4 ± 0.1 ps. However, the shape of the temporal profiles in 800 – 1000 nm region is sensitive to wavelength. The temporal profiles recorded at 830 nm could be fitted by a function having three exponential terms. Before the decay of the transient species (lifetime is about 0.15 ps), which initially grows with instrument response time (~120 fs), is complete, the absorption due to another species starts growing up to about 5 ps with growth lifetime of about 0.55 ps. The latter decays with longer lifetime, which has been determined to be 160 ps. The temporal absorption profiles at longer wavelengths, e.g. at 900 nm and above, show a single growth component. The growth lifetime this component is wavelength dependent. The growth lifetime increases as the wavelength shifts to shorter wavelength in the 800 – 1000 nm absorption band. However, we also observe the presence of an additional decay component at 950 and 1000 nm following the initial growth of the transient absorption.

Following photo-excitation of DMABP, the FC state or the locally excited (LE) state, in which the two halves of the molecule have the initial dihedral angle of about 54°, should undergo twisting, to attain the more stable geometry with the two halves orthogonally oriented with each other. However, in polar solvents, the surrounding solvent dipoles require reorganization and reorientation around the newly created polar S1 state to attain a new arrangement of solvent dipoles around it, which is known as solvation. Hence, we expect to observe mainly two kinds of relaxation processes following photo-excitation of DMABP: (i) solvation and population spreading in the excited state and (ii) twisting of the phenyl groups. Both of these two processes may be happening simultaneously, immediately after the creation of the excited state. However, in non-polar solvents, solvation process takes a minor role in
Fig. 11 Potential energy surface for relaxation of the $S_1$ state of dimethylaminobenzophenone

Fig. 12 Laser driven accelerator & subpicosecond pulse radiolysis set up (Proposed)
the relaxation dynamics of the $S_1$ state. The schematic potential energy surface diagram for the relaxation dynamics of the $S_1$ state of the DMABP molecule both in non-polar and polar solvents has been presented in Figure 11. In this Figure, we denote the conformationally unrelaxed and relaxed singlet excited states as $S_1$ and $S_1'$, respectively.

**Subpicosecond Pulse Radiolysis Study**

So far, our activities have been limited in studying photoinduced chemical reactions in ultrafast (pico and femtosecond) time domain. Also we have been able to study radiation induced chemical reactions in nanosecond time domain by using electron pulse radiolysis technique. However, in the next Xth Five year plan project, we have taken up the task of building a laser driven sub-picosecond electron accelerator (schematic diagram of the set up is shown in figure 12) to study the short-lived transient intermediate species produced in high energy radiation induced chemical reactions with pico ($10^{-12}$ sec) and sub-picosecond ($<10^{-12}$ sec) time resolution. We like to investigate the following aspects of radiation induced chemical processes using this set-up: a. solvation of excess electrons in polar liquids, b. radiation induced processes in solutes with high concentrations, such as Nuclear Waste, electron mobility in liquids, solids and dense gases, d. slowing down of electrons in spur and electron – cation recombination, e. mechanism of hydrogen generation in LOCA condition.

**Epilogue**

Over the years we have developed the transient absorption spectroscopic techniques to study the ultrafast dynamics of molecular processes. We have chosen simple model molecules to understand the dynamics of electron or charge transfer, proton transfer, solvation and conformational relaxation processes. These informations should be useful to design molecular systems, which will be suitable for applications in photonics and solar energy conversion.

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**References**

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