

INVESTIGATION OF DENDRIMERS AS LIGHT HARVESTING AGENTS.

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ABSTRACT

Dendritic molecules are well suited materials for trapping photon energy, which can be converted back to visible light. Their tree-like structure acts as an energy gradient for the funneling of energy while their periphery provides large absorbing units, resulting in high probability of photon capture and the closeness of the periphery to the core makes energy transfer highly efficient. Using dendritic molecular samples from a Michigan-based company – Dendritic Nano-Technology (DNT), spectroscopic techniques were used to investigate the light harvesting properties of dendrimers. Pure dye samples dissolved in alcohol were mixed with dendrimers. The mixture was excited with 1064 nm Nd:YAG laser and the resulting fluorescent signal collected. Preliminary investigation shows that while dendrimers themselves do not produce fluorescence, they act as suitable encapsulating agents for dye molecules. arising from the 3-photon absorption that resulted in the enhancement of dye fluorescence. Furthermore, while stilbene dye could produce fluorescence by direct 3-photon absorption, other dyes species like BDN and Q5 needed the dendrimer environment for enhanced fluorescence activities, while other dyes like LDS 765 showed a tendency towards lasing at low laser energy. This encapsulating feature suggests that dendrimers have possible application in the production of drug capsules.

Keywords: Dendrimers, Spectroscopy, Photon trapping, Light harvesting, Encapsulation.

INTRODUCTION

Dendrimers are a class of highly branched macromolecules Feuerbacher & Vögtle, 1998). Similar to linear polymers, they consist of a large number of monomers, which are chemically linked together, but unlike linear polymers, which are poly-dispersed (containing long coiled chains of molecules, crisscrossing each other), dendrimers consist of mono-dispersed molecular chains (having the same structure, composition and molecular weight), that branch out from a common center, without any molecular entanglement (Balagani *et al*, 2009). The synthesis and structure of dendritic branches have been adequately discussed elsewhere (Buhleier *et al*, 1978; Oyebola & Sieradzan, 2013).

Dendrimers exhibit properties which make them suitable for a number of potential applications. In particular, their inert, stable and non-toxic nature (Janaszewska *et al*, 2019) as well as their possession of internal cavities (Abbasi *et al.*, 2014) makes them suitable as encapsulation agents for potential applications including

drug delivery, energy harvesting, ion sensing, catalysis and information storage, of which the most widely reported in literature the potential medical applications and energy conversion processes (Bielinska *et al*, 1996; Bourne *et al.*, 1996; Hawthorne, 1993; Jansen *et al*, 1994; Klajnert & Bryszewska, 2001; Kukowska-Latallo *et al.*, 2000). The possibility of encapsulating ‘foreign’ molecules inside dendrimers was first experimented by trapping several molecules of Bengal Pose dye in the cavities of water soluble dendrimer molecules of about 5 nm diameter, resulting in the so-called “dendritic box”, which may become a vehicle for drug delivery, especially given its non-toxic nature (Jansen *et al.*, 1994).

LIGHT HARVESTING

Light harvesting is the trapping of photon energy via peripheral chromophores and funneling to a central point where it can be converted back to visible light (Scholes, 2017). The dendrimer is a good molecular candidate suitable for such conversion process (Gilat *et al*, 1999). Its tree-like

structure acts as an energy gradient for the funneling of energy (Nantalaksakul *et al.*, 2006). Also, the dendrimer periphery provides large absorbing units (Abbasi *et al.*, 2014), resulting in a high probability of photon capture, while the short distance from the periphery to the core makes energy transfer highly efficient (Bradshaw & Andrews, 2011).

Energy thus captured in the core is capable of being released to activate photochemical processes. One such process is photo-isomerization, a process whereby a compound is made to transform from one structural form to another (Jiang & Aida, 1997). Since photo-isomerization requires a finite amount of energy and absorbed photons are usually of low energy, light harvesting must involve a sequential process of multiple photon absorption and storage until the required energy of isomerization is attained (Nakazato *et al.*, 2012).

Furthermore, since molecules excited by photon absorption are easily de-excited through inter-molecular collision and/or fluorescence, the transfer and storage medium must disallow de-excitation that may result from inter-molecular collision (Kelley, 1970).

Dendrimers, with their tree-like structure are well suited for this. A good isomeric compound is the family of azobenzenes (Jiang & Aida, 1997). Azobenzenes are photo-chromic molecules that undergo *trans-cis* isomerization on ultraviolet irradiation, and *cis-trans* isomerization either thermally or on exposure to visible light (Kumar & Neckers, 1989). A series of azobenzene containing aryl ether dendrimers (*trans-L_nAZO*, having *n* aromatic layers numbered 1, 3, 4, & 5), have been synthesized during which it was found that infrared light ($\lambda=6.26\mu\text{m}$, 0.2eV , $\nu=1597\text{cm}^{-1}$) from an incoherent Nichrome source could induce *cis-trans* isomerization of the azobenzene core

located at the center of the aryl-ether dendrimer, contrary to expectation, since the *cis-trans* activation energy (0.82eV) is not expected to be attained by the $6.26\mu\text{m}$ radiation (Jiang & Aida, 1997).

For the 0.2eV infrared light to supply the required activation energy of 0.82eV, it must release at least 5 photons of energy. This is consistent with the observation that the isomerization reaction rate showed a fifth-order dependence on the photon flux, indicating that the L5AZO isomerization is a five-photon process (Jiang & Aida, 1997).

It has been established that an incoherent infrared source is too weak to induce a multi-photon process (Wakabayashi *et al.*, 2000). Even a high-output laser source will hardly induce a five-photon absorption simultaneously, and if it must, the absorption cross section must be extraordinarily large, which was found not to be so for L5AZO during the five-photon absorption process, but rather of comparable order to other phenyl-containing substances (Wakabayashi *et al.*, 2000). This clearly suggests that the L5AZO molecule did not absorb five photons simultaneously, but rather sequentially (Wakabayashi *et al.*, 2000). This interesting photochemical phenomenon has been observed only in large-sized aryl-ether dendrimers having spherical shape (Wakabayashi *et al.*, 2000), suggesting that such dendritic structures can be used as light harvesting antennae (Bradshaw & Andrews, 2011), which can absorb series of photon energy, store it within its core for a finite period of time and then release for some other use (Klajnert & Bryszewska, 2001). This way, it is possible to enhance the lasing action of some dye molecules that can be encapsulated within the dendrimer cavity.

MATERIALS AND METHOD

It has been known that higher generation dendrimer molecules have the ability to

store electromagnetic energy and release it in bigger packets (Gilat *et al.*, 1999). And it has also been known that dye molecules can be encapsulated in dendrimers, either by chemical attachment to the core, or simply by mixing and random “swallowing” (Adronov *et al.*, 2000).

In our experiment, we tested the possibility of having these two phenomena happening together. Stilbene dye was used in these tests. Pure stilbene dye dissolved in alcohol has a wide absorption band around 349 nm and fluoresces around 425 nm. There is no normal absorption at longer wavelengths. First, stilbene dye solution was mixed with the dendrimer, with a purpose of dye encapsulation, and experiment was designed to search for dendrimer-assisted energy conversion, with the infrared radiation from Nd:YAG laser at 1064nm absorbed in the system, and accumulated within dendrimer moiety to a level equivalent to photon energy necessary for excitation of the stilbene

molecule. The evidence of such process would come in form of a blue (~ 420nm) fluorescence from the sample under laser excitation at 1064nm. Behavior of pure stilbene solution in methanol was compared to that of solutions containing dendrimers.

All solutions were stilbene-saturated at room temperature. The experimental setup is shown schematically in fig. 1. The sample is irradiated by infrared light produced by the Nd:YAG laser. Laser light transmitted through the sample is collected in a beam dump (a black absorbing medium). Infrared laser light absorbed in the sample is responsible for excitation. Fluorescence signal (if any) is directed by a lens arrangement into the monochromator. The monochromator allows us to monitor fluorescence at different wavelengths. Signal from the photomultiplier tube (PMT) is amplified and displayed on the oscilloscope screen.

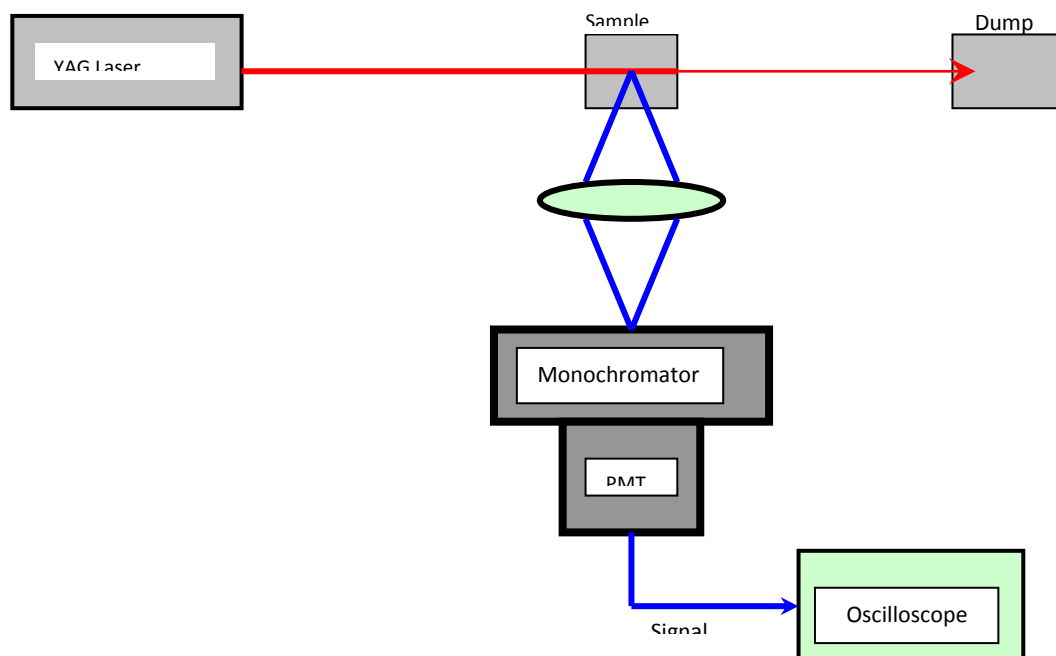


Fig. 1. Experimental Setup for Investigating Production of Fluorescence.

RESULTS AND DISCUSSION

Stilbene in Alcohol Results

First just the pure solution of stilbene in alcohol (methanol) was investigated. Stilbene dye is organic compound *trans-1,2-diphenylethylene*, the simplest in the group of alkene hydrocarbons, with two phenyl groups on either carbon of the parent chain. It has a molecular formula

$C_{14}H_{12}$, a molar mass of 180.24928 g/mol and it is practically insoluble in water. The excitation of the pure saturated stilbene solution in methanol produced relatively strong fluorescence emission at 440nm as well as a weaker SHG at approximately 530nm. The observed emission pattern is shown in Fig.2.

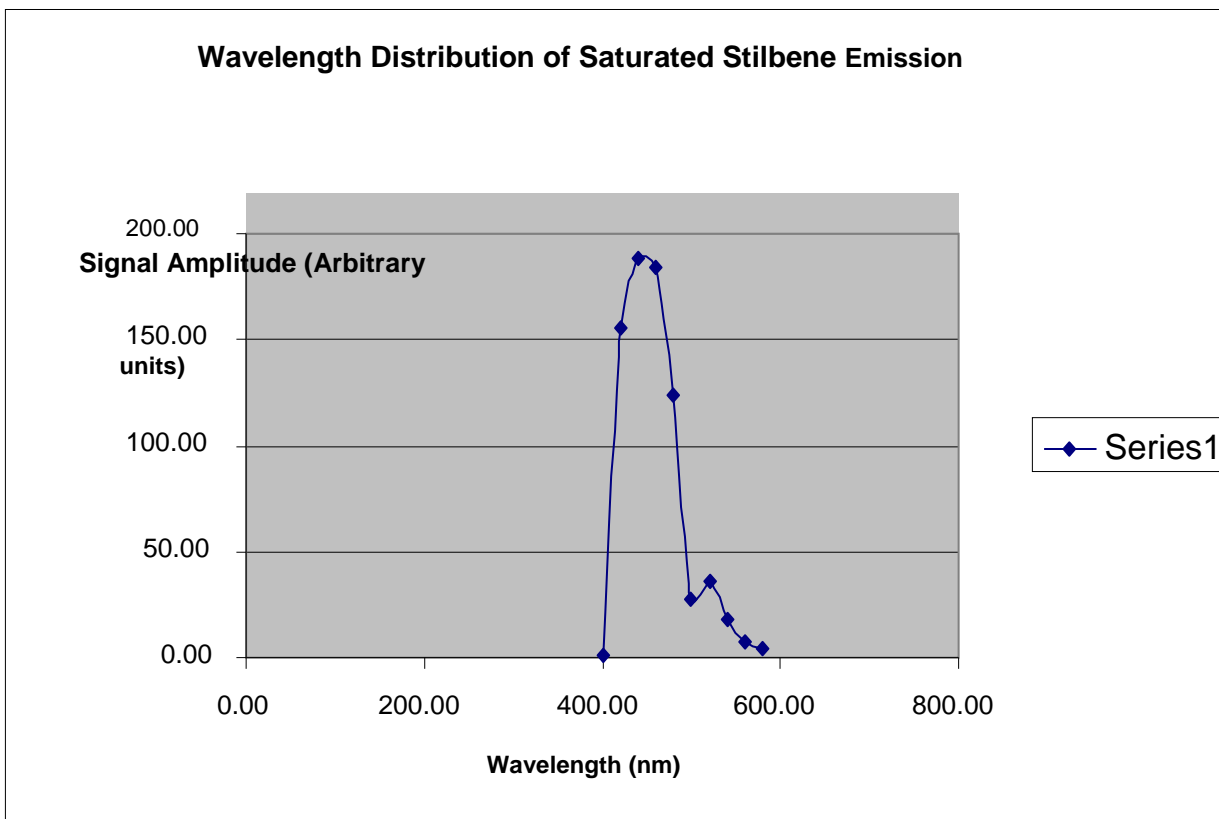


Fig.2. Emission in Stilbene showing strong Fluorescence at 440nm and weak SHG at ~532nm.

As stilbene excitation normally requires radiation at about 300 nm wavelength, which has much higher energy than the 1064 nm infrared (Liu *et al.*, 2008), the origin of the fluorescence is not immediately clear. With the monochromator slit made narrow enough to prevent the overdriving of the photomultiplier tube (PMT) detector and to remove noise, a clear dominance of blue fluorescence was observed, with easy to interpret power dependence.

To identify the origin of the blue light, fluorescence signals were measured as a

function of the Nd:YAG laser light intensity. The log-log graph of the signal/laser intensity relation, as shown in fig.3 is linear graph with a slope of 3. This is indicative of a third power relation between fluorescent signal and laser intensity, and consequently a 3-photon absorption process. Since such a process could satisfy conserving energy as well as angular momentum, it is evident that a 3-photon absorption must be responsible for the observed fluorescence emission. Indeed, since the energy of photon at 1064nm is ~ 1.2eV and blue fluorescence

(~440nm) would have an approximate energy of 2.8eV, it is evident that at least three quanta of photon energy (at 1064nm)

would be required for the emission of blue fluorescence.

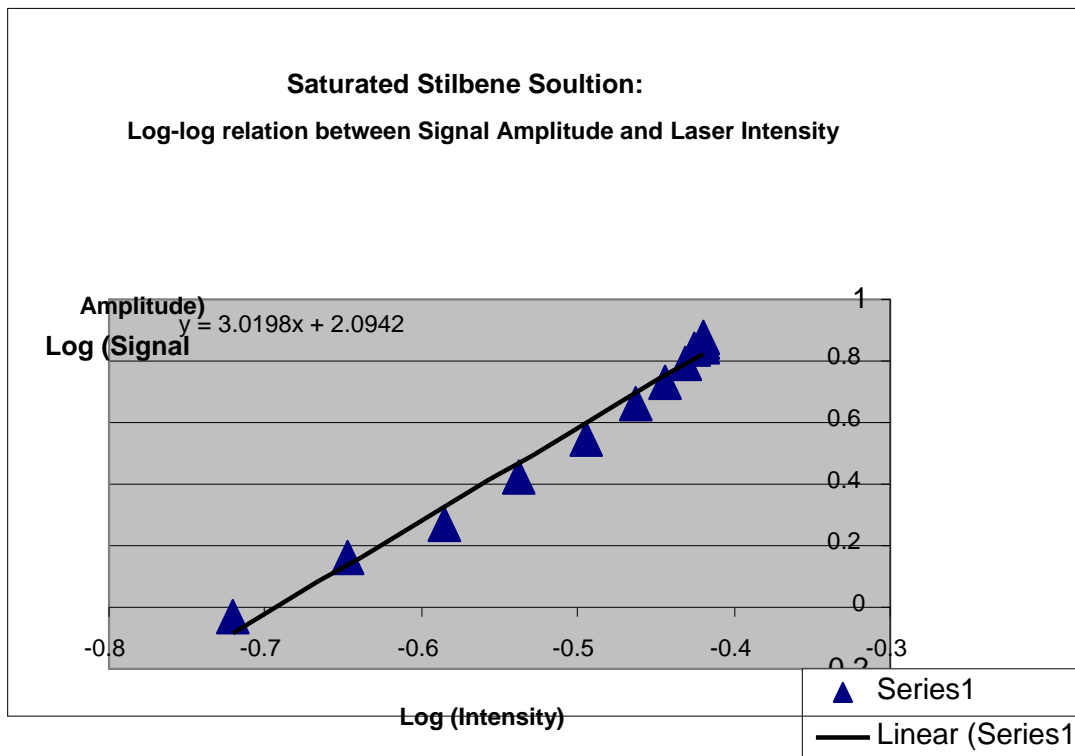


Fig.3. Log-log graph of Signal Amplitude and Laser Intensity – Slope of 3 suggests Stilbene Fluorescence arising from 3-photon absorption.

Dye/Dendrimer Mixtures

Tests performed on the two-component, dendrimer/Stilbene, mixtures did not produce noticeable improvement in the intensity of the blue light. A possible reason for this is that dendrimers do not appreciably absorb around the 1064 nm region, so the Nd:YAG laser light would not harvest well.

To deliver the laser energy more effectively to the solution, an extra component was introduced to the solution in form of a dye characterized by strong IR absorption. Such a dye would either be encapsulated with stilbene or absorb and emit energy (in form of far infrared) outside the dendrimer, to be later caught and accumulated inside the dendrimer for stilbene excitation. The two kinds of IR dyes, which were tested are commercially known as BDN and Q5.

BDN dye is in the class of “saturable” absorbers with a molecular mass of 629.55 and a maximum absorption wavelength of 1060nm when used in 1,2-Dichloroethane solvent. The dye does not normally dissolve in methanol. The 1,2-Dichloroethane solvent, in which it is typically dissolved is a hazardous substance, and for that reason the dye is considered unsafe to use as a laser medium. Hence, while focus was on energy conversion, it was also attempted to affect BDN solubility in user-friendly methanol, through ultrasound mixing of BDN/dendrimer and subsequent dye encapsulation.

Q-5 or Q-Switch 5 is a laser dye with a lasing wavelength range of 1180 to 1530nm, peaking at 1330nm. It has a molecular weight of 775, a maximum absorption wavelength of 1090nm and a

maximum fluorescence wavelength of 1170nm. Q-Switch 5 dissolves well in methanol.

As in the case of pure stilbene in alcohol solution, saturated solutions of the dye/dendrimer mixtures were prepared. To prepare a saturated solution, the substance was added to the solvent in excess, stirred vigorously for an extended period with ultrasound, and then allowed to settle for a couple of hours. Sample solution was carefully drawn from the top.

The pure BDN saturated solution, when irradiated with laser light produced no significant light emission over a wide range of observation wavelengths (400 ~ 820nm). This is not surprising as BDN, if excited would normally fluoresce at a wavelength greater than 1 micron, which falls beyond our PMT sensibility range. The addition of the dendrimer (G6) to the mixture did not result in fluorescence either.

The addition of stilbene to the mixture produced a fluorescence pattern similar to that obtained with pure saturated stilbene, but this time with one strong fluorescent peak at 460nm (still within the blue) and another weak peak at 640 nm (a shift towards the red), as shown in fig.4. While the blue fluorescence was still clearly dominant, the appearance of the red is rather interesting. It may be speculated here that there seemed to be some energy exchange between the two dye materials in the presence of the G6 dendrimer, but the appearance of the red fluorescence needs further study.

The power dependence of the fluorescent signals at 460 nm obtained from this mixture, as shown in the log-log graph of Fig.5 still showed the 3-quanta absorption

signature observed earlier with the saturated stilbene solution, reinforcing the suggestion that the dominant fluorescent activity observed is due to the stilbene dye. Switching to Q5 dye, it was found that saturated alcohol solutions of Q5 were highly absorbing at 1064nm, causing the liquid to heat up and evaporate rather rapidly, even at low laser pulse energy.

Using the Nd:YAG laser, and irradiating the saturated Q5 solution at lower laser pulse energy at 190mJ, weak fluorescence signals were observed at 620nm. Because of the highly absorbing nature of the saturated Q5 solution, it was natural to study its fluorescence pattern over a range of concentration of the solution. The observation wavelength was kept at 620nm, minimum laser pulse energy (190 mJ) was used, and various concentration Q5 solutions were irradiated. A progressive decrease in fluorescence signals was observed as the solution concentration increases, as shown in fig.6. This is known as fluorescence quenching, a suppression of fluorescence at higher dye concentrations.

At the optimum concentration of 28 mg/liter and at 620 nm, the Q5 sample was irradiated with higher intensities of the YAG laser. The results, as given in fig.7, showed a fairly linear relation between fluorescent signal and laser intensity, suggesting that a single photon absorption may be sufficient to produce fluorescence in highly absorbing Q5. As single photon at 1064nm is not energetic enough to excite 620 nm fluorescence, the alternative explanation for the effect is that the fluorescence is thermally produced, and as such it may be proportional to pulse energy.

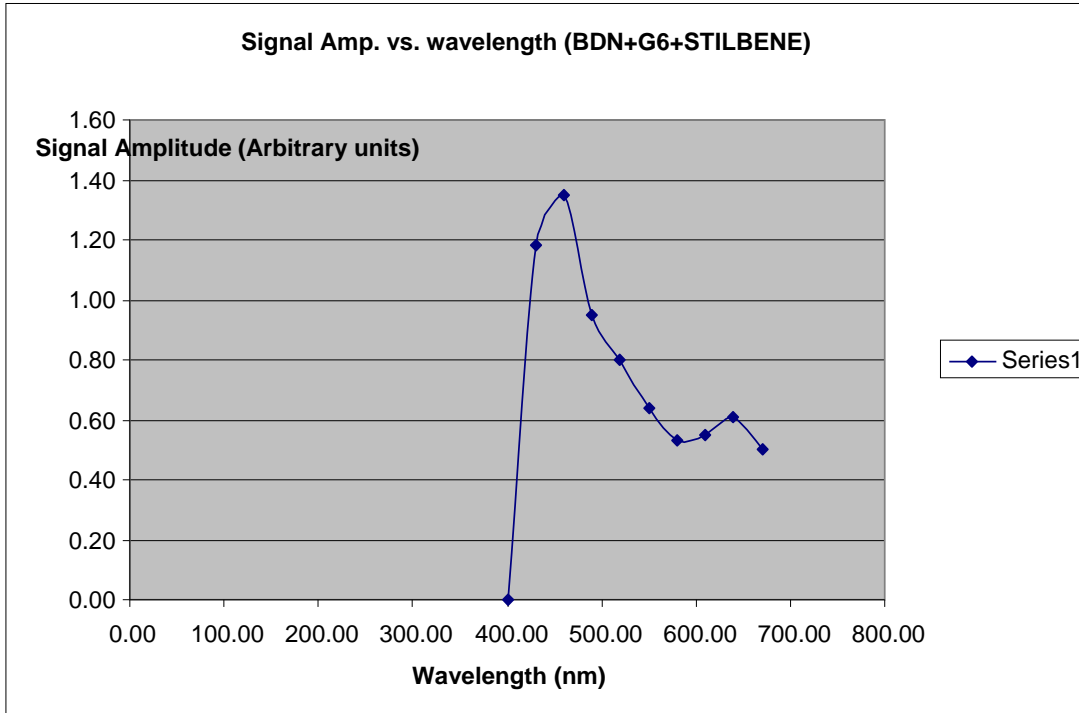


Fig.4. Wavelength distribution of Fluorescence Signals in BDN/G6/Stilbene mixture showing blue and red peaks.

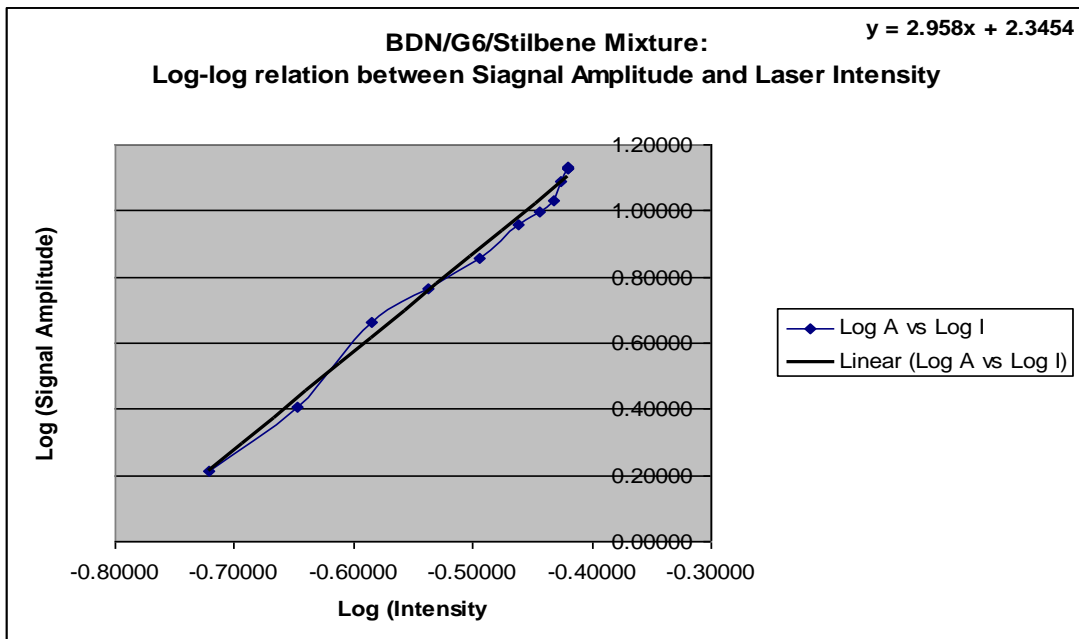


Fig.5. Log-log graph of Signal Amplitude (at 460nm) and Laser Intensity - Slope ~ 3 suggests a dominance of Stilbene Fluorescence.

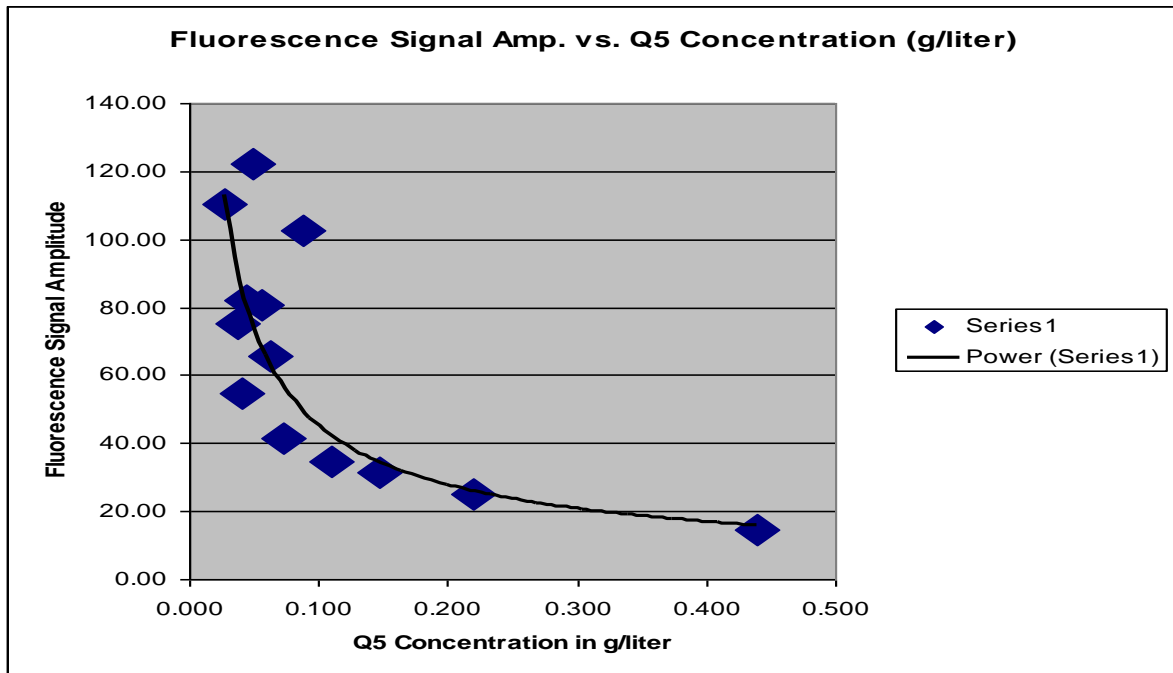


Fig.6. Decay Pattern of Fluorescence with Increase in Q5 Concentration.

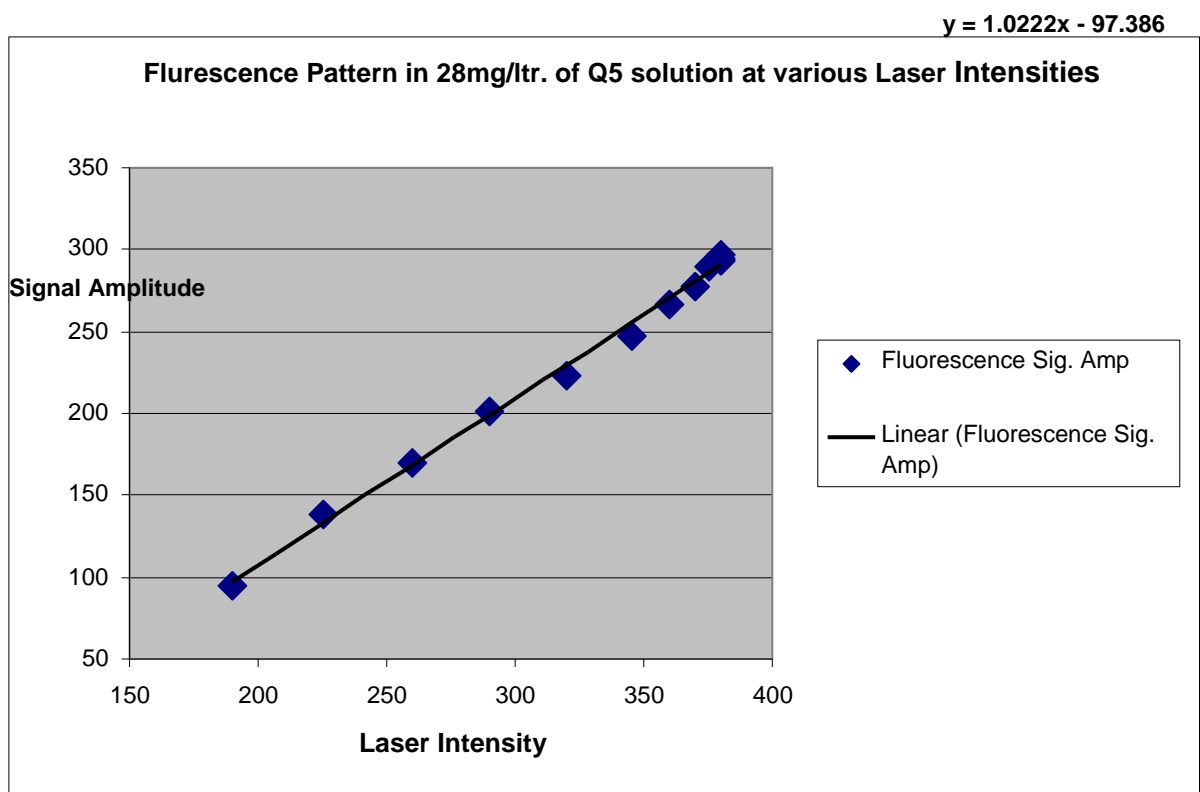


Fig.7. Linearity between Fluorescence Signal and Laser Intensity, suggesting Single Photon Absorption

In the Japanese group experiments, dendrimer-harvested radiation were found to have a wavelength of ~6 microns, which

corresponds to a strong absorption peak in dendrimer molecule (Jansen, Meijer, & de

Brabander-van den Berg, 1995; Otomo *et al.*, 2001).

In this study, the source of electromagnetic energy was the Nd:YAG laser operating at 1.06 microns, which is unfortunately not strongly absorbed by dendrimers. It was therefore necessary to see if energy could be delivered to the dendrimer by the encapsulation of a selected dye molecule that is known to strongly absorb at 1.06 microns, such as Q5, BDN and stilbene. 1.06 μ quanta would be absorbed by the dye and lost to dendrimer in “smaller packages”, corresponding to dye vibronic frequencies. The goal then was:

to see if substantial energy (in the form of several photons) could be stored in dendrimer molecule, to get this energy out in the form of shorter wavelength radiation and to see a rather complicated process whereby the dendrimer would act as a transfer medium between an energy absorbing dye (at 1.06 microns) and an energy acceptor dye (such as stilbene).

Note that short wavelength radiation necessary for visible/UV dyes could not be produced by Q5, BDN, or dendrimer alone.

Generally, the above stated goals were not exactly achieved. In the case of stilbene, it was seen that more than one photon of 1.06 μ m equivalent energy was needed for excitation. Although stilbene molecules showed blue fluorescence, its third order power dependence on laser intensity ($\sim I^3$), indicated that it could simply be achieved by a direct 3-photon absorption, with no dendrimer role in the process.

Considering other investigations involving 1.06 microns source, it was decided to try

a less energy-demanding dye, which could involve lower order laser processes, namely LDS 765.

LDS 765 dissolves very well in methanol and is highly absorbing at visible wavelengths. It has a lasing wavelength range of 738 – 800 nm at a molar concentration of 170 mg/liter. Using 11.0 mg of LDS 765 in 10ml of methanol, a saturated solution of the dye was prepared and irradiated with 1.06 μ m Nd:YAG laser light. Fluorescence signals were measured within the wavelength range of 600 to 930 nm, and a broad fluorescence peak was observed around 750 nm, as shown in fig.8.

At the wavelength of fluorescence peak, and using low energies of the laser beam, the variation of fluorescence signal amplitude with the energy of the laser was observed.

The log-log graph of signal amplitude and laser intensity, shown in fig 9, is a roughly linear graph with a slope of ~ 1.74 . Based on this result, the source of the fluorescence emission is not clear, but the fact that the power of the dependence is almost of the second order (quadratic relation between fluorescence and the energy of laser) indicates strong contribution from 2-quanta process. If it was strictly a quadratic relation, it would have been a case of 2-quanta absorption, similar to the 3-quanta absorption needed for stilbene fluorescence. The fact that the slope is not exactly 2 may be associated with some contribution from first power absorption, most likely arising from heat.

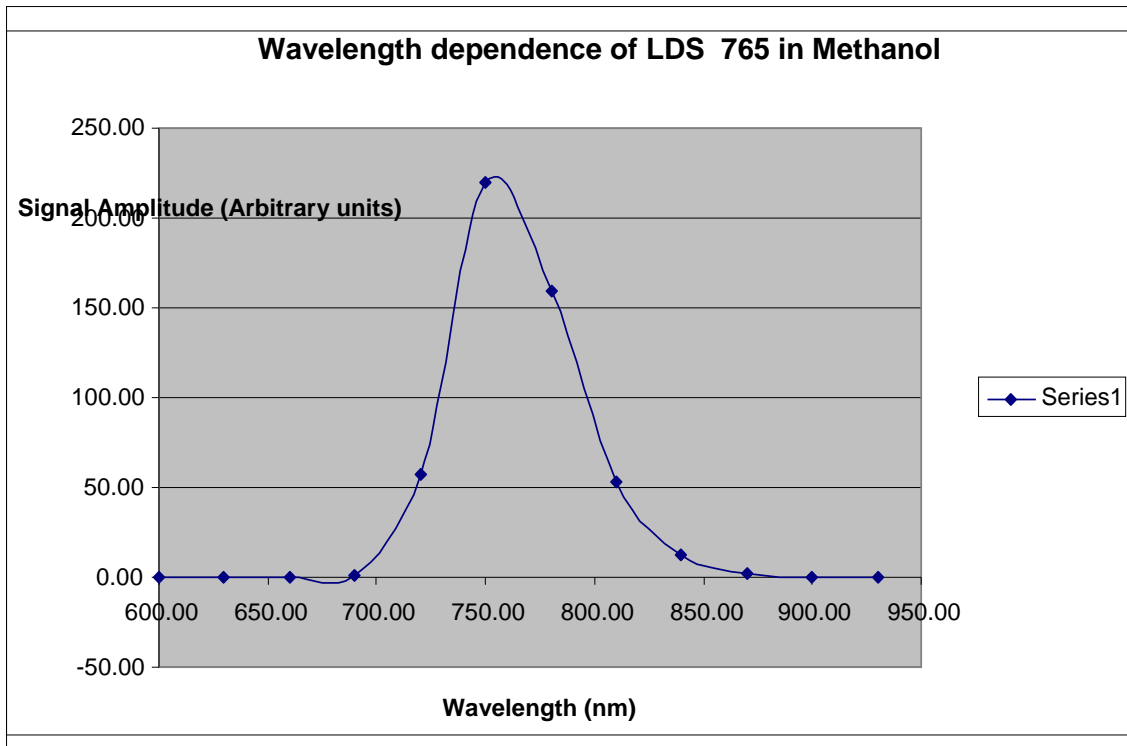


Fig.8. Wavelength Dependence of Saturated Solution of LDS 765 showing Peak Fluorescence at 750nm.

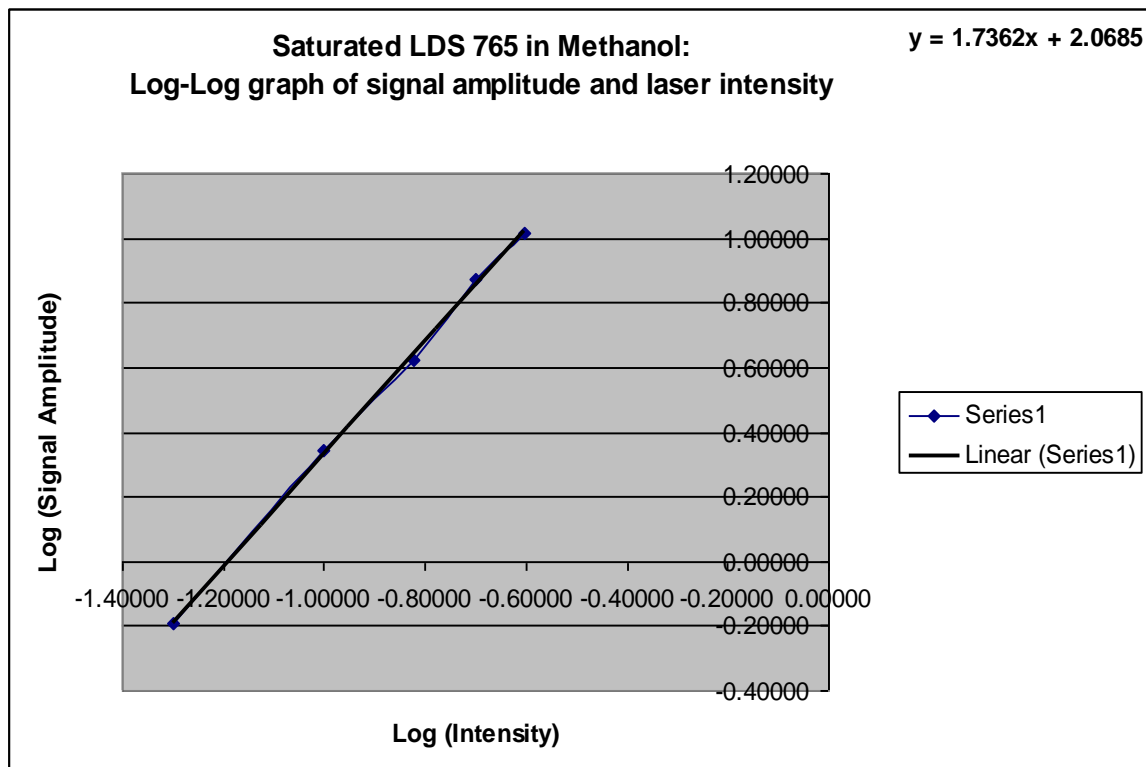


Fig.9. Log-log relation between Fluorescence Amplitude and Laser Pulse Energy in Saturated LDS 765 Solution.

CONCLUSION

The goal was to test for dendrimer-assisted energy conversion either by storing some quanta of photon energy in the dendrimer moiety, or by transferring energy between the molecules of different dye species in the presence of dendrimer. Various kinds of dyes were tested, including those that do not normally dissolve in alcohol. The possibility of solubility increase in alcohol by vigorous ultrasound mixing and encapsulating in dendrimers was also tested.

Observance of fluorescence in the blue, and sometimes red is clear indication of up-conversion from infrared to the visible region. The encapsulation of dyes inside dendrimers has greatly enhanced this up-conversion process, with the degree of conversion varying from one sample to the other. For example, while stilbene dye could produce fluorescence by direct 3-photon absorption, other dyes species like

BDN and Q5 needed the dendrimer environment for enhanced fluorescence activities, while other dyes like LDS 765 showed a tendency towards lasing at low laser energy. This goes to show that dendrimers, apart from being nonlinear optical materials (NLO), as revealed in the production of second harmonics (Oyebola & Sieradzan, 2013), are suitable spectroscopic materials suitable for applications ranging from light harvest to drug delivery.

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