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Volume 2, Issue 3, March 2014 ISSN 2321-6441

Sol-Gel Synthesis Of Silica Xerogel Film Doped Uv Dye Bisbenzimide

Seenaa Ibrahem[(1)], Harith Ibrahem[(2)]

[(1)]University of Baghdad, college of sciences, depertment of physics
[(2)]University of Baghdad, college of sciences, depertment of physics

Abstract

Sol – gel Method was used for the preparation of bisbenzimide H33258doped silica xerogels film , using tetraethylorthosilicate [TEOS, Si(OC2H5)4] as the precursor for the silica network. Silica alcogol was prepared by hydrolysis and polycondensation of ethanol (EtOH) diluted TEOS in the presence of HCL catalyst. Silica solution containing TEOS and pure ethonal were mixed in volume ratio of 5:5 the sol(A) ,and deionized water mixed with HCL and dye solution at volume ratio 10:0.81(0.1M):5 the sol(B) .sol (B) were slowly added to sol(A) with magnetic stirring for two hour at the tamp. of reaction 60°C.From the absorption spectra for organic dyes in ethanol it is clearly appear that the dye molecules interactions depend on the local electric fields generated by the surrounding polar solvent molecules. The change spectrum width and the intensity of peak for different concentrations.

Key words:- TEOS, Bisbenzimide, Sol-Gel, Film

1. Introduction :-

The sol–gel method has received enormous attention in the area of materials research. The advantages of the method are low temperature materials processing, high homogeneity of final product and its capacity to generate materials with controlled surface property and shape and pore structures[1]. The process includes the following steps: preparation of the solution, gelation, aging and finally drying of synthesized materials. Various works has been reported in literature on sol–gel processes for manufac-turing glasses, ceramics and inorganic materials [2] . Zaidi and Farooqui [3] and Farooqui have studied several dyes and come across a finding that the silica glass prepared by sol gel technique can be a good active host material for dye molecules [4]. In recent year's, sol-gel technique have been extensively used for in corporation of organic dyes into inorganic host matrix and this area becoming increasingly important for optical applications [5]. Raschella, et al. (2006)photoinduced dichroism measurements have been performed inhybrid silica based sol-gel films containing carbazole units, 2,4,7-trinitro-9-fluorenone (TNF) and disperse Red 1 (DR1) at different concentrations. The dynamicsof the parallel and perpendicular absorbance gives information on the azo-dye orientation under illumination and suggests that the dominant mechanism for the photoinduced anisotropy is the angular redistribution of the DR1 trans molecules[6]. Bisbenzimide H33258 dye,the molecular formula $C_{25}H_{27}CL_3N_6O$,Physical form dark yellow to tan powder with green cast,. soluble in water and ethanol ,the melting point 300 ℃ [7].

Bonding of the organic phase with the inorganic phase in hybrids:-

The nature of interaction at the interface of the inorganic and organic phases is an important criterion in the synthesis of hybrid materials. Inorganic-organic hybrid materials are generally prepared by either the covalent bonding of the inorganic and organic components, or by specific intermolecular interactions between the inorganic and the organic moieties, which includes van der Waals forces, hydrogen bonding or electrostatic forces [8].

Phenomena of Fluorescence :-

Luminescence is the emission of light from any substance, and occurs from electronically excited states. Luminescence is formally divided into two categories fluorescence and phosphorescence depending on the nature of the excited state. In excited singlet states, the electron in the excited orbital is paired (by opposite spin) to the second electron in the ground-state orbital. Consequently, return to the ground state is spin allowed and occurs rapidly by emission of a photon. The emission rates of fluorescence are typically 10^{-8}s , so that a typical fluorescence lifetime is near 10 ns (10 x 10^{-9}s) [9]. The processes that occur between the absorption and emission of light are usually illustrated by the Jablonski diagram. Jablonski diagrams are often used as the starting point for discussing light absorption and emission. Jablonski diagrams are used in a variety of forms, to illustrate various molecular processes that can occur in excited states.These diagrams are named after Professor Alexander Jablonski.

Experimental part:-

1-Chemical Materials:-

All chemical materials used were of the highest purity available,the materials used in the research in the Table(1)

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Volume 2, Issue 3, March 2014 ISSN 2321-6441

Table (1) chemical materials used in this study

Raw Material	supplier	Molecular Formula	Molecular Weight (g/mol)	Density (g/cm^3)	Purity	State of Raw Material
Tetraethoxysilane (TEOS)	Sigma-Aldrich (Germany)	$Si(OC_2H_5)_4$	208.3	0.933	>98%	Liquid
Deionized Water	University of Baghdad/ College of Science/Laboratory of service	H_2O	18	1	high degree of purity/empty of additional ions	Liquid
Ethanol (EtOH)	GCC	C_2H_5OH	46.07	0.785	99.9%	Liquid
Hydrochloric Acid (HCl)	BDH	HCl	36.46	1.19	37%	Liquid
Bisbenzimide H33258 dye	Sigma-Aldrich (Germany)	$C_{25}H_{24}N_6O$ 3HCL	533.88		>98%	solid

2-Sample preparation

2-1 Preparation of dye solutions:-

The dye solutions were prepared by dissolving the required amount of the dye into the solvent; the weight of the dye was measured using a Mattler balance of 0.53mg sensitivity. The weight of the dye W (in g) was calculated using the following equation

$$W=M_w V C/1000 \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where M_w molecular weight of the dye (g/mole), V the volume of the solvent (ml), C the molar concentration (mole/litter).To prepare the diluted solutions, the following equation was used:

$$C_1 V_1 = C_2 V_2 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (2)$$

Where C_1 is the high concentration [M], V_1 is the volume before dilution (l), C_2 is the low concentration [M], and V_2 is the total volume after dilution (l).At first we prepared the dye Bisbenzimide H33258 solution where was dissolved in ethanol at different concentrations (10^{-8},10^{-7}, 10^{-6} and 10^{-5}M).the sample of dye solution shows in figure (1)

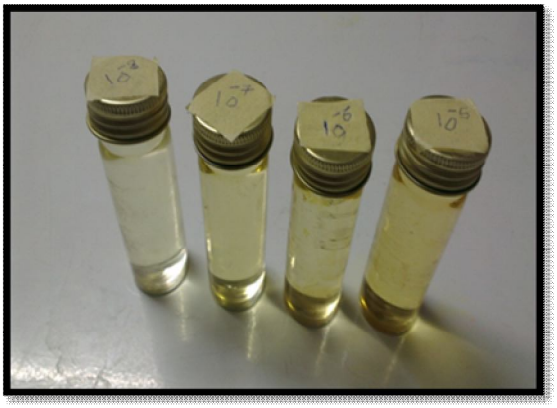

Fig.(1) Dye Solution

2-2 Preparation Of The Silica –Dye Films :-

The preparedsilica solution containing TEOS and pure ethanol were mixed in a volume ratio of 5:5 and denoted as sol (A). In another molds, catalyst solution containing deionized water mixed with catalyst, HCl and dye solution at volume ratio10:0.81(0.1M):5 denoted as sol (B). Types of sol (B) were slowly added to sol (A) with magnetic stirring for two hour at the tamp. of reaction 60° C , preparation the film by spin coating method, and drying the sample 100° C fig.(2) .the sample of silica –dye hybrids film shows in figure (3)

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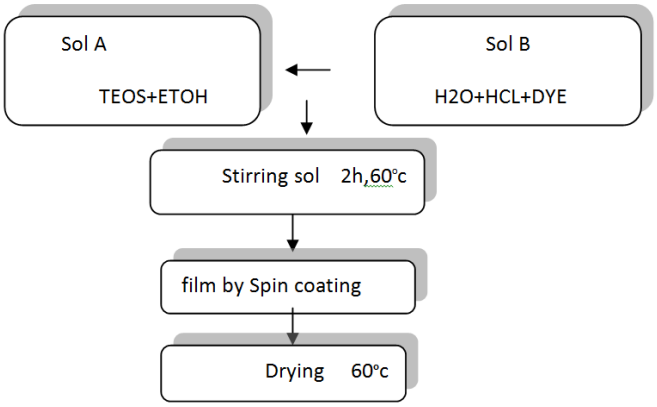


Fig. (2): The Schematic Of The Synthesis Silica Film With Bis.H33258

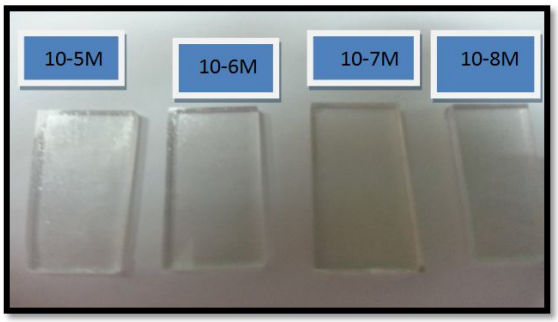


Fig.(3) Silica-Dye Hybrids Film

3. Result and Dissection:-

1-The Absorption Measurement:-

From the absorption spectra for organic dyes in liquid solutions it is clearly appear that the dye molecules interactions depend on the local electric fields generated by the surrounding polar solvent molecules, this is coming from intermolecular solute-solvent interaction forces like dipole-dipole interaction which lead to stretch molecular bonds and shift charge distribution on molecules, causing a change of energy difference between the ground and excited states of the solute[10]**.** The absorption spectra of Bisbenzamide dye solutions at different concentrations at room temperature are presented in figure (4).The observed that the position of absorption band was around 335nm. The intensity of this band increases with increasing the concentration up to 1×10^{-6}M. While at high concentrations (1×10^{-5}M), the absorbance decreased with the increase of the concentration and this is due to high aggregates and dimmer formation[11]. these spectra can be achieved Several indications, the first one is that the Bis.dye is solvent polarity dependent. The second there is a change of the absorption profile with the change of concentration. At high concentrations a wide Also a red shift was observed of the absorption peaks with the increasing of the concentration and this may be ascribed to the solvent effect. Fig.(5) shows the absorption spectra of Bisbenzamide dye in silica sol-gel films at different concentrations. The observed that the position of absorption band was around 335-350nm. The intensity of this band increases with increasing the concentration up to 1×10^{-6}M.

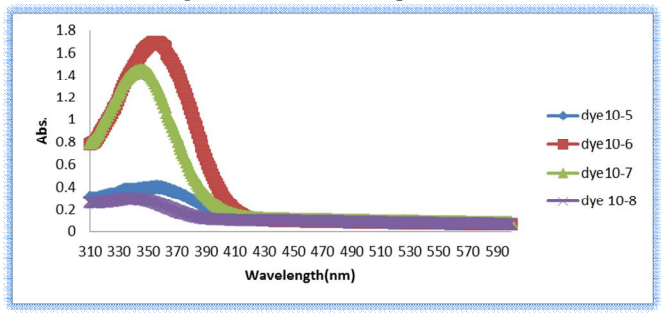


Fig.(4) The absorption Spectra of Bisbenzamide in ethanol solution at different molar concentrations

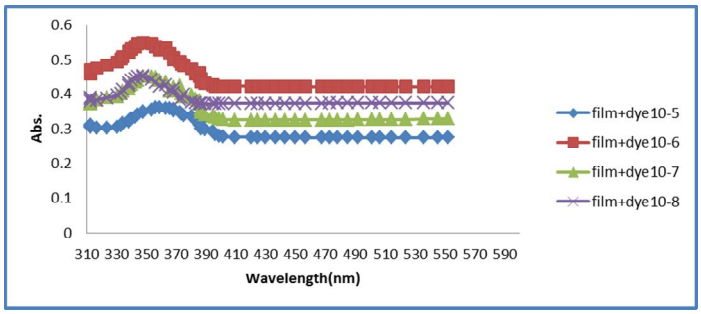
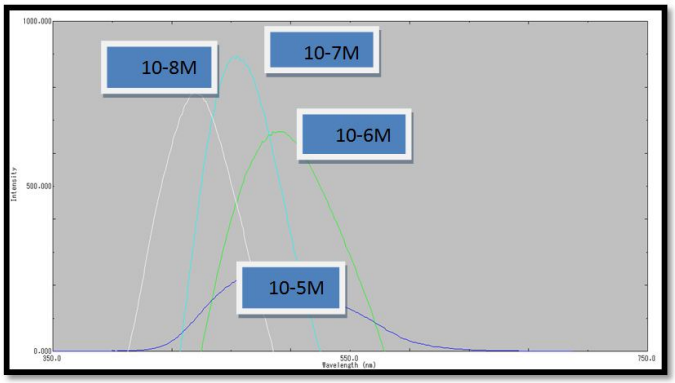


Fig.(5) The absorption spectra of Bisbenzamide in silica films at different molar concentrations

2- Fluorescence Measurements:-

The fluorescence spectra of many organic dyes in liquid solution depend on the local electric field which is induced by the surrounding polar solvent molecules, so this effect is a result of intermolecular solute-solvent interaction forces (such as dipole-dipole or dipole-induced dipole) that tend to stretch the molecular bonds and shift the charge distribution on molecules and thus altering the energy difference between the ground and excited states of the solute molecules. Spectroscopic studies on solutions of polar molecules in polar solvent show that the absorption peak of a solute undergoes red shift in the absorption wavelength as the solvent polarity increases, that mean the dipole-dipole interaction is effective . This shift is not special case for fluorescence spectra but in the same principles in absorption spectra where the fluorescence represents a mirror image for the absorption. In general, at low concentrations, dyes dissolve completely into monomers and obviously no any influence of the dye-dye interaction. Hence, at very low concentrations, fluorescence intensity increases linearly with increasing of concentration, while at higher concentrations fluorescence intensity reaches a limiting value and then decreases with further increase in concentration. Several factors are responsible for this behavior. This can be related to the phenomenon of re absorption and re emission, which ultimately reduces fluorescence emission[12]. This transfer of The fluorescence spectra of Bisbenzamide dye solutions (in ethanol) at different concentrations, the Bis.dye fluorescence around 500 nm is clearly observed. It is clear that the fluorescence intensity increases as the concentration increases as shown in the figure (6,7). This means that the concentration plays important role in affecting the fluorescence intensity and spectral shift.



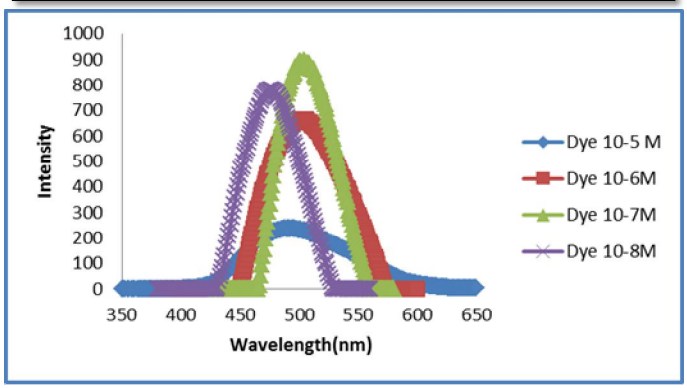


Fig.(6) the Fluorescence spectra of Bisbenzamide dye solution(in ethanol)

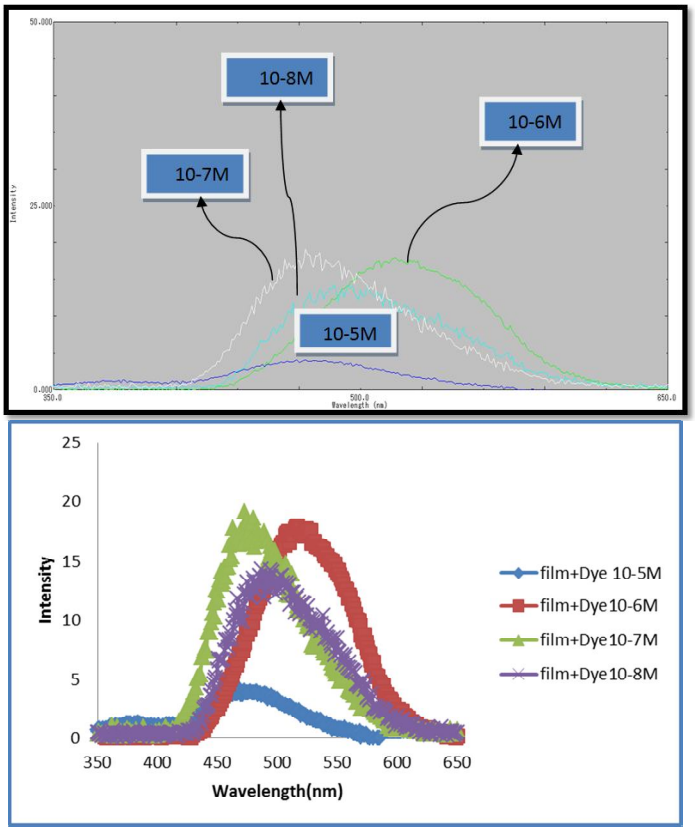


Fig.(7) the Fluorescence spectra of film doped Bisbenzamide dye

4. Conclusions:-

1-The intensity of this band increases with increasing the concentration up to 1×10^{-6}M. While at high concentrations (1×10^{-5}M), the absorbance decreased with the increase of the concentration and this is due to high aggregates and dimmer formation .

2-these spectra of absorbance can be achieved Several indications, the first one is that the Bis.dye is solvent polarity dependent. The second there is a change of the absorption profile with the change of concentration. At high concentrations a wide Also a red shift was observed of the absorption peaks with the increasing of the concentration and this may be ascribed to the solvent effect.

3-This transfer of The fluorescence spectra of Bisbenzamide dye solutions (in ethanol) at different concentrations, the Bis.dye fluorescence around 500 nm is clearly observed. It is clear that the fluorescence intensity increases as the concentration increases.

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