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Synthesis a Sol-Gel for Chemical Fiber Sensor

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ABSTRACT

Sol–gel materials may found plenty of applications, *e.g.*, as carriers for various substances, which can be exploited for sensing purposes or as drug releasing carriers. In this work, the fluorescence properties of two photosensitizers immobilized in silica based sol–gel materials were examined. Sol-gel derived silica possesses many promising features, including room-temperature preparation procedure, porosity, chemical and physical stability. The material preparation procedure is described and practical remarks on silica-based sol-gels are included. It is reported that sol-gels with entrapped various molecules may be used in construction of implants and coatings with bioactive properties. It is shown how to exploit the sol-gel production route for construction of sol-gel coated fiber optic applicators for chemical sensor. We used a Sodium silicate ($Na_2 O_3 Si$) instead of the Trathyl Ortho Silicate (TEOS) ($Si (OC_2 H_5)_4$).We study the optical characteristics for this sample.

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1. INTRODUCTION

Sol–gel glasses find many important applications ranging from everyday applications of silica gel in cleaning, polishing, brewing, printing and adhesive agents to applications in photonics and sensors. These represent multi-million pound industries worldwide, all being potential benefactors of a better understanding of the underlying science and monitoring of the industrial processes [1].

A sol-gel method enables the powder less processing of glasses, ceramics and thin films or fibers, directly from solution. Precursors are mixed at the molecular level and variously shaped materials may be formed at much lower temperatures than it is possible by traditional methods of preparation. One of the major advantages of sol-gel processing is the possibility to synthesize hybrid organic-inorganic materials. engineering materials with exciting properties for a wide range of applications.

The pioneering works on those materials were leaded by Schmidt [1] and Wilkes [2]. They had investigated the possibilities of changing the properties of sol-gel derived materials by incorporating the organic components to a silica matrix. Organic-inorganic hybrids have been called ORMOSILS (Organically Modified Silicates), ORMOCERS (Organically Modified Ceramics) and Ceramers (ceramic polymers) or Polyceram (Polymeric Ceramics).

2. SOL-GEL PROCESS

The sol-gel process is a relatively straightforward method of producing high-quality glasses and ceramicsm [2]. In the context of optical sensors the process is generally employed in the production of silica or titania glass, or mixtures of these two .

The sol-gel process itself leads to formation of gels from mixtures of liquid reagents (sols) at ambient temperatures. It involves several steps: the evolution of inorganic networks, formation of colloidal suspension (sol) and gelation of the sol to form a network in a continuos liquid phase (gel). Drying of the obtained gels, even at room temperature, produces glasslike materials called xerogels (xeros – dry).

The popular sol-gels are silica based sol-gel materials.The generic precursor solution for sol-gel derived silica may be represented by

$Si(OR)4 + H_2O + ROH$ + acid/base catalyst

where R is an alkyl group.

The production process comprises several steps. First, silicate precursor (e.g. tetraethylortosilicate TEOS or tetramethylosilicate TMOS) is mixed with solvent and catalyst and stirred for a few hours. This process leads to hydrolysis of the Si–O–R bonds.The hydrolysis reaction can be catalyzed by acids (HCl, HF etc.) or bases (NH4OH, NaOH etc.). The hydrolysis, gelation and aging/drying processes occur simultaneously. During the "aging" step (after gelation and before drying) the sol-gel derived material expulses the liquid phase (solvent which can be water or alcohol) in the process called syneresis.

Pronounced drying at temperatures not exceeding 100±C leads to formation of "dry" gels, called xerogels. They are relatively sturdy, typically transparent but porous materials. The pore size depends on such factors as time and temperature of the hydrolysis and the kind of catalyst used. The diameter of the pores is directly related to the shrinkage of the "wet" gels. During the drying process the gel volume decreases even several times (which is the main reason of cracking). Addition to the hydrolyzate of small amounts of simple organic solvents (e.g., dimethylformamide or dimethylsulfoxide), stabilizes the resultant gels increasing their mechanical strength and improving transparency .The whole process of forming sol-gels is schematically presented in Fig. 1.

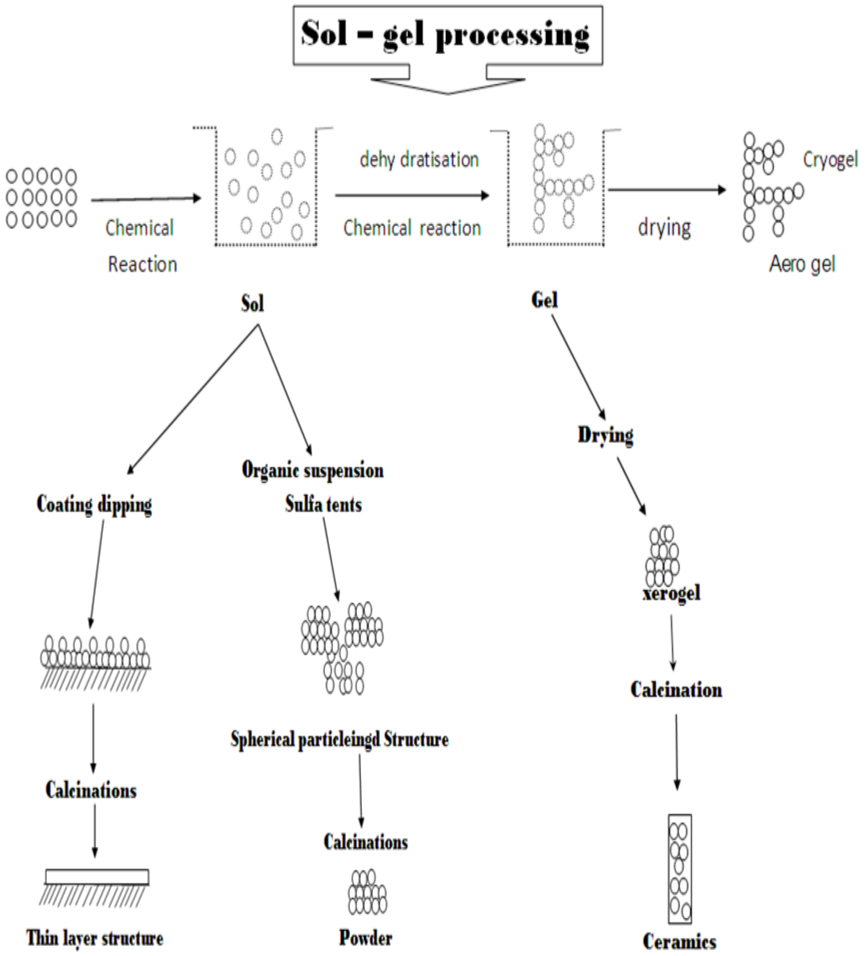


Figure 1. The schematic representation of the sol-gel production process.

These Sol gel utilize the reaction of ammonia vapor with either a pH-dependent dye material or a pH-sensitive film [4],[5],which undergoes a suitable color change or an absorption change. In general, these sensing mechanisms are based on monitoring the absorption or fluorescence characteristics of indicator dyes/sensing films entrapped within a membrane deposited onto a wave guiding substrate or an optical fiber as substituted cladding. The targeted ammonia molecules interact with the immobilized indicator, resulting in changes in absorbance or emission spectra, which are monitored using a proper detector module via an optical fiber or planar waveguide.

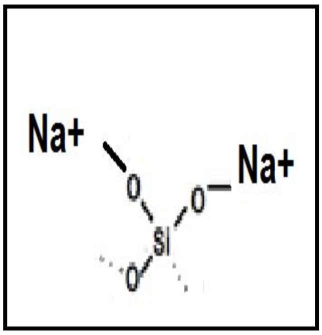
3. EXPERIMENTAL WORK

The most frequently used alkoxides in chamical sensor are tetraethyl ortho silicate (TEOS) and tetramethyl ortho silicate (TMOS).Some properties of the materials are used in the fiber layer coating are:

TEOS (Tetraethly Ortho Silicate)

Formula- $Si(OC_2H_5)_4$
Density = 0.933 gm /ml-
Melting point = -77 c-
Boling point = 166 – 169 c-
Soluble in water-
$Si(OC_2H_5)_4 + 2H_2O \rightarrow SiO_2 + 4C_2H_5OH$
$Si(OC_2H_5)_4 \rightarrow SiO_2 + 20(C_2H_5)_2$
$SiCl_4 + 4ROH \rightarrow Si(OR)_4 + 4HCl$

We used a new material instead of the previous one is:



Sodium silicate

Formula Na_2O_3Si-
Density = 2.4 gm /cm^3-
Melting point = 1088 c to glass-
$Na_2CO_3 + SiO_2 \rightarrow Na_2SiO_3 + CO_{2-}$
Soluble in water-

Sodium silicate prepared from mixed Sodium Carbonate (Soda Ash) In temperature 1300 C^o in special furnaces rotary produces sodium silicate in the form of strength fusible solidifies easily and be on two alkaline or neutral color transparent green blue of blue and can the solid Solubility in water at a temperature room of 20 C^o .

Silicate melt with ethanol to become the Sol Gel after 20-30 minutes with continuous mixing. Leaves the solution 48 hours after preparation for purpose of homogenizing dive fiber.

(1)10 ml deionized water was acidified by 37% HCl to pH=1.
(2) 45 ml of Sodium Silicate(Na_2SiO_3) was mixed with 55 ml of ethanol for 20 min.
(3) 10 ml of the acidified water was added and mixing continued for 1 h.
(4) 10 ml of ethanol containing 10 mg bromocresol purple was added, with stirring being continued for 1 h.

Method

The prepared sol-gel was examined. This process was did by coating the sol-gel in glass slide ,then tested by UV-Vis spectrometer and Fluorescence spectrophotometer.

The absorption spectra was obtained by using the coated layer on the glass sheet sample and an spectro UV-Vis double beam from LABOMED.USA.

The Xenon lamb emitting 200nm -1100nm were used as an excitation source, what fits the absorption band of our photo sensitizers [6]. Fluorescence spectra were obtained by means of the spectrophotometer VARIAN CARY ECLIPSE. Spectra were recorded in the wavelengths range from 200 nm to 1100 nm. Fluorescence spectra were measured for the freshly prepared samples, 24 hours after the end of stirring process. The samples were stored in darkness and at room temperature. Sols were placed into the plastic cuvettes (optical length 0.5 cm, Sigma) and illuminated from above. Emitted light was captured from side, at the angle 90° to lamp beam and guided to the detector. The spectra was recorded. The spectra represent the mean values of the recorded fluorescence intensity normalized to the exciting light intensity.

4. RESULT AND DISCUSSION

Absorption and Fluorescence

The absorption spectra, shown in Figure (2), was obtained by using the coated layer on the glass sheet sample and an spectro UV-Vis double beam from LABOMED.USA. A xenon arc lamp provided “white light” illumination. The absorption maximum of sol gel is found to be at 300nm.

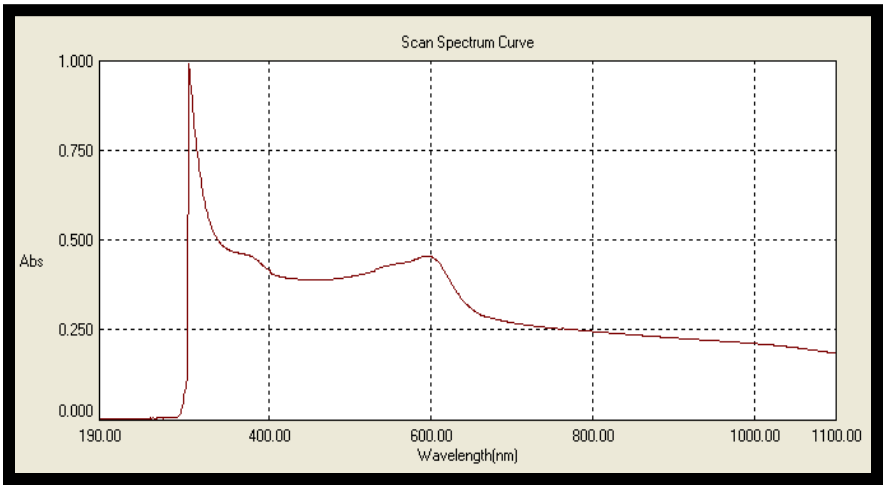


Figure 2. The absorption spectrum of sol gel

Bromocresol purple dye doped sol-gel silica a for possible applications as transmitters for chemical sensor. A fluorescence spectrum, also shown in Figure (3), was obtained using the same sample and spectrometer while under low-intensity 300nm excitation. The fluorescence peaks at 630nm with a shoulder at approximately 650nm.The emission peaks tended to red-shift in sol-gel silica..
The wavelength separation of the absorption maximum and fluorescence maximum, known as the Stoke's shift, is very large, ($\Delta\lambda$ = 300 nm).

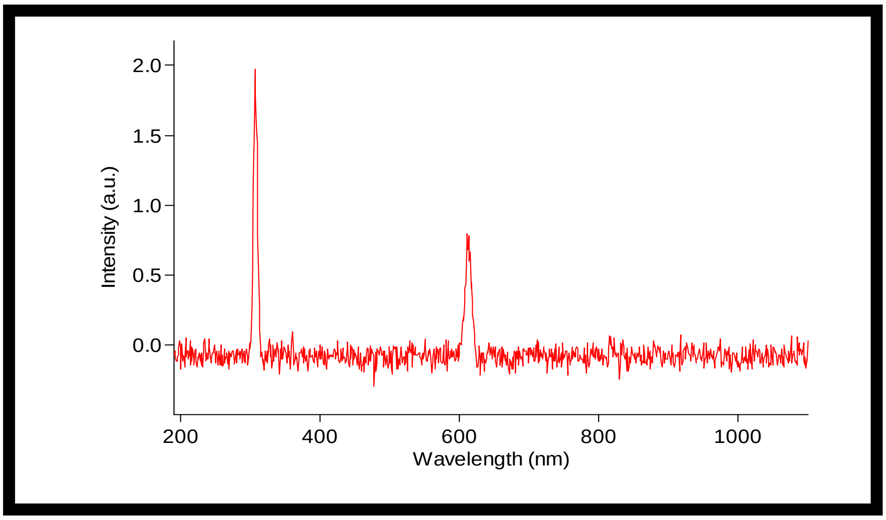


Figure 3. A fluorescence spectrum of the sol gel

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