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Preparation of Orthosilicic Acid by Sol-Gel Technique using Tetraethyl orthosilicic acid (TEOS) and its applications.

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Abstract- Recent trends in the field of new developments demands key features like high sensitivity and high stability of the core like 'Silicon' as in various fields such as silicon chips using high silica technology, Silicon wafers, has all requisite semi-conducting properties, doping ,photoconductivity, junction formation etc.together can be named as "Silicon Valley". Application of Silicon in the bio-physical engineering, as fibre optic sensors has drawn a lot of interest to researchers in the field of Science and technology. A sol is a dispersion of the solid particles (~ 0.1-1 mm) in a liquid. A gel is a state where both liquid and solid are dispersed in each other, which is present in the form of a solid network containing liquid components. Silicon from Orthosilicic acid is taken as a binder /core for the application in broad areas of clinical and biomedical industries. Tetraethylorthosilicate (AR) is reflux hydrolyzed with ethanol as the medium using KOH as catalyst by the sol-gel technique. A white solid compound orthosilicic acid is formed which is collected, washed and vacuum dried. Spectral Characterization of the silicic acid is done by FTIR Spectroscopy, ^{1}HNMR Spectroscopy. Surface morphology of the silicic acid molecule is studied by Scanning Electron Microscopy. Silicon with PVP additive thin films are of industrial applications. Silicon with different ratios of PVP-derivatives can be widely applied for various pharmaceutical applications. Sol-gel doped matrices are of the form of xerogels and possess a network of internal pores and cavities enabling the entrapped molecules to interact with the surrounding medium. The doped matrices possess good optical characteristics which are of key importance for production of optical sensors (optodes).

Index Terms- Silicon, sol-gel, xerogel, binder, sensors, biomedical, PVP.

I. Introduction

Mesoporous silica with regular geometries are in great demand owing to their scientific importance and great potentials in practical applications such as catalysis, adsorption, separation, sensing, medical usage, cosmetology ecology, and nanotechnology[1]. Silicon is considered to be important in human physiology in protecting against the toxic effects of aluminium, and the kinetics of uptake and excretion of silicic acid, the bioavailable form, are studied. On thin layers of silicic acid or alumina, adsorption chromatography a new and highly efficient analytical tool can be used for the rapid separation of lipids of different classes of compounds [2]. The integration of organic or inorganic dopants, like dyes or nanoparticles, highly sophisticated multifunctional hybrid polymers or nanocomposites have become important to achieve active optical functions and formulations e.g. switching, light harvesting, or storage media[3].

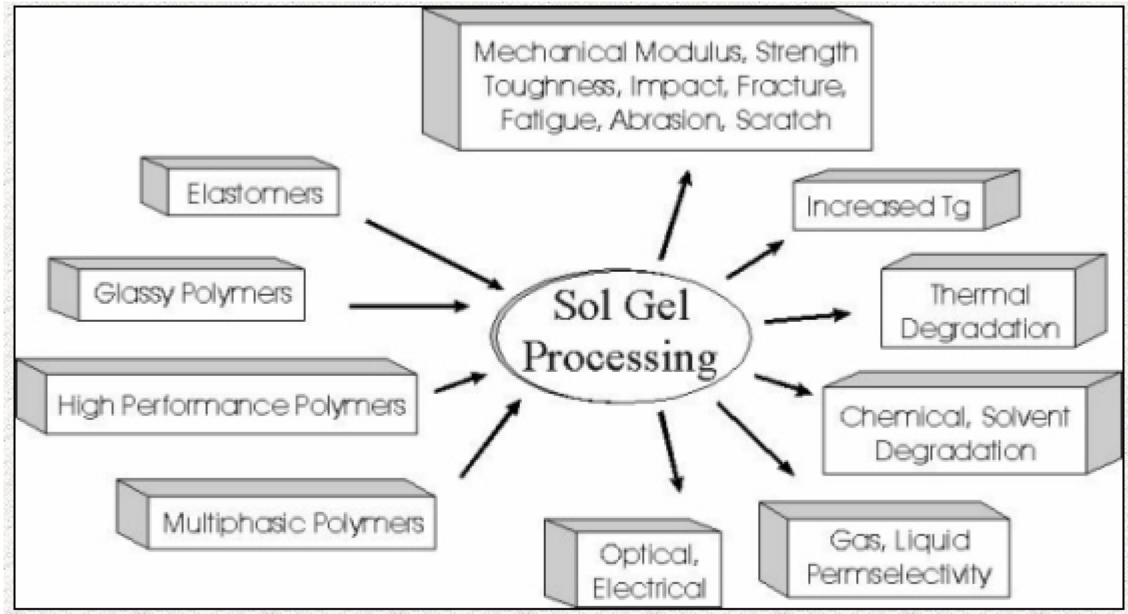


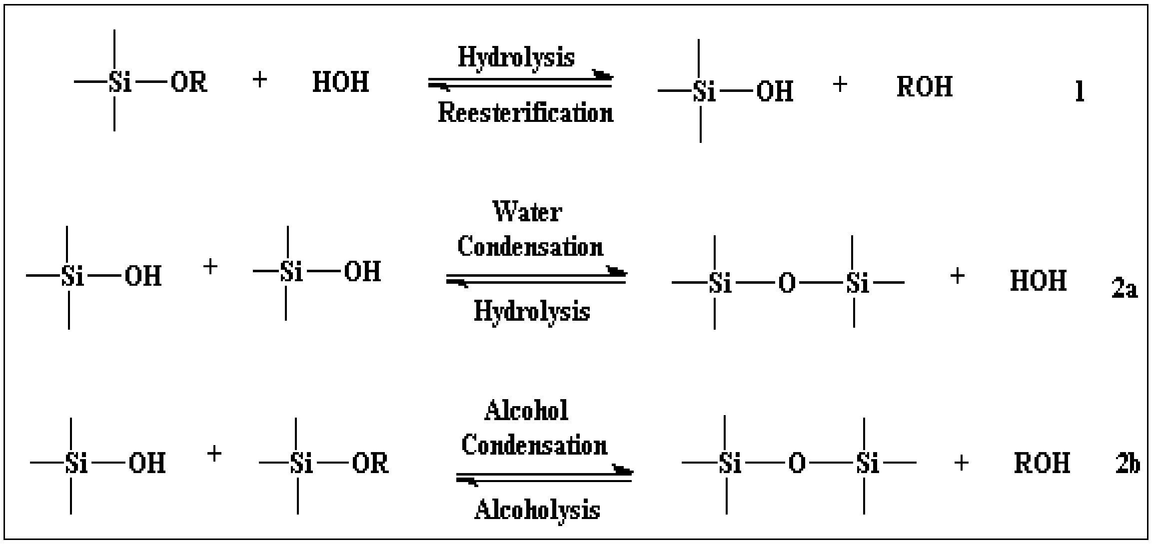
Fig 1.1 Sol- Gel Chemistry

The recent trends in implications of organically-modified silica-based materials include the electro-assisted generation of organosilica films on solid electrode surfaces, the electrochemical characterization of mass transfer reactions in porous functionalized silica, and gas sensors involving sol–gel materials, and the electrochemical characterization and applications of ordered mesoporous organosilicas, and sol gel nanocomposites has been studied. Procedure for applying molecular imprinting functional groups to the inner surfaces of the sol-gel nanotubes for chemical separation of estrone has been reported thereby developing the silica nanotubes for the biological applications. The sol-gel-derived ceramic carbon nanotube nanocomposite electrodes prepared by doping the multiwalled carbon nanotubes (MWNTs) into a silicate gel matrix are a new class of nanocomposite electrodes that are very applicable in electrocatalysis, electrochemical measurements, and development of carbon nano tubes (CNT) - based electronic biodevices.

From macroscopic synthesis of silica nanotubes by the sol–gel template method strong photoluminescence was studied by Ming Zhang et al.. A hybrid nanocomposite film of chitosan/sol–gel/multi-walled carbon nanotubes was developed which can provide a favorable microenvironment for bioelectrocatalytic activity of horseradish peroxidase (HRP) towards H_2O_2 . Nanostructured thin films using sol-gel technology are used to study the semiconductor film structures for gas-sensitive adsorption sensors. ZnO wurtzite clusters prepared from sol gel method exhibit the bright luminescence and can be used as semiconductors [4-11].

II. Sol - Gel Technique

Sol-Gel Process is a wet-chemical technique widely used recently in the fields of materials science and ceramic engineering. Such methods are primarily used for the fabrication of materials starting from a chemical solution which acts as the precursor for an integrated network (or gel) of either discrete particles or network polymers. Sol-gel chemistry produces a variety of inorganic networks from silicon or metal alkoxide monomer precursors. First discovered in the late 1800s and extensively studied since the early 1930s. The evolution of inorganic networks through the formation of a colloidal suspension (sol) and gelation of the sol to form a network in a continuous liquid phase (gel) is involved in this process. A metal or a metalloid element surrounded by various reactive ligands is taken as precursors for the synthesis of these colloids. Metal alkoxides such as tetramethoxysilane (TMOS) and tetraethoxysilane (TEOS) are preferred because they react readily with water. Three reactions are generally used to describe the sol-gel process: hydrolysis, alcohol condensation, and water condensation.



Equation 1.shows the hydrolysis reaction, through the addition of water, replaces alkoxide groups (OR) with hydroxyl groups (OH). Equation 2a. & 2b. shows condensation reactions involving the silanol groups (Si-OH) produce siloxane bonds (Si-O-Si) plus the by-products water or alcohol. A mutual solvent such as an alcohol is utilized which acts as a homogenizing agent; hydrolysis is facilitated due to the miscibility of the alkoxide and water in alcohol. As the number of siloxane bonds increases, the individual molecules are bridged and jointly aggregate in the sol. The sol particles inter knit to form a network a gel is formed [12-18]. Factors that affect the rate of hydrolysis and condensation reactions are:

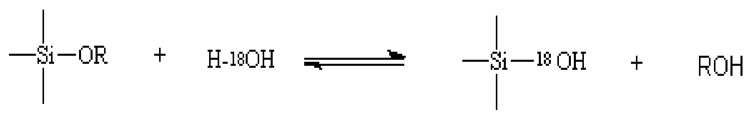
a) pH,
b) Temperature and time of reaction,
c) Reagent concentrations,
d) Catalyst nature and concentration,
e) H_2O/Si molar ratio,
f) Aging temperature and time,
g) Drying.

The structure and properties of the sol-gel-derived inorganic network can be varied by controlling the important factors:

a) pH,
b) Nature and concentration of catalyst,
c) H_2O/Si molar ratio (R), and
d) Temperature.

pH

Hydrolysis occurs by the nucleophilic attack of the oxygen of water on the silicon atom by the reaction of isotopically labeled water [19] with TEOS that produces only unlabelled alcohol in both acid- and base-catalyzed systems as shown below:



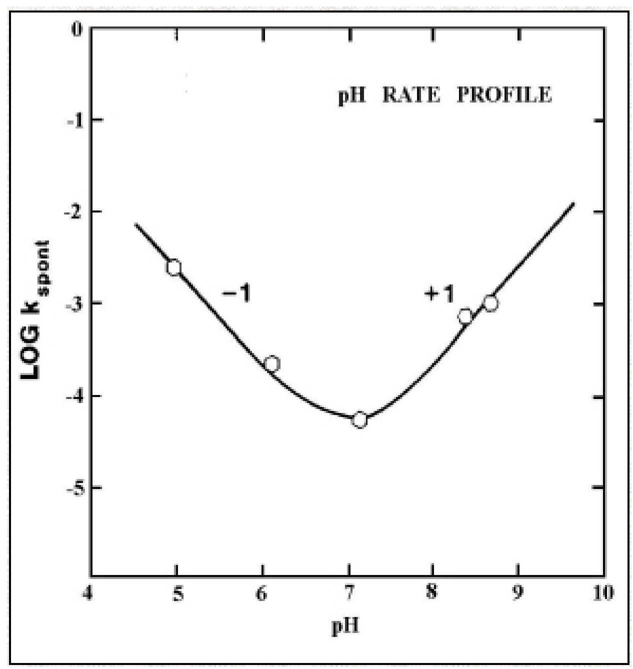


Fig 1.2 pH rate for hydrolysis in aqueous solution

Nature and concentration of catalyst:

The rate and extent of hydrolysis reaction is very much influenced by the strength and concentration of the acid- or base catalyst (HCl, NH_3, acetic acid, KOH, amines, KF, and HF).

Base-Catalyzed Mechanism:

Base-catalyzed hydrolysis of silicon alkoxides proceeds much more slowly than acid-catalyzed hydrolysis at an equivalent catalyst concentration. Basic alkoxide oxygens tend to repel the nucleophile. Under basic conditions, in an SN^2-type

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mechanism, water dissociates to produce hydroxyl anions in a rapid first step. The hydroxyl anion then attacks the silicon atom.

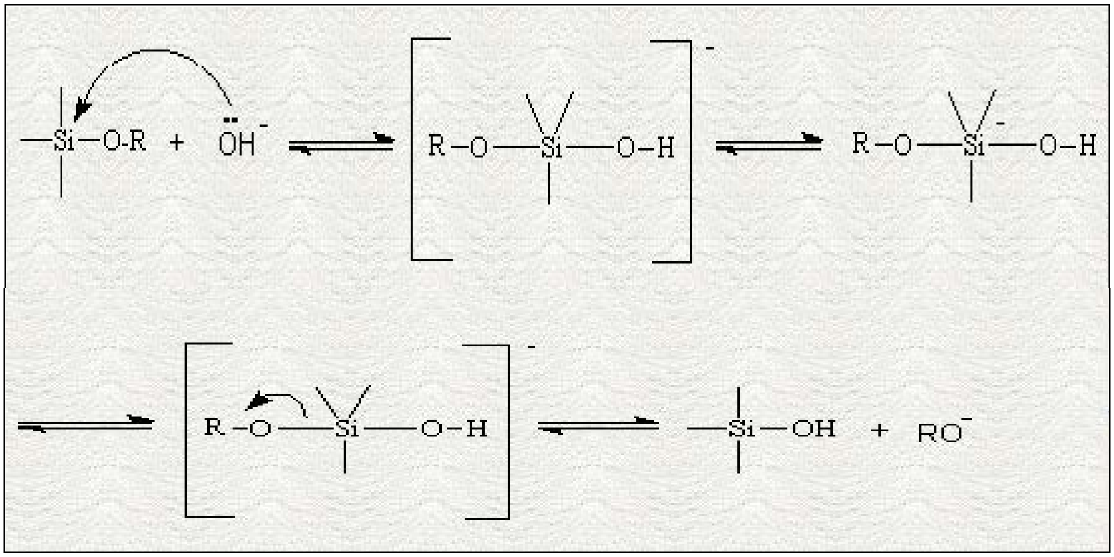

Fig 1.3 Base Catalyzed Hydrolysis

H_2O/Si molar ratio:

An increased value of alkyl groups promotes the hydrolysis reaction. Aelion et al. found that the acid-catalyzed hydrolysis of TEOS to be first-order in water concentration.There is an apparent zero-order dependence of the water concentration under basic conditions. Increased values of alkyl groups (R) accelerate hydrolysis, when R is increased while maintaining a constant solvent: silicate ratio, the silicate concentration is reduced [20-22].

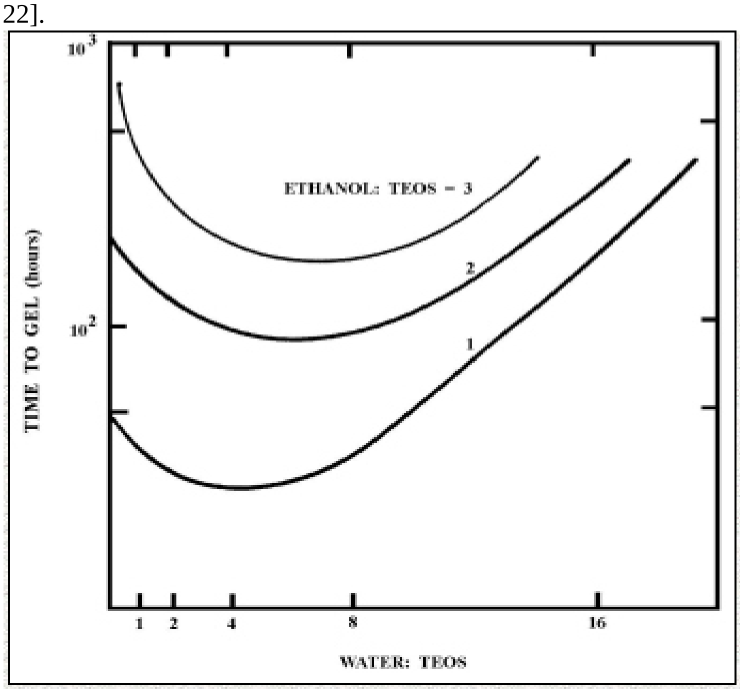

Fig. 1.4 Gel Times dependent on H_2O: TEOS Ratio

Many factors affect the silica network, such as, pH, temperature and time of reaction, reagent concentrations, catalyst nature and concentration, H2O/Si molar ratio (R), aging temperature and time. Silicon oxide networks derived under base-catalyzed conditions yield more highly branched clusters which do not interpenetrate prior to gelation and thus behave as discrete clusters.

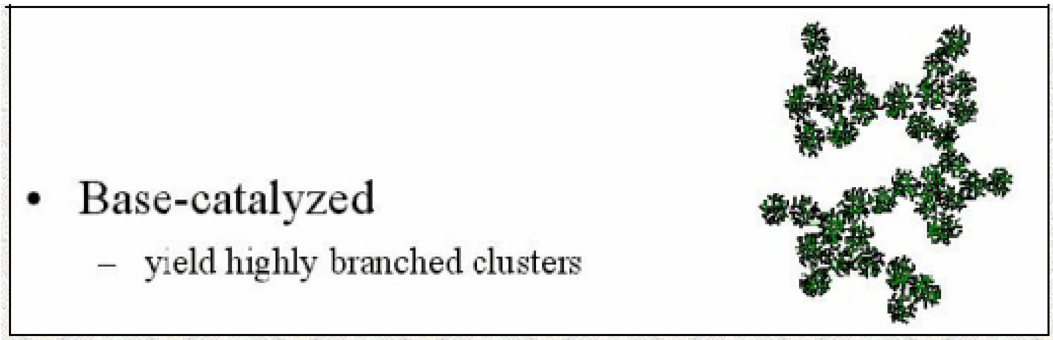

Fig. 1.5 Branched clusters of Silica Network

Sol-gel polymerization takes place in three stages: 1.Polymerization of monomers to form particles 2. Growth of particles 3.Linking of particles into chains, then networks that extend throughout the liquid medium, thickening into a gel [23].

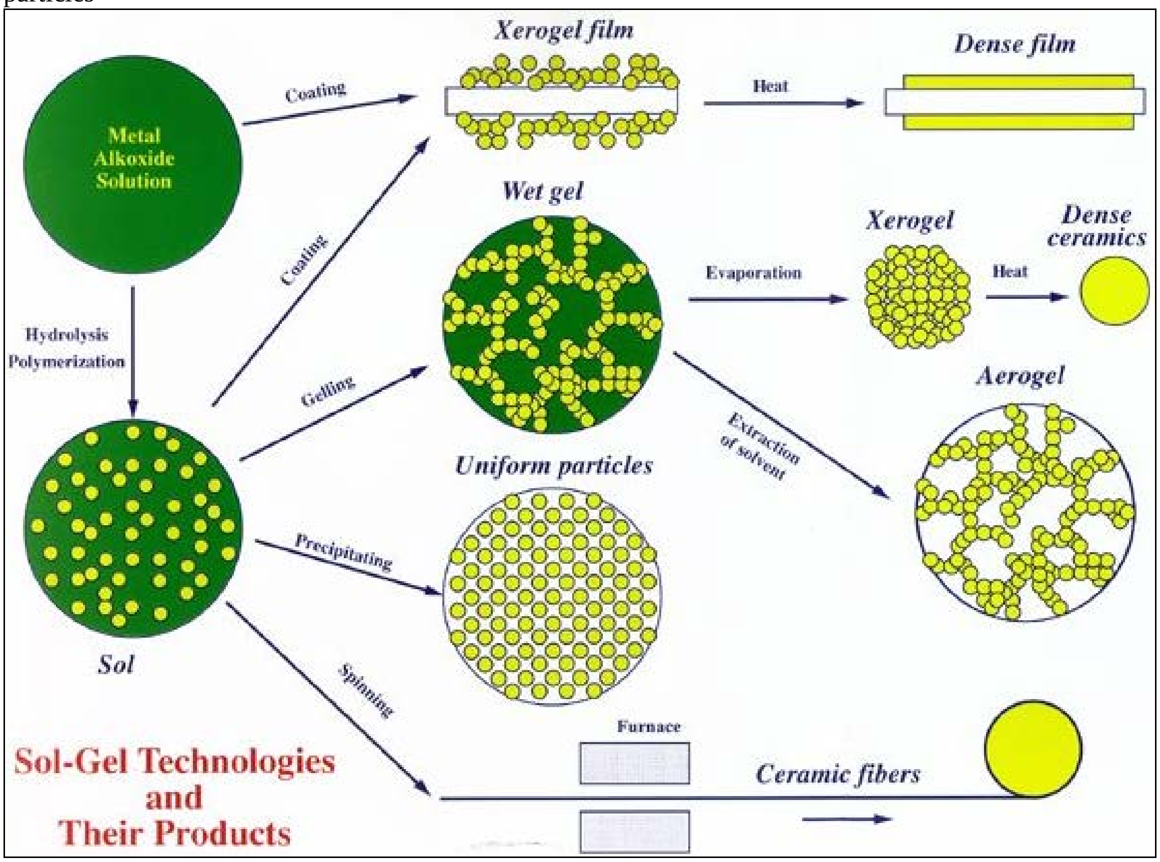

Fig 1.6 Sol-Gel Technologies

Advantages of Sol-Gel Technique:

- Sol-Gel process can produce thin bond-coating to provide excellent adhesion between the metallic substrate and the top coat.
- It can produce thick coating to provide corrosion protection performance.
- This process can easily shape materials into complex geometries in a gel state.
- High purity products can be produced because the organo-metallic precursor of the desired ceramic oxides can be mixed, dissolved in a specified solvent and hydrolyzed into a sol, and subsequently a gel, the composition can be highly controllable.
- It can have low temperature sintering capability, usually 200-600°C.
- It provides a simple, economic and effective method to produce high quality coatings.

- Due to their high chemical homogeneity, low processing temperatures, and the possibility of controlling the size and morphology of particles this technique is in demand.
- The sol-gel-derived materials provide excellent matrices for a variety of organic and inorganic compounds.
- One of the most important features of doped sol-gel materials is their ability for preservation of chemical and physical properties of the dopants.
- This feature marks those materials as almost unique hosts for a number of biologically important molecules which can be utilized in a number of biomedical applications.
- The advantages of sol-gel technology used for construction of biomedical sensors, laser materials or for delayed drug delivery.
- The pore structure and large surface areas associated with sol-gel materials are essential to the development of catalysts and adsorbents to improve the production of gasoline and removing impurities for automobile exhausts and new photocatalysts for splitting water.

III. Applications

Using tetraethylorthosilicate (TEOS) hydrolysis sol–gel reaction followed by solution casting, composite silica/Nafion membranes were prepared by Ruichun Jiang, H. Russell Kunz and James M. Fenton. Membranes were investigated in direct methanol fuel cells (DMFCs). Lower silica loadings in the composite membranes helped to inhibit methanol crossover through the membrane, while higher silica loadings demonstrated a smaller contribution to lowering the methanol crossover [24-26]. Process improvements in silica membrane fabrication were done by Renate M. de Vos, Henk Verweij which can be used for the industrial applications such as purification of H_2 and natural gas as well as the selective removal of CO_2. An ideal zeolite membrane combines many advantages of inorganic membranes (temperature stability, solvent resistance) with perfect shape selectivity. Their "molecular sieve" function can principally discriminate the components of gaseous or liquid mixtures dependent on their molecular size. Composite membranes which consist of a zeolite top layer on a mesoporous ceramic or metal support are very much useful in this regard. Europium complex, $Eu(BA)_3Phen$ (BA = benzoic acid and Phen = 1,10-phenanthroline), was encapsulated in meso-structured silica monoliths, which were using PEG (PEG = polyethylene glycol) as a low cost template and synthesized via sol–gel methods shows increased properties of luminescence intensity and lifetime luminescence [27-29]. Effects of silica on the experimental spinal cord injury by the implication of macrophages in secondary tissue damage. Hypervascularity of the lesion was significantly reduced in animals injected with silica within one day of injury [30]. Silicones are used to treat the surface of almost any material. It is only a temporary surface coating like paint on a board and does not change the substrate material. Siliconized glass will keep blood unclotted for a much longer time. This property makes silicones to be extensively used in Plastic surgery. A superparamagnetic iron oxide (SPIO) nanoparticle is emerging out as an ideal probe for noninvasive cell tracking. A fluorescein isothiocyanate (FITC)-incorporated silica-coated core shell SPIO nanoparticles, SPIO@SiO_2 (FITC), with diameters of 50 nm, as a bifunctionally magnetic vector that can efficiently label human mesenchymal stem cells (hMSCs) has been developed. It is reported that hMSCs can be efficiently labeled with MRI contrast nanoparticles and can be monitored in vitro and in vivo with a clinical 1.5-T MRI imager under low incubation concentration of iron oxide, short incubation time, and low detection [30-31].

IV. Silicon as Binder

Sol-gel processes have been used for the powderless processing of glasses and ceramics from a long time. Novel materials have been prepared during the past few years with the synthesis of hybrid organic—inorganic compounds. Intermediate between glasses and polymers, these nanocomposites open up new possibilities in the field of materials science and have led to the development of functionalized coatings, optical devices, chemical sensors and various biosensors. With the development of optical immunosensor, the sol-gel process has been used to prepare a thin film of amorphous silica, deposited by spin coating on a gold-coated glass slide, and possessing chemically active functional groups (SH, NH_2...). The sol-gel film is activated in aqueous buffers by a bifunctional coupling agent; biological molecules such as antibodies could be covalently bonded on or inside the sol-gel film.

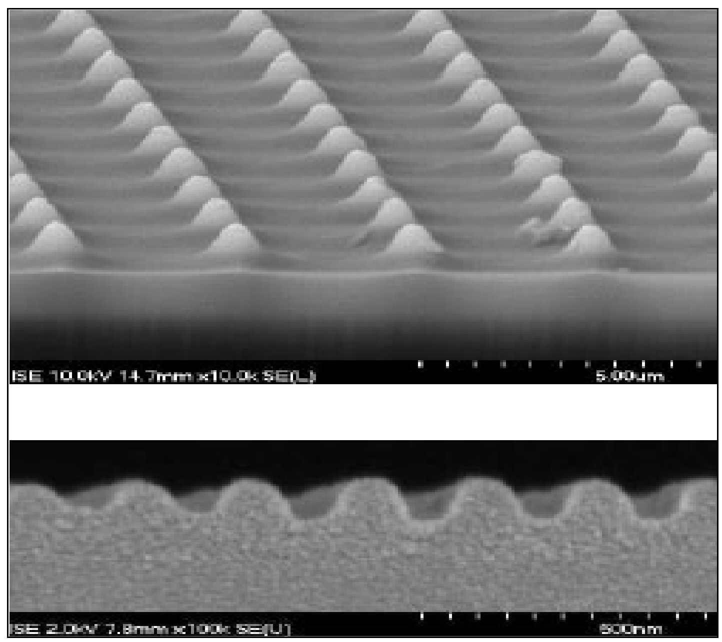

Fig 1.7 Sol gel thin films micro structured surfaces.

Electrochemical biosensors employing sol–gel materials are developed. Low-temperature, porous sol–gel ceramics represent a relatively new class of materials for the immobilization of biomolecules. The various advantages of biogels for amperometric biosensing along with sol–gel-derived bioelectrodes, recent advances and trends, and future prospects have been reported. A sol-gel-based optical CO_2 sensor that employs dual luminophore internal referencing and can be used for application in food packaging technology. A fluorescent pH indicator was immobilized in a hydrophobic organically modified silica matrix, along with cetyltrimethylammonium hydroxide as an internal buffer [31-35]. Siloxane coatings on steel substrates were prepared by the hydrolysis/condensation of alkoxysilanes and/or silanols in the presence of Polyoxometalates (POM) acids (e.g. $H_4SiMo12O40$) that function as catalysts for the process. The siloxane coatings showed good adhesion, and because of their low surface tension/water repellency they exhibited self-cleaning characteristics. The acid catalysis properties of POMs in conjunction with their photochromic, electrochromic, and ion conductive properties to produce composite siloxane materials through the sol-gel approach has been used. Using a silicon binder a photo catalyst was prepared by attaching TiO_2 powder in the sol state to fluidizing spherical ceramic carriers. When sodium ethoxide was added to the silicon binder at a sodium ion to Si ratio as much as 80% of the initial photo catalytic activity was maintained after the photo catalyst had been agitated at 180 rpm for 300 min. Adding both boric acid and sodium ethoxide at a certain ratio has increased the photo catalytic activity and stability by three and four times, respectively [36]. Based on the deposition of a 10 to 30 mum thick hydrogenated amorphous silicon (-Si:H) sensor directly on top of integrated circuits radiation detectors have been developed. Monolithic arrays of silicon drift detectors (SDDs) have been proposed to be used with scintillators for high-position-resolution γ-ray imaging applications, gives better noise performance and also used as photo detectors. Real-time dosimetry is an important issue in most radiotherapy applications. Silicon Ultra fast Cameras for electron and gamma sources in Medical applications, is addressing the development of an imaging device for extended radioactive sources based on monolithic and hybrid-position-sensitive silicon sensors. Monolithic active pixel sensor detectors are produced aiming at development of future vertex detectors, e.g. the Linear Collider, and for medical imaging, e.g. radiotherapy, dosimetry, etc. Advancement in microelectronic industry has made Silicon one of the most popular materials for radiation detector in the biomedical applications in diagnostics and cancer treatments. Silicone because of its high biocompatible and biodurable nature and the material properties attributed are hydrophobicity, low surface tension, and high thermal and chemical stability. Haemocompatibility testing has suggested that Pt cored silicone tubing are much superior to PVC. Silicone implants are widely used in breast, scrotum, nose, chin, cheek, calf, and buttocks [37-42].

Fig 1.8 Silicone Heart Valves

V. PVP-Silica Additive

Poly (vinyl pyrrolidone) (PVP) and silica/PVP hybrid films exhibit bluish Photoluminescence (PL) in response to ultraviolet (UV) light excitation. This behavior was made good use of in preparing luminescent silica/PVP hybrid films [43]. Lead zirconate titanate (PZT) films were deposited on platinized silicon substrates by spin coating using organic macromolecule polyvinylpyrrolidone (PVP) as an additive by Peng Shi et al. The fatigue resistance properties were improved and the polarization value decreased only 8% of the initial polarization at 10^8 cycles. Silica-based hybrid nanostructures using poly(N-vinylpyrrolidone)-coated [60]fullerene single-walled carbon nanotube and block copolymer templates has been used to construct the novel organic/ inorganic-hybridized materials which offer novel technical solutions that can combine organic and inorganic functional synthons. Liquisolid formulations containing PVP as additive using silica as adsorbing surface, exhibited significantly higher drug dissolution rates (carbamazepine) compared to the compacts prepared by the direct compression technique. A novel amperometric biosensor for the determination of lactate was developed by immobilizing lactate oxidase and an osmium redox polymer ([Os $(bpy)_2(PVP)_{10}Cl]Cl$; abbreviated Os-polymer) on the surface of a glassy carbon electrode, which is followed by coating with a sol-gel film derived from methyltriethoxysilane (MTEOS). The main developments on the different types of biosensors, including DNA-based, enzymatic, optical, self-assembled monolayers and the third generation of biosensors as done by PVP-Silica organic inorganic thin hybrid films. Incorporation of PVP into the sol precursor has enabled uniform and crack free films with thicknesses of up to 2.4 microns can be widely applied in microelectromechanical systems. Amorphous highly dispersed silica which is effectively used for the treatment of complex therapy patients of surgical, infectious and other disorders is modified by PVP adsorption, a promising agent for medicinal application. The main advantages of the use of hybrid organic—inorganic nanocomposites result from their high versatility which offers a wide range of possibilities to fabricate tailor-made materials in terms of their chemical and physical properties, and macroscopic shape molding. Such materials emerging out in this field are known as 'sol-gel photonics' [44-50]. Rapid advances in biosensors is due to rapid growth in the development of new biomaterials such as conducting polymers, copolymers and sol gels etc and the reported improvements in sensing techniques. Due to specificity, portability, simplicity, high sensitivity, potential ability for real-time and on-site analysis coupled with the speed and low cost, biosensors [51] have broad applications in food analysis, environment control, clinical detection, drug and agriculture industries etc. An amperometric tyrosinase enzyme electrode for the determination of phenols was developed. A grafting copolymer was introduced into sol–gel solution. The tyrosinase retained its activity in the sol-gel thin film and its response to several phenol compounds was determined at 0 mV vs. Ag/AgCl (sat. KCl). Sol-gel glasses can be cast into desired shapes and are optically transparent, so it is possible to couple optics and bioactivity to make photonic devices and biosensors. High specificity and sensitivity of enzymes and antibodies allows the detection of traces of chemicals. Thus encapsulation of biomolecules in sol-gel glasses can be used for the production of metabolites, the realization of immunoassays and even for cell transplantation. Silica core–polypyrrole shell composite particles were fabricated by chemical polymerization of pyrrole monomer on the surface of the silica spheres. The steric agent poly (*N*-vinylpyrrolidone) (PVP) was used as anchor-molecule between core and polymer monomer. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) images show that the polypyrrole (PPy) shell is uniformly coated over the silica surface. UV-Vis spectra show the macromolecule PVP was retained in the capsules [52-54]. An improved approach for the coating of super paramagnetic magnetite (Fe_3O_4) nanoparticles with shells of amorphous silica was done and can be extensively used for various biological applications. Magnetic Fe_3O_4/SiO_2 particles with rod-like structure and hollow interior have been constructed by a template method in which, β-FeOOH was firstly synthesized as the rod-like template to fabricate β-FeOOH/SiO_2 core/shell-like particles [55-56]. These particles showed ferromagnetic behavior which may provide potential applications in biological area. Poly (vinyl pyrrolidone) was used successfully to control the size and distribution of silver nanoparticles generated on inorganic silica nanofibers. The inorganic nanofibers were electrospun using sol-gel process of silicates, and the diameter of the prepared nanofibers was unaffected by adding up to 7% of poly (vinyl pyrrolidone).

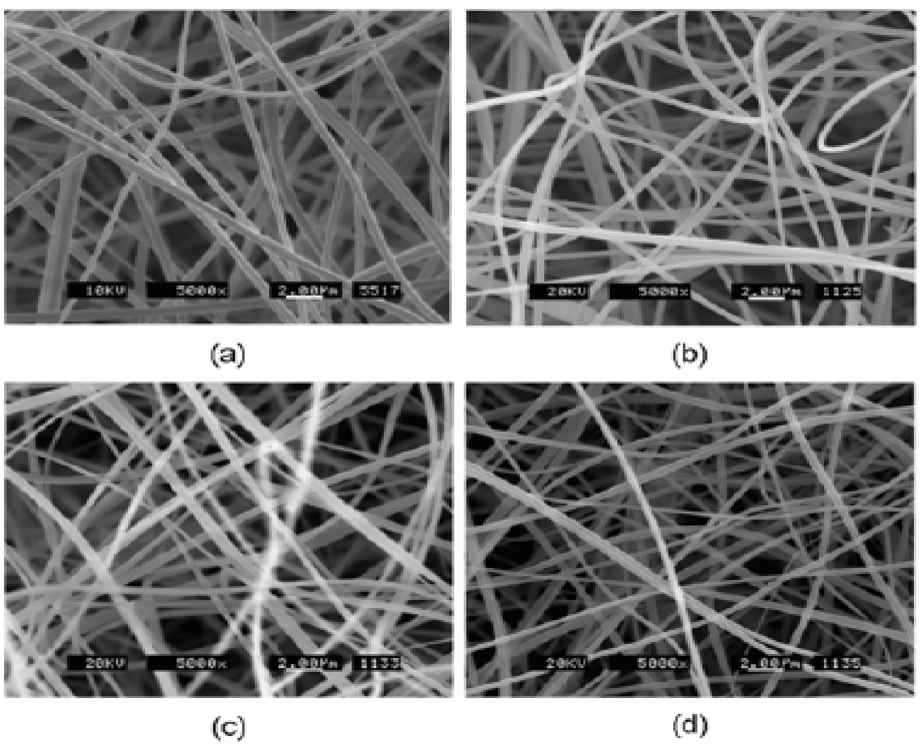

Fig 1.9 SEM images of 0.5 wt% AgNO3-containing silica nanofibers with different PVP contents: (a) 0%, (b) 3%, (c) 5%,and (d) 7%. (Scale bars 2 µm.)

PVP is very effective on controlling nanoparticle size, and with only 1% addition of PVP, the size of the generated Ag nanoparticle had decreased to about 30% to the one without PVP. The silica nanofiber web containing Ag nanoparticles may find its uses in various filtering applications where good thermal stability and antibacterial activities are required. Different polyethersulfone membranes with *p*-toluenesulfonic acid and poly vinyl pyrrolidone additives have been prepared that can be widely applied in various biomedical fields [57-58]. Recent trends show the sol-gel conversion process can be studied through the EPR techniques and the high performance single layer antireflection coatings can be extensively used in solar energy applications, enhanced photoelectrochemical and photoactive catalytic processes [59-61].

VI. Conclusions

Sol-Gel method with metal alkoxide is very suitable method for preparation of glasses, powders, ceramics and thin films at low temperature. Silica materials blended with biocompatible polymers are applied in various pharmacological fields. Silicon encapsulated within the nanofibrils of PVP derivatives has certain bioclinical properties. Materials derived from sol-gel technology blended with the requisite metal ions can derive into a product with desirable properties and thereby enhancing the broad spectrum of applications.

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