International Archive of Applied Sciences and Technology
Int. Arch. App. Sci. Technol; Vol 5 [1]March 2014: 01- 05
© 2014 Society of Education, India
[ISO9001: 2008 Certified Organization]
www.soeagra.co/iaast.html

IAAST
ONLINE ISSN 2277- 1565
PRINT ISSN 0976 - 4828

CODEN: IAASCA ORIGINAL ARTICLE

Synthesis and Study the Effect of H_2O/TEOS ratio of the Silica xerogel by Sol- Gel method

Seenaa Ibrahem , Harith Ibrahem
Deprtment of Physics ,College of Science, University of Baghdad
Email: Sinna633@gmail.com, Harith ibrahem@yahoo.com

ABSTRACT
In this research, preparation of silica xerogel by sol-gel method using precursor solutions were made by mixing tetraethylorthosilicate (TEOS), ethanol , water and HCL . The sample prepared by mixing TEOS,ethonal,water,and HCL ,TEOS and ethonal were mixing and stirred for 10 min at room temp.then 0.1 MHCL was gradually added to the solution until a water to TEOS molar ratio of R=2,R=5. The silica xerogel characterized using infrared spectroscopy (FTIR), thermal gravimetric analysis(TGA),and micro hardness .The results shows that, FTIR spectroscopy of silica xerogel two region, one region the band Si-O-Si at 450-800 $cm^{-1}$and the two region O-H at 1500-3600cm^{-1} .TGA of silica xerogel Aera under the curve represents the amount of energy needed to get ride of the water,in the case R=5 the amount of water absorbed more would need more energy to be evaporating . Microhardness, Vickers hardness (H_v) the sample R= 5 has been given higher hardness values of R=2 and the horizontal given hardness higher than the vertical position.
Keywords: silica xerogel,TEOS,sol-gel,FTIR,TGA,microhardness

Received 09/10/2013 **Revised 12/12/2013** **Accepted 23/02/2014**

INTRODUCTION

Sol-gel is One of the methods for the preparation of silica xerogel materials [1] The sol–gel technology 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 new materials with controlled surface property and shape and pore structures[2]. " The method includes the following steps: preparation of the solution, mixing the solution , gelation, aging and finally drying of synthesized materials"[3]. Various works has been reported in literature on sol–gel processes for manufacturing glasses[4] , inorganic materials and organic –inorganic hybrids[5] . A *sol* is a colloidal suspension of solid particles in a liquid. A *colloid* can be defined as a suspension in which the dimension of the dispersed phase is so small (1-1000 nm) that gravitation forces can be neglected and interactions are dominated by short-range forces (van der Waals or surface charges). A suitable definition of a *gel* is a two-component system that consists of a continuous solid and fluid phase of colloidal dimensions. The sol-gel process thus involves the formation of a colloidal suspension (*sol*) and further polymerization of the sol to form an inorganic network in a continuous liquid phase (*gel*)[6]. A porous and amorphous structure are one of the most characteristic features of sol-gel derived silica xerogel. Reactivity of the matrix, due to free hydroxyl groups, is the other typical property of silica xerogel. The microstructure of silica xerogel can be controlled by changing the water/alkoxide molar ratio, the catalyst type or concentration, by consolidating /sintering the silica xerogel by heat treatment or by using alkyl-substituted alkoxides or other additives [7]. Silica is an interesting ceramic material in the field of materials science due to its low cost, availability and particular properties. Natural and synthetic silica has been used in the industry and engineering due to its interesting optical properties and other features such as thermal and electrical insulation, hardness and chemical stability[3]. A xerogel is formed by the solvent evaporation from the wet gel, resulting in collapse of the wet-gel structure. If the network is compliant, the gel deforms because of capillary forces generated by the liquid. Supercritical drying or ambient pressure drying of the wet gels result in aerogels. Aerogels are actually nanoscale mesoporous materials of low density and high surface area[8]. The objective of this Study the effect of H_2O/ TEOS ratio(R) of thermal stability and distribution of solidification surface for silica xerogel .

EXPERIMENTAL PART

Synthesis of silica xerogel monolithic Samples:-

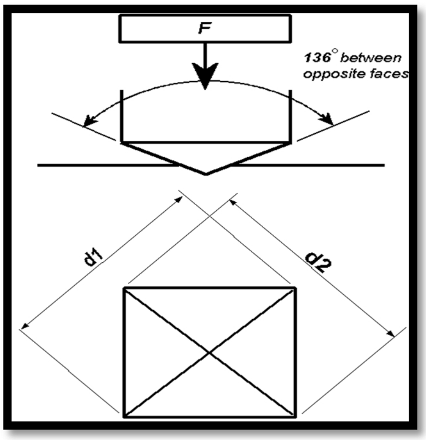
sol-gel solutions were prepared by mixing tetraethylorthosilicate (TEOS, ≥98% purity), ethanol, water and HCl. Equal volumes of tetraethylorthosilicate (TEOS) and ethanol were mixed and stirred vigorously for 10 min at room temperature. Then 0.1M HC1 was gradually added to the solutions, until a water to TEOS molar ratio of R=2 was attained. Additional deionised water was added to give solutions with R =5 so that for all solutions the molecular ratio TEOS:HCI was maintained, as summarised in Table 1. The solutions were placed in the refluxing bath immediately after mixing, and the temperature of the bath was increased to 60°C in 15 min, while stirring, and kept there for 2 h and then cooled to room temperature. drying the sample at 100◦ c

Table 1:-Reagent volumes used in sol preparation

Sample (R ratio)	TEOS ml	ethanol ml	H20 ml	HCI (0.1M) ml
2	5.0	5.0	10	0.81
5	5.0	5.0	25	0.81

Many tests were carried out to the sample :-

1. Mid-IR spectra, from 4000 to 400 cm^{-1}, were obtained for some pure samples using FTIR-Spectrometer, supplied by Shimadzu, on KBr pellets of the samples.
2. TGA use helium as inert gas in rate 20ml/min at temperature range of 0 to 1000◦ Cat a heating rate of 10◦ C/min. the samples were ground into fine powder .the measurements were taken using 3-5mg samples.
3. Micro hardness tester (Adolph I. Buehler Inc. USA) for Vickers hardness (HV) measurements of the prepared samples,an indent time 45sec and the load 500g .



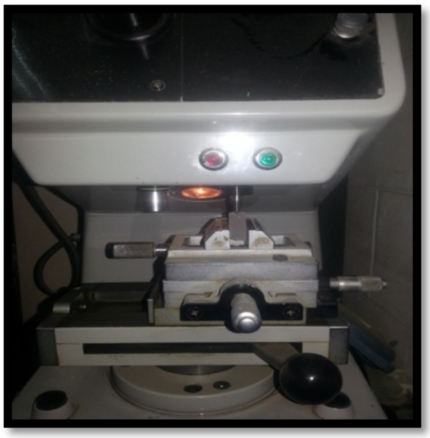


Fig. (1) micro hardness tester

RESULTS AND DISCUSSION

Fig.(1a) shows FTIR spectra of TEOS solution which agree with smith,A.L.[9] ,and fig.(1b,c) shows FTIR spectra of silica xerogel typically exhibits peaks in two distinct regions peaks at 3600 cm^{-1} - 1500 cm^{-1} . The first region corresponds to hydroxyl (–OH) stretching from absorbed or molecular water, while the second region occurs due to several silica modes. While containing a number of peaks, the silica region can be primarily separated into three peaks centre at: ~ 450 cm^{-1}, ~ 800 $cm^{-1}$and a broad peak at ~ 1100 cm^{-1} . The absorption at ~ 450 cm^{-1}has been assigned to rocking motion of oxygen atoms bridging silicon atoms in siloxane bonds(Si-O-Si). The symmetric vibrations of silicon atoms in a silixane bond occur at ~ 800 $cm^{-1}$and are termed as υ as -Si-O-Si. The largest peak observed in a silica spectrum ispresent at ~ 1100 $cm^{-1}$and is dominated by antisymmetric motion of silicon atoms in siloxane bonds (Si-O-Si) for all samples (R=2 and R=5) which agree with G. E. A. Swann et al. [10] .

Fig. (2) shows TGA&DTG analyses for TEOS solution and silica xerogel R=2,R=5 at 100 ◦ C . the weight percent 51.2% at 138.77 ◦ c,the silica xerogel R=2 the weight 96.4% at 87.4◦ c and silica xerogel R=5 the weight 93.2% at 111.2◦ C .Aera under the curve represents the amount of energy needed to get ride of the water,in the case R=5 the amount of water absorbed more would need more energy to be evaporating .

Microhardness, Vickers hardness (HV) measurements of the prepared samples, Table (1) an indent time 45sec and the load 500g . We take two each sample a vertical position and the horizontal , the sample R=

2has been given higher hardness values of R=5 and the horizontal given hardness higher than the vertical position.The sample of R=2 are hard than the sample of R=5, leads to increase the intensity of the cross link between the bonds of the materials and by following the increase the hardness of materials.

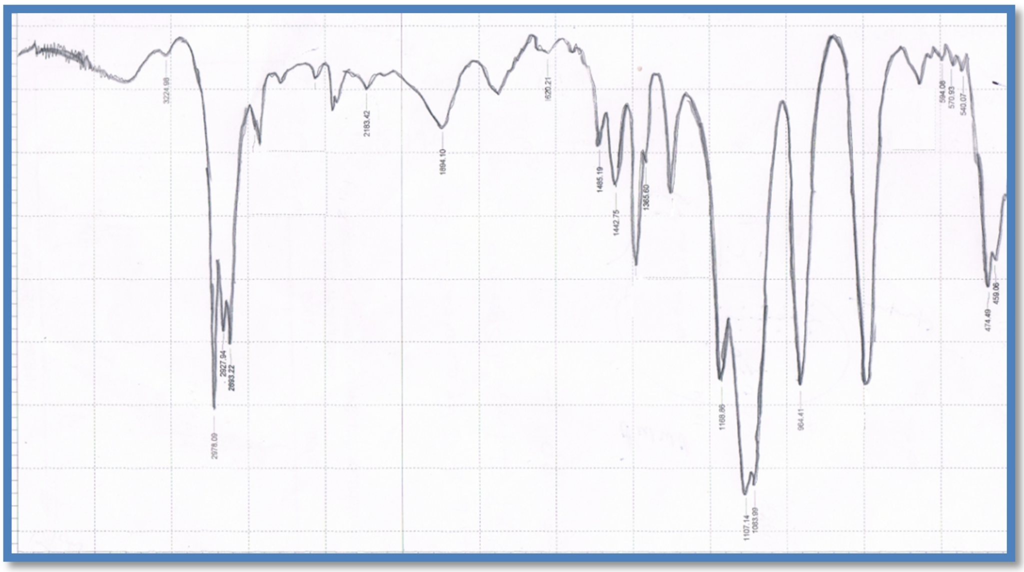


Figure (1a)FT-IR spectrum of the TEOS solution

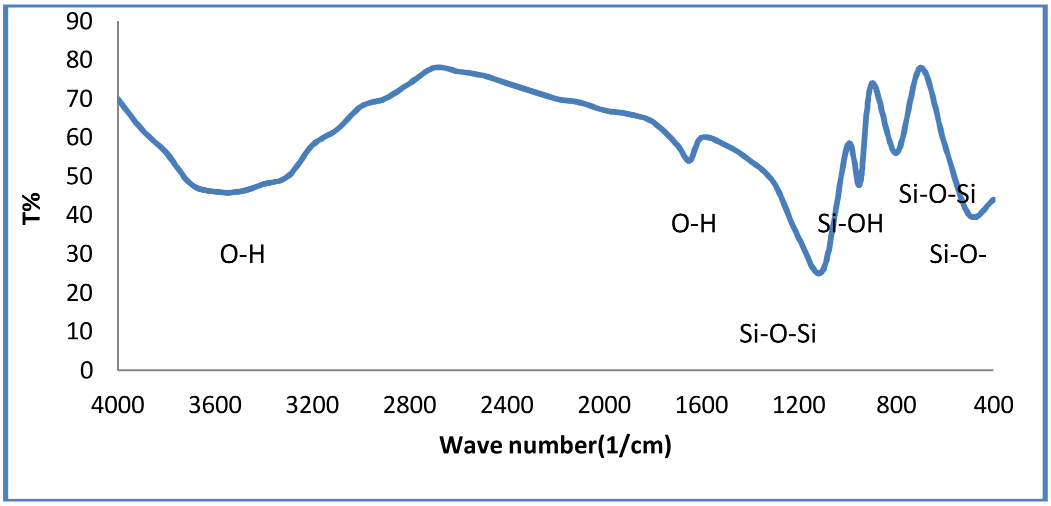
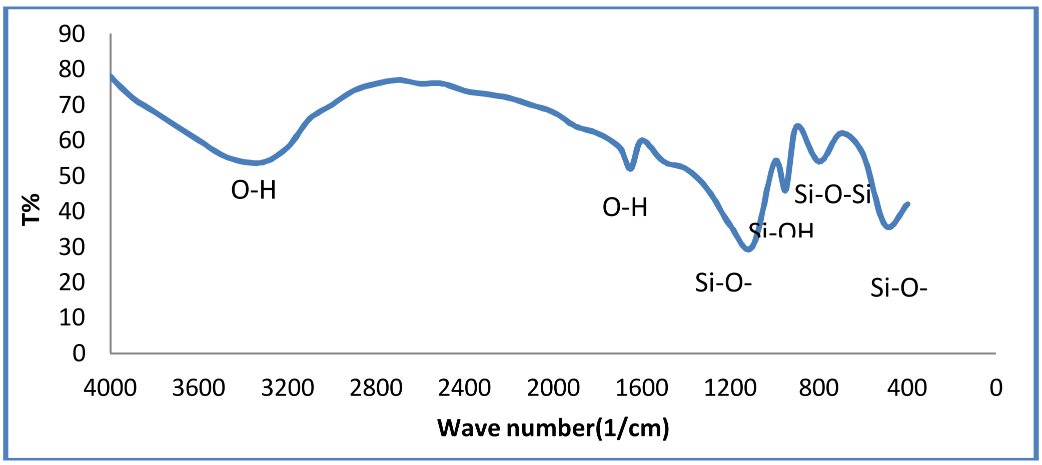
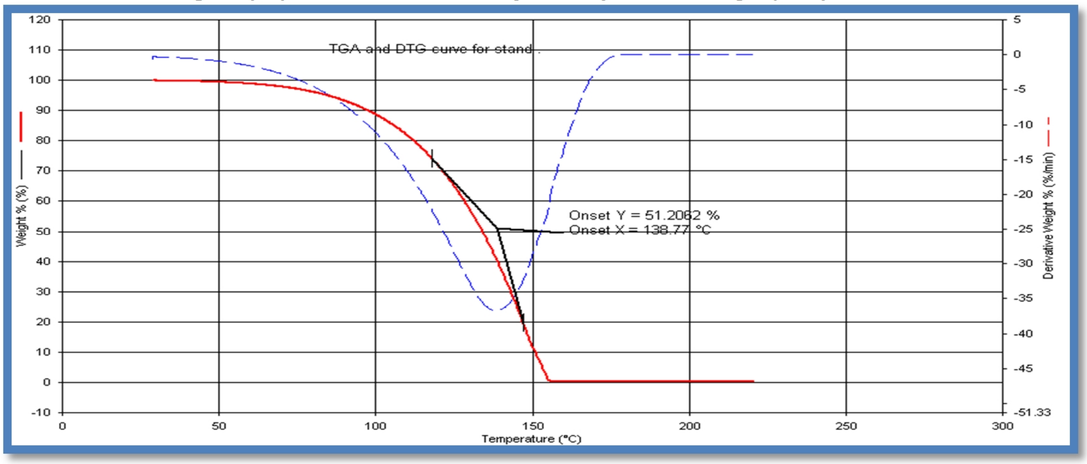


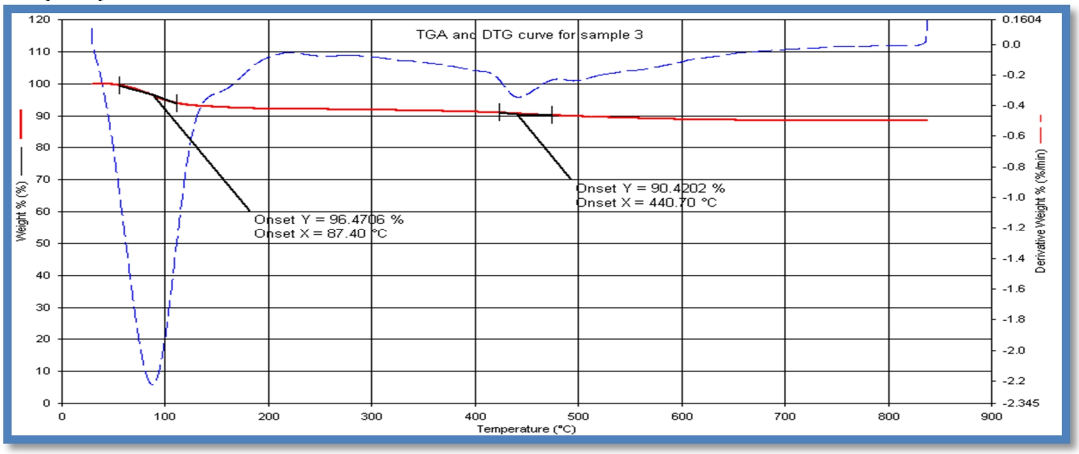
Figure (1b) *FTIR transmission spectra of Silica Xerogel(R=2) at100* ºC.



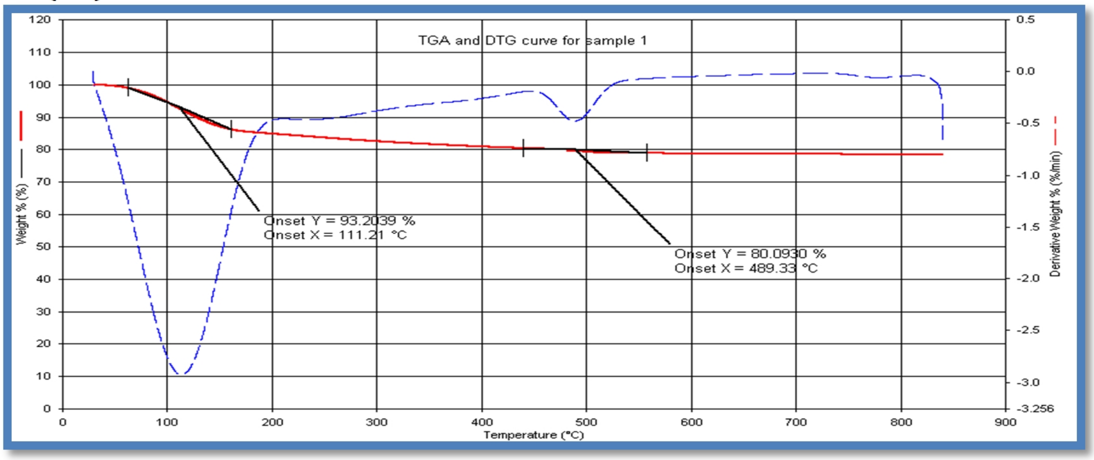
Figure(1c) *FTIR transmission spectra of Silica Xerogel(R=5) at 100°c*



(a)



(b)



(c)

Fig.(2) TGA&DTG curve of a- TEOS b- silica xerogel R=2 c- silica xerogel R=5

Table (2) values hardness for all sample

	Load (gm)	Hv (GPa)
R=2,vertical	500	3.19
R=2,horizontal	500	4.03
R=5,vertical	500	2.43
R=5,horizontal	500	3.7

CONCLUSIONS

1- The samples of case R=5 the amount of water absorbed more than the samples of case R=2 ,would need more energy to be evaporating .
2- The sample of case R=2 are hard than the sample of case R=5, leads to increase the intensity of the cross link between the bonds of the material and by following the increase the hardness of material.

REFERENCES

1. Szczepan Bednarz, Barbara Ryśand Dariusz Bogdał.(2012)." Application of Hydrogen Peroxide Encapsulated in Silica Xerogels to Oxidation Reactions", Molecules 17, pp.8068-8078..
2. Fozia Z. Haquea, Vazid Ali , M. Husain(2013). "Synthesis and spectroscopic characterization of Nile Blue doped silica gel rods" ,journal of Elsevier,Optik 124,pp. 4287– 4291
3. Ana Catalina Duque Salazar. (2011). "Development of silica containing materials for the adsorption of organic compounds" , Universidad Nacional de Colombia.
4. Aryeh, M.W.,Tsala,S. and Renta ,R.,optical materials ,16,15-20,2001
5. Achanai Buasril,Chaitawat singpracha,Chainan junpraset,and Tawin Chotwatcharin. (2008). "Synthesis and Characterization of Sol-GelProcessed Organic/Inorganic Composite Materials"Kasetsart J. (Nat. Sci.) 42 : 367 – 372.
6. Sinaa ibrahem,harth ibrahem. (2013). " Preparation and study properties of xerogel silica using sol-gel method ", International Journal of Application or Innovation in Engineering & Management (IJAIEM), Volume 2, Issue 9.
7. Pirjokortesuo" (2001). sol-gel derived silica gel monoliths and microparticles as carrier in controlled drug delivery in tissue administration ", Division of iopharmaceutics and Pharmacokinetics Department of Pharmacy University of Helsinki.
8. Tomasz Błaszczyński , Agnieszka Ślosarczyk.(2009). Maciej Morawski "Synthesis of Silica Aerogel by Supercritical Drying Method", rs. Published by Elsevier, Procedia Engineering 57 ,pp. 200 – 206,201
9. Smith,A.L."The coblentz society desk book of infrared spectra "second edition,pp.1-24.
10. G. E. A. Swann and S. V. Patwardhan .(2011)." Application of Fourier Transform Infrared Spectroscopy (FTIR) for assessing biogenic silica sample purity in geochemical analyses and palaeoenvironmental research", Clim. Past, 7, pp.65–74.

Ciation of this article
Seenaa Ibrahem , Harith Ibrahem. Synthesis and Study the Effect of H_2O/TEOS ratio of the Silica xerogel by Sol- Gel method. Int. Arch. App. Sci. Technol; Vol 5 [1]March 2014: 01-05