CHAPTER 1
INTRODUCTION

1.1 OVERVIEW

The topic of the research project is Studies on the Gelation Kinetic of Silica Sol. The keywords from the topic are gelation kinetic, silica sol and gel. Gelation kinetic means the motion of particles during a process of forming gel from a particular solution (liquid). Secondly, silica sol is a colloidal suspension of silica (SO_2) particles in a liquid phase. The size of particles is 1-500μm. Sol is usually prepared by dispersion and condensation. While the term gel means a 3-D structured system that cannot flow in the steady state condition. It is identified as liquid in term of mass but behave like solid.

Silica sol belongs to colloid. Since colloid grains are very small (10-20 nm) and have large surface area, so the natural colour of the materials to be covered is not changed. It is soluble in water but insoluble in organic solvent. If the viscosity is low, they can soak the places and holes that water soaks. The dispersion and permeability are good when silica sol is mixed with other substance. When the moisture contained in silica sol is evaporated, the colloid grains firmly adhere to the material surface and the combination of silicon and oxygen is then formed between grains. The sticky characteristic of silica explains its potential as adhesive agent.

The first sol-gel synthesis of silica was carried out 150 years ago; it is done by J.J. Ebelmen. He observed that silicic esters (alkoxides) hydrolyze slowly in the presence of moisture to produce hydrate silica. Then, further studies were carried out in seventies and eighties to explore the potential and characteristic of silica. They found that the sol-gel synthesis of oxide materials occurs at temperature lower than usual solid-state reactions.

Today, sol-gel methods are almost reaching their full potential. For instance silica has a very bright potential today. The application of sol gel method to prepare silica can be found industry such as to make optical devices, catalyst carrier, adhesive agent, machinery parts, in paper making industry as well as for coating purposes. To extend the potential of silica, the gelation behavior as well as the properties of silica sol must be studied further.

1.2 BACKGROUND OF STUDY

Silica sol is produced from sol-gel processing. It is expected that the demand of silica gel will increase and the usage will be widened. As it is highly potential in industries, the understanding of the gelation behavior is essential in order to control the particle size of silica and the stability. The silica stability means how long silica can sustain in high or critical pressure and temperature, plus wide range of relative humidity. Literature review and research are being carried out to find the best method to prepare stable silica sol. After that, the gel formation will be indicated by refractive index value using refractometer.

1.3 PROBLEM STATEMENT

Silica sol has a very high potential application in industries. There are several techniques used to investigate the process of sol-gel. However, the kinetics of the individual reactions is still not clear and well studied. The reactions in sol gel process are hydrolysis and condensation reactions. In addition, there are several factors affect the process of making silica. Some of the factors are temperature, relative humidity and concentration. The condition during the preparation of silica sol gives different strength/ characteristic to the particular silica because the kinetic during the reaction will be different. It is desired to study how each factor affect the formation of silica particles. In this research, the method to prepare stable silica sol is the main concern.

1.4 OBJECTIVES AND SCOPE OF STUDIES

The objectives of this research are:

1. To prepare stable silica sol with different concentration.
2. To study the gelation behaviour of silica sol.
3. To study the role of different factors on the gelation behaviour of silica sol. The factors are the concentration of silica sol and the relative humidity.

Studies on silica are very wide. So, in order to fit to the timeframe which is one year time, the research area will be narrowed down. It is decided that to use acid as the catalyst to prepare silica sol instead of base. In other words, the experimental work will be focusing on the gelation behavior of acid catalyzed silica sol. Nitric acid is decided as the catalyst for all silica sol sample.

For the first objective, 5 samples with different concentration are prepared. The formation of silica gel will be observed for every 24 hours to achieve second objectives. This is done by taking the refractive index value for each sample.

To achieve third objective, a series of experimental work will be conducted. Some of them will be done by varying the relative humidity value to study the effect of this factor to the gelation behavior of silica sol during the sol gel processing.

CHAPTER 2

LITERATURE REVIEW

2.1 THE DEFINITION OF SOL AND GEL

A sol is a colloidal suspension of solid particles in a liquid. The size of sol particles is between 1 – 500 nanometers. Artificial sols may be prepared by dispersion or condensation. Dispersion techniques include grinding solids to colloidal dimensions in a ball mill (dry or wet). Condensation or precipitation methods work by making the colloidal particle come out of solution into the colloidal phase, either by adding a precipitating agent (salt) or by changing the temperature. The stability of sols may be maintained by using dispersing agents such as ethanol.

Sols are commonly used in preparing sol-gels. Sols may be a component of total dissolved solids, since the particle size is small enough to pass through a two micrometer filter.

Gel means a dilute cross-linked system that cannot flow when it is in steady state. It is a solid, jelly-like material that is having properties ranging from soft and weak to hard and tough. Before a mixture is called gel, it is initially liquid. So, a gel is also liquid in term of mass except that they behave like solid because of its 3-dimensional cross-linked network. This network causes the structure and stickiness of gel.

2.2 SOL-GEL PROCCESING

A colloid is a suspension while a sol is a colloidal suspension of solid particles in a liquid. In sol-gel process, the starting compound to prepare a particular colloid is called precursors. Common precursors for aluminum oxide include inorganic salt such as $Al(NO_3)_3$ and organic compounds such as $AL(OC_4H_9)_3$. Metal alkoxides are members of the family of metalorganic compounds, which have an organic ligand attached to a metal or metalloid atom. The most studied is silicon tetraethoxide or tetraethoxysilane or tetraethyl orthosilicate, TEOS. (Brinker and Scherer, 1990).

Metal oxides react readily with water making them the most popular precursors. The reaction is known as hydrolysis as water is added. The sol-gel material is prepared by sol-gel processing is observed to encompass a wide range of inorganic and organic composite material such as ceramic and glass.

The interest of sol-gel processing of silica sol began in the mid-1800s by Ebelmen and Graham. They observed that the hydrolysis of TEOS under acidic condition yields SiO_2 in the form of glass-like material. (Hench and West, 1989)

Generally, sol-gel processing consists of two reactions which are hydrolysis and condensation. The hydrolysis of a silicon alkoxide can occur either by acid-catalyzed or base-catalyzed process. It is a reaction where hydroxyl group ions attached to the metal atom, as follows:

$$Si(OR_4) + H_2O \rightarrow HO\text{-}Si(OR)_3 + ROH \qquad (1)$$

R is a proton or other ligand.

Hydrolysis may be completed (all OR groups are replaced by OH) depending on amount of water and catalyst present, as follows:

$Si(OR_4) + 4H_2O \rightarrow Si(OH)_4 + 4ROH$ (2)

Tetraalkoxysilanes, organotrialkoxysilanes and diorganodialkoxysilaneshydrolyze undergo hydrolysis upon exposure to water vapor. And hydrolysis may stop when the metal only partially hydrolyzed, $Si(OR)_{4-n}(OH)_n$.

Isoelectric point of silica is when equilibrium species has zero net charge is at pH 2.2. The effect of catalyst that is either acid or base may be judged by comparing the rate of reaction at different pH values. This comparison is a simple way of measuring relative hydrolysis rate because it includes both hydrolysis and condensation. The gelation point (t_{gel}) indicates the relative rates. (Wright and Sommerdijk, 2003)

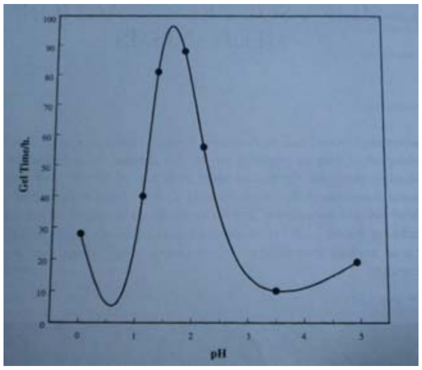

Figure 2.1: Gel Time as a Function of pH for HCl Catalyzed TEOS
(Wright and Sommerdijk, 2001)

From the graph, at point pH is 2.2 (isoelectric point of silica sol), the gel time is the highest. At this point also, the condition of acid or base is said to be decrease.

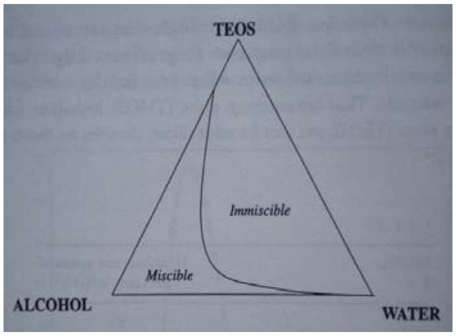

Figure 2.2: The Phase Diagram for TEOS/Ethanol/Water
(Wright and Sommerdijk, 2001)

Condensation reaction is classified as either water condensation or alcohol condensation. During the reaction, 2 hydrolyzed molecules will link together (water condensation) such as:

$$(OR)_3Si\text{-}OH + HO\text{-}Si(OR)_3 \rightarrow (OR)_3Si\text{-}O\text{-}Si(OR)_3 + H_2O \qquad (3)$$

Or

$$(OR)_3Si\text{-}OH + HO\text{-}Si(OR)_3 \rightarrow (OR)_3Si\text{-}O\text{-}Si(OR)_3 + ROH \qquad (4)$$

For alcohol condensation, the reaction is:

$$Si(OEt)_3OR + HOSi(OEt)_3 \leftrightarrow (EtO)_3SiOSi(OEt)_3 + ROH \qquad (5)$$

The relative rates of reaction of different species depend on steric effect and the charge on the transition state. For acid hydrolysis, with positively charged trasition state stabilized by electron donating groups $(RO)Si(OH_3)$ condensed faster than $(RO)_2Si(OH)_2$.

This means that for acid catalyzed reaction, the first step of hydrolysis is fastest. Also, the product will undergo fastest condensation. Then, open network results followed by further hydrolysis and cross-condensation reaction.

In base catalyzed conditions the negatively charged transition state becomes more stables as more hydroxy groups replace the electron donating alkoxy group (Wright and Sommerdijk, 2001). As a result, successive hydrolysis steps occur rapidly, and the fully hyrolysed species undergoes the fastest condensation. It forms high cross-linked large sol particles and large pores between interconnected particles.

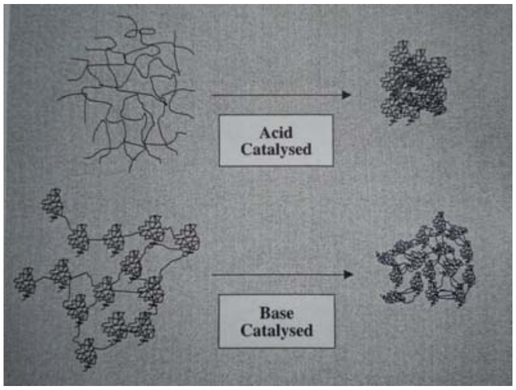

Figure 2.3: Gel Structure for Acid and Base Catalyzed Reaction
(Wright and Sommerdijk, 2003)

Table 2.1:The advantages and disadvantages of the sol-gel method.

Advantages	Disadvantages
1. Better homogeneity from raw materials	1. High cost of raw materials
2. Better purity from raw materials	2. Large shrinkage during processing
3. Lower temperature of preparation (save energy, minimize evaporation losses, minimize air pollution, no reactions with containers, bypass phase separation, bypass crystallization)	3. Residual fine pores
4. New noncrystalline solids outside the range of normal glass formation	4. Residual hydroxyl
5.New crystalline phases from new noncrystalline solids	5. Residual carbon
6. Better glass products from special properties of gel	6. Health hazard of organic solution
7. Special products such as films	7. Long processing time

2.3 GELATION

The hydrolysis and condensation reactions will lead to the growth of cluster that collide and link together to form gel. Weak and strong bonds connecting the solid phase are a matter of time scale. Usually, the bond is to be said strong if it is permanent and weak when the bonding is reversible.

The gelation time is easy to be observed qualitatively but difficult to measure it analytically. When the particles grow and collide, condensation is happening and microparticles formed. The sol is said to be gel when it can support a stress elastically. This point also known as gelation point. As there is no activation energy than can be measured, no one can precisely define the point. This grow will continue till more particles are interconnected. (Hench and West, 1989)

Brinker and Scherer highlighted that the sharp increase in viscosity that accompanies gelation essentially freezes in a polymer structure at the gel point. The resulted structure is affected by time, temperature, solvent, pH condition or upon removal of solvent. (Brinker and Scherer, 1990)

To measure t_{gel}, the most precise method proposed by Sacks and Sheu may be used. The viscoelastic response of gel as a function of shear rate is measured.
The complex shear modulus,G is measured by using viscometer with a narrow gap.

The equation is:

$$G = G'(\omega) + iG''(\omega) \tag{6}$$

Where G = the complex shear modulus
G' = storage modulus
G''= loss modulus
ω = frequency

The relative measure of the viscous energy losses to the energy stored in the system is defined as the loss tangent:

$\tan \delta = G'' / G'$

The rapid increase in the storage modulus near t_{gel} indicates that interconnection of particles become more sufficient to support load elastically.

This initial theory was developed by Flory and Stockmayer. It is called as Classical or Mean Field Theory of Gelation. Their theory is to account for the gel point and the molecular weight distribution in the sol. They addressed the question of what fraction of all the possible bonds that could form in a polymerizing system actually need to be form before infinite large molecules appears. (Wright and Sommerdijk, 2001)

The basis structure by Flory is the Cayley tree or also called as Bethe lattice. In the Cayley tree, the functionality or maximum number of bonds, z, that are allowed to form at each bond site is used to calculate the probability of bond forming at each site, P:

$$P = \frac{\text{Number of node bonds}}{\text{Total number of node bonds}} \qquad (7)$$

$P = n/(Nz)$

Where n = number of node bonds

N = number of sites

z= functionality or maximum number of bonds

When the cluster is continuously connected from one side to another, the system is forming a gel structure. There must be at least 2 connections per node in order for cluster to be a gel. This leads to critical probability, P_c :

$P_c = 1/2$

Or $P_c = 1/(z-1)$

The other equation related to this theory is calculating the extent of reaction, p_c . The critical extent of reaction, p_c for the gel point to be formed is given by:

$$p_c = \frac{1}{N-1} \approx \frac{1}{N} \tag{8}$$

The critical extent of reaction for gelation, p_c also can be determined as a function of the properties of the monomer mixture, r, ρ, and f.

$$p_c = \frac{1}{(r + rp(f-2))^{0.5}} \tag{9}$$

This theory raised few questions such as of all the bonds that could form in a polymerizing system, what fraction P_c must form before infinitely large molecules appears. The first assumption we make is that the reactivity of all the functional group on a monomer is equal, means the probability that any particular bond has formed is equal to p. (Brinker and Scherer, 1990)

However, this theory however neglects the formation of closed loops within the growing clusters, leading to unrealistic predictions about geometry of polymers.

There is also another theory that is known as Percolation theory. Percolation concerns on the movement and filtering of fluids through porous materials such as sol. Percolation theory offers a description of gelation that does not exclude the formation of closed loops and so does not predict a divergent density for large cluster. The disadvantage of the theory is that it generally does not lead to analytical solutions for such properties as the percolation threshold or the size distribution of polymers. However the features can be determined with great accuracy from computer simulations, and the result often quite different from the predictions of the classical theory. (Brinker and Scherer, 1990)

2.4 KINETIC MODELS

The well-known kinetic model is Smoluchowski coagulation equation. It is an integrodifferential equation introduced by Marian Smoluchowski in a seminal 1916 publication, describing the evolution of the number density of particles of size x at a time t. In the continuous case the equation is

$$\frac{\partial n(x,t)}{\partial t} = \frac{1}{2}\int_0^x K(x-y,y)n(x-y,t)n(y,t)\,dy - \int_0^\infty K(x,y)n(x,t)n(y,t)\,dy. \tag{10}$$

If dy is interpreted as a discrete measure then the discrete form of the equation is recovered:

$$\frac{\partial n(x_i,t)}{\partial t} = \frac{1}{2}\sum_{j=1}^{i-1} K(x_i-x_j,x_j)n(x_i-x_j,t)n(x_j,t) - \sum_{j=1}^{\infty} K(x_i,x_j)n(x_i,t)n(x_j,t). \tag{11}$$

The operator, K, is known as the coagulation kernel and describes the rate at which particles of size x coagulate with particles of size y. Analytic solutions to the equation exist when the kernel takes one of three simple forms:

$$K = 1, \quad K = x + y, \quad K = xy,$$

known as the constant, additive, and multiplicative kernels respectively. However, in most practical applications the kernel takes on a significantly more complex form, for example the free-molecular kernel which describes collisions in a dilute gas-phase system,

$$K = \sqrt{\frac{\pi k_b T}{2}}\left(\frac{1}{m(x)} + \frac{1}{m(y)}\right)^{1/2}\left(d(x) + d(y)\right)^2. \tag{12}$$

Generally the coagulation equations which result from physically realistic kernels are contradicts. To avoid that, it is necessary to appeal to numerical methods. The other good method is to use only one property of particle (x). In the multi-variables case, when two or more properties (such as size, shape, composition etc.) are introduced, it is become more complicated and the alternative to be used is Monte-Carlo method.

CHAPTER 3
METHODOLOGY

3.1 RESEARCH METHODOLOGY

3.1.1 Preparation of Silica Sol by Using Nitric Acid as the Catalyst

The preparation of silica sol is done by adding 4 solutions which are tetraortho silicate, nitric acid, ethanol as the solvent and water. All apparatus such as volumetric flasks must be clean and dried. For instance, 5 volumetric flasks are prepared to make 5 different concentration of silica sol. Number them and label them with sticky papers.

Preparing 5 different solutions is done by mixing tetraortho silicate (TEOS), nitric acid, ethanol and water in 5 different beakers (at room temperature and pressure). The concentration is different as the amount of water is different for each solution. The exact amount in volume (mL) of each solution is shown in the Table 3.1 below:

Table 3.1:The amount of chemicals in each sample.

Sample	TEOS (mL)	HNO_3	H_2O	C_2H_5OH
1	10	0.5	20	25
2	10	0.5	40	25
3	10	0.5	60	25
4	10	0.5	80	25
5	10	0.5	100	25

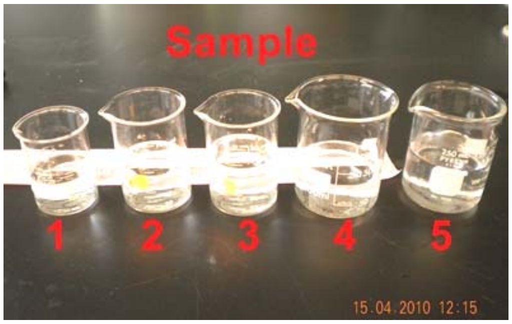

Figure 3.1: Beakers contain TEOS, HNO_3, H_2O, and C_2H_5OH

All solution is then transfer/pour into the labeled volumetric flasks so that they mixed well. The volumetric flask must be covered so that the ethanol is not vaporized.

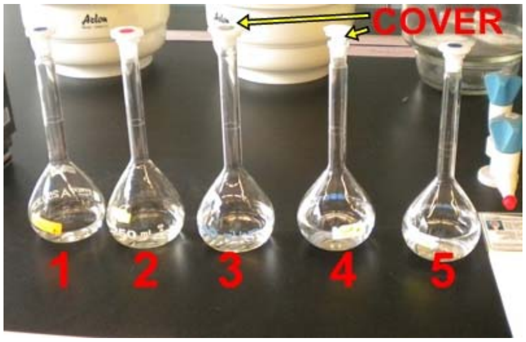

Figure 3.2: Volumetric flasks contain solution.

Keep them at a safe place under room temperature and pressure. Take refractive index readings for each sample for every 24 hours. Set the temperature of 25.0 ºC at the equipment.

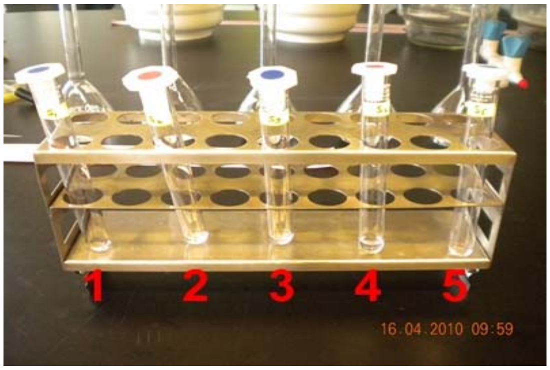

Figure 3.3: Samples taken from stock solutions.

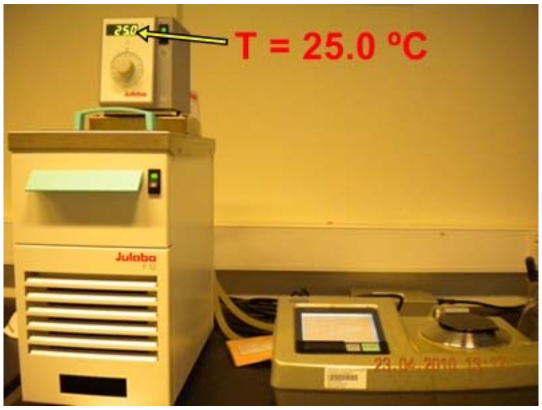

Figure 3.4: Refractometer

The data of refractive index (RI) is shown in the next chapter in Table 5.2 (under result and discussion part). After observing the RI, the samples were dried in the oven at 105.0C for 24 hour. This is done to remove all volatile components which are ethanol and water. The weight of samples before and after the drying process is shown in Table 5.2. Then, grinding process was carried out to get smaller and fine particles.

3.2 PROJECT ACTIVITIES

1. Literature Review

Literature review is carried out from January till now. The source of information is from textbooks, journals, online resources and websites. Literature reading is essential so that student become familiar with the process and terms used from the earlier studies by others.

2. Meeting and Discussions with Supervisor

Discussion is carried out to enhance the understanding of student on sol-gel processing. Supervisors suggested the student to read book and find journals to be read. Co-supervisor is also assisting the student to understand the reading materials and clarify the process of sol-gel and data collected by other journalists.

3. Lab Works

Several lab sessions will be carried out to study the gelation kinetic of silica sol. There are 2 experiments in this research:

1. Preparation of silica sol samples with different concentration.
2. The effect of the relative humidity on the silica sol kinetic.

3.3 LIST OF TOOLS AND EQUIPMENTS

The tools and equipment that are identified till now:

1. Test tubes
2. Beakers
3. Volumetric flaks
4. Graduated cylinders
5. Pipettes
6. Refractometer
7. Magnetic Stirrer
8. Oven
9. Dialyzer
10. Descistor
11. Agetatemortar

3.4 LIST OF CONSUMABLES REQUIRED

There are 5 chemicals including distilled water to be used to prepare silica sol. The ammonia solution and hydrochloric acid will not be used as the student is advised to use nitric acid as the catalyst to narrow down the research project. Also, the hydrogen fluoride solution is added to observe the effect of fluoride ion to the silica sol gelation behaviour. List of the chemicals and consumables:

1. TEOS, Tetraethoxysilane
2. Nitrate acid
3. Ethanol
4. Distilled water
5. Sulphuric Acid

CHAPTER 4

RESULTS AND DISCUSSION

4.1 RESULT OF REFRACTIVE INDEX (RI) AT T = 25.0 °C AND P = 1 atm

Up to this point, silica sols with different concentrations were prepared. After 24 hours, the reading of refractive index is taken for each sample. The result obtained for every 24 hours is recorded. The objectives of doing this are:

1. To observe the pattern of refractive index value from day 1 to day 9
2. To compare the refractive index value for sample 1 until sample 5. We will be able to observe how concentration of silica sol affecting the refractive index value that actually indicates the formation of silica particles.

The result in table 4.1 below shows the refractive index value for 5 different samples from day 1 to day 9.

Table 4.1:Result of refractive index for 5 samples.

	Sample No.				
Time (day)	**1**	**2**	**3**	**4**	**5**
1	1.36582	1.35885	1.35456	1.35111	1.34807
2	1.36649	1.35900	1.35326	1.35176	1.34936
3	1.36681	1.35966	1.35539	1.35234	1.35003
4	1.36688	1.36076	1.35618	1.35247	1.35001
5	1.36475	1.36704	1.35601	1.35206	1.35006
6	1.36706	1.36107	1.35632	1.35245	1.34997
7	1.36708	1.36111	1.35635	1.35251	1.35003
8	1.36695	1.36112	1.35636	1.35250	1.35007
9	1.36703	1.36101	1.35616	1.35192	1.34988

In order to observe the pattern of refractive index with respect to time, graph of refractive index versus time is plotted. Referring to Graph 4.1, line that connects all 9 points for each sample clearly shows the pattern of the reading from the first day till the day 9.

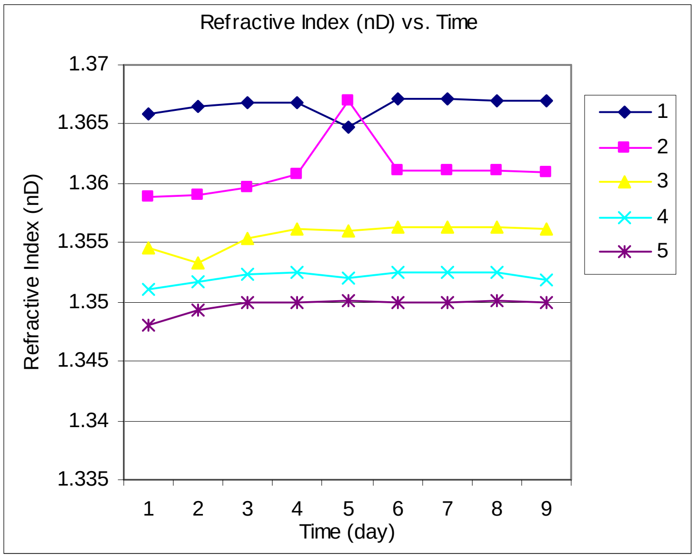

Figure 4.1: Graph 1 of Refractive Index vs. Time

For sample 1 and 2, initially the y-axis value increases directly with x-axis value. Meaning, the refractive index is increasing with respect to time. However at day 4, refractive index for sample one suddenly drops while refractive index for sample 2 increased significantly. For sample 3, the second reading is lesser than the first reading, and after that the reading increases with respect to time. For sample 4 and 5, the reading is increases from time to time but the increase is insignificant. This is because sample 4 and 5 is less concentrated compared to sample 1, 2 and 3.

The Graph 1 above is modified become Graph 4.2.The trend lines are plotted. Observing the trendlines from second graph, all shows straight lines with positive slope. It means that refractive index increases with respect to time for all 5 samples of silica sol. The 3 points each from sample 1, 2 and 3 are considered as outliers because they deviate from trend. By removing/ ignoring outliers (refer Graph 4.2), it is observed that the refractive index is increasing with respect to time. The refractive index is increasing as the hydrolysis reaction is ongoing; more solid particle is formed with respect to time. Refractive index measures the resistance that is encountered by light when travels through a particular substance. When the RI value is high, it means the speed of light pass through the substance is slower compared to in vacuum. This is because; the substance has high permittivity that resists the light from passing through.

In a simple mathematical understanding:

$$\mathbf{RI},\ \boldsymbol{\eta} = \frac{\text{velocity of light in a vacuum}}{\text{velocity of light in medium}} = \frac{\mathbf{c}}{\boldsymbol{\nu}_{\mathbf{p}}} \tag{13}$$

$$\mathbf{RI},\ \boldsymbol{\eta} = (\boldsymbol{\varepsilon\mu})\ \hat{}\ ^{1/2}$$

Where:

ε is the permittivity of a medium

μ is the relative permeability

In most cases, the μ is nearly 1 at optical frequency.

Then the equation become $\mathbf{RI},\ \boldsymbol{\eta} = (\boldsymbol{\varepsilon})\ \hat{}\ ^{1/2}$

This equation means the RI is depending on the permittivity value. As the permittivity increases, the RI also increases. It makes sense as permittivity is measuring how much resistance is faced by light when forming an electric field in a vacuum. As more resistance is encountered, the speed of light will be slower while the speed of light in vacuum remains constant making the value of RI increase.(Wikipedia, 24th August 2010)

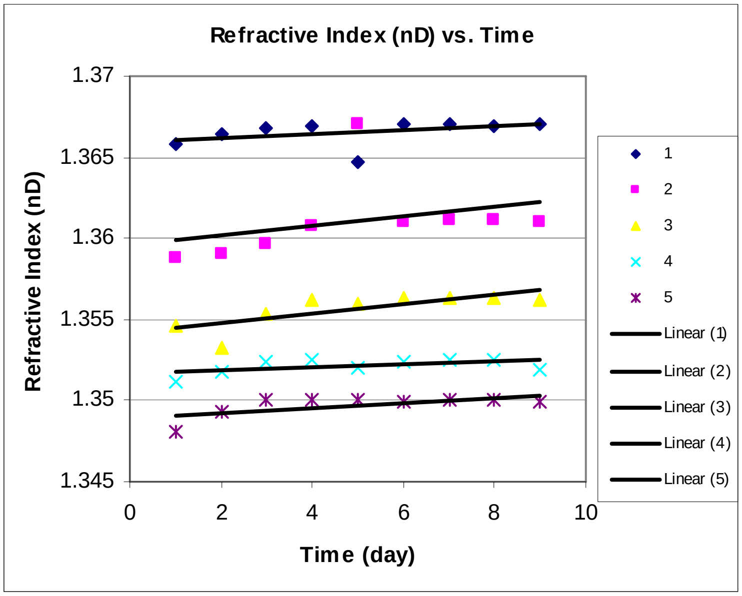

Figure 4.2: Graph 2 of Refractive Index vs. Time (observe trendlines)

After one approximately one month, Sample 1 is in gel form. After that sol Sample 2, 3 and 4 became totally gel. It is approximated that 1 month, 2 months, 3.5 month and 5 months is taken for the liquid to become totally gel for Sample 1, 2, 3 and 4 respectively. However, after 6months, Sample 5 is observed as a mixture of liquid and gel.

The fist one, Sample 1 is the most viscous compared to other at the moment before drying process took place. Table 4.2 below shows the percent of silica that was formed from the sol-gel process.

Table 4.2: Weight of the samples (before and after drying process)

SAMPLE	WEIGHT, grams		Percent of Silica formed (%)
	Before	After	
1	9.07	0.59	6.50
2	18.21	0.80	4.39
3	19.32	0.61	3.16
4	28.59	0.75	2.62

Sample 1 shows the highest silica formation which is 6.50 %, followed by Sample 2 with 4.39%, Sample 3 with 3.16% and finally Sample 4 with lowest percentage of

2.62%. From this graph, it is clearly shows that the highest amount of TEOS in the liquid results in the biggest amount of silica formed. For this case, 18.018% of TEOS give high yield of silica particles.

4.2 RESULTS OF REFRACTIVE INDEX (RI) AT DIFFERENT RELATIVE HUMIDITY (RH)

The same methodology is used to prepare 3 samples of silica sol. Each sample has different composition of TEOS, ethanol, nitric acid and water. After the preparation at room temperature, each sample is divided into 3, each in different descistor. The 3 descistors is having 16.1 %, 66.0% and 95.6% relative humidity (RH) respectively. The objective of varying the RH is to observe how RH affects the sol-gel process. Also, we wanted to observe the best composition of silica sol for this case. Table 4.3 shows the result for the experiment.

Table 4.3: Data of Refractive Index (different concentration and relative humidity)

Sample	**Time (day)**	**Relative Humidity (RH), %**		
		16.1	**66.0**	**95.6**
S1 (18.018 % TEOS)	**0**	**1.36734**	**1.36734**	**1.36734**
	1	1.36704	1.36679	1.36582
	2	1.36678	1.36647	1.36565
S2 (13.245 % TEOS)	**0**	**1.36174**	**1.36174**	**1.36174**
	1	1.35897	1.35932	1.35685
	2	1.35706	1.35810	1.35857
S3 (10.471 % TEOS)	**0**	**1.36163**	**1.36163**	**1.36163**
	1	1.35362	1.35371	1.35425
	2	1.35146	1.35341	1.35384

Graphs are plotted to observe the pattern of RI clearly. They are presented in Figure 4.3, Figure 4.4 and Figure 4.5:

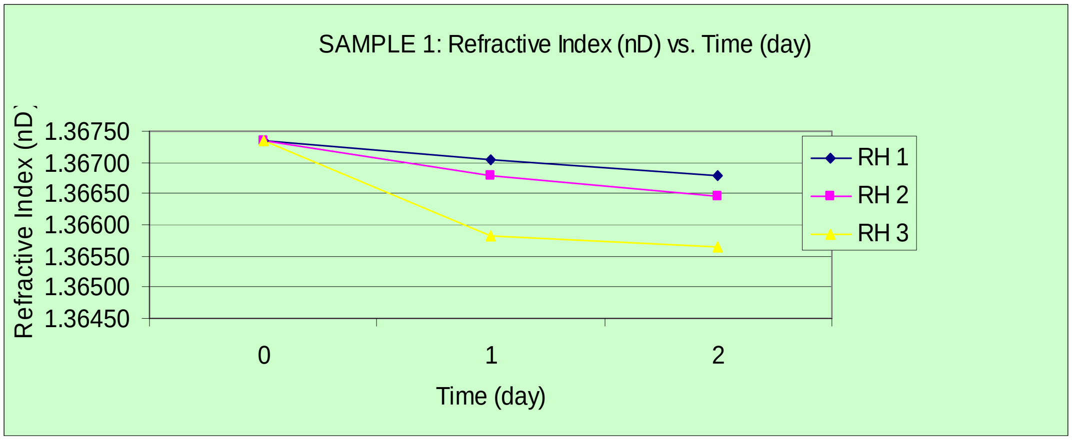

Figure 4.3: Graph of Refractive Index vs. Time for Sample 1 at 3 RH.

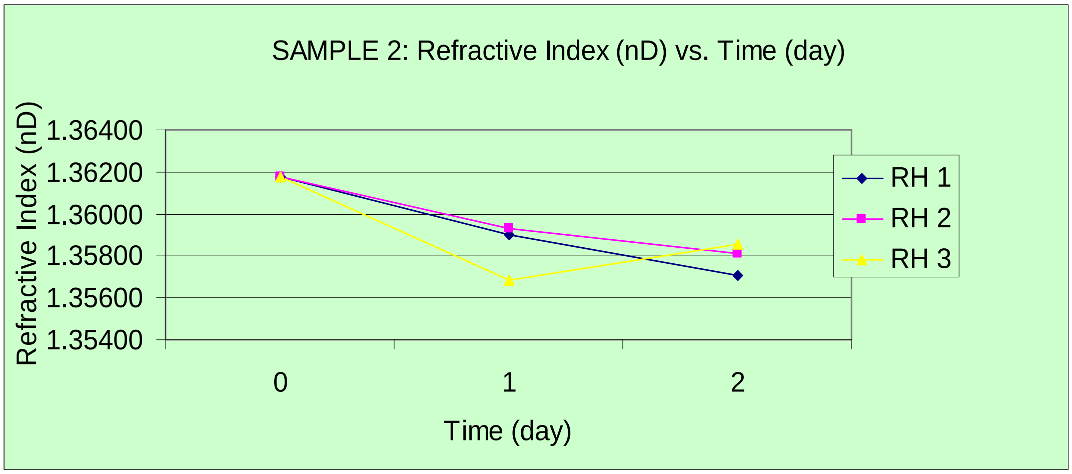

Figure 4.4: Graph of Refractive Index vs. Time for Sample 2 at 3 relative humidity.

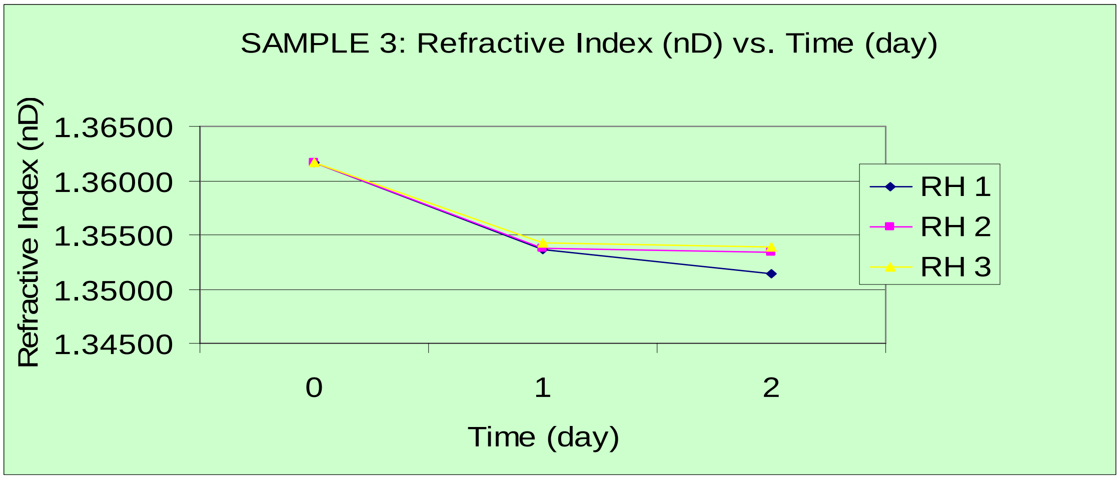

Figure 4.5: Graph of Refractive Index vs. Time for Sample 3 at 3 relative humidity.

From Figure 4.3, 4.4 and 4.5, it is shown that the RI for all 3 samples at RH1 and RH2 decreases with respect to time. Also, RI for Sample 1 and Sample 3 at RH3 decreases with respect to time. At this point, it indicates that the sol-gel process is not occurring.

In the other hand, from Figure 4.4, the RI for Sample 2 at RH3 initially decreased but then increases. This means the sol-gel process is occurring. By analyzing this pattern from the graphs, it is said that the higher RI provides better condition for the process. For this case, 95.6 % of relative humidity provides a suitable condition for the process. From this experiment also, only sample 2 at 95.6% RH undergo the process, means the composition is having sufficient amount of water and precursor.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

1. Generally, increase in refractive index with respect to time indicates that the more silica gel is formed.
2. At the room temperature and pressure, the higher concentration of silica sol takes shorter time to become totally silica gel.
3. Higher relative humidity (95.6%) provides a better condition for sol-gel process to occur.

5.2 RECOMMENDATION

There are some modifications to current experimental work such as:

1. Measure the relative humidity in the first experiment where the amount of TEOS is the manipulative variable
2. Obtain Scanning Electron Microscopy (SEM) pictures to observe the morphology or each sample.

Other scopes of study in future that can be considered are:

1. To study the effect of flouride ion to the gelation kinetic.
2. To study the effect of temperature to the sol-gel processing.

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