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ABSTRACT (100-200 WORDS):

The feasibility of using ultrasonic cavitation to reduce the size of silica particles in order to form a silica sol is discussed. A silica sol can be processed into silica glass via sol-gel processing. Cavitation is the formation, growth, and implosive collapse of bubbles in a liquid. The collapse produces intense heating and high pressures resulting in the potential to erode or fragment surfaces. Cavitation may be induced using ultrasound. A sol is a suspension of solid particles that are small enough to be dispersed in the liquid without dissolving or settling. Ultrasonic cavitation has the potential to reduce silica particles to the appropriate sizes to form a sol. An analysis of existing literature predicts the most effective conditions to produce a silica sol via ultrasonic cavitation. To increase the potential for getting particles small enough to form a sol (5 to 100 nanometers), the initial particle size should be at least as large as the cavitation bubbles produced. At a potent insonication frequency of 20 kilohertz, the particles should be at least 200 microns in diameter.

Degassed water at 40° Celsius under one atmosphere of pressure is a good subject to insonicate. The pH of the water may be adjusted with aqueous ammonia to produce a more stable sol.

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**The University of Alabama in Huntsville
Senior Honors Project**

Feasibility of Using Ultrasonic Cavitation to Produce a Silica Sol

Gary Vines

March 22, 1994

Abstract

The feasibility of using ultrasonic cavitation to reduce the size of silica particles in order to form a silica sol is discussed. A silica sol can be processed into silica glass via sol-gel processing. Cavitation is the formation, growth, and implosive collapse of bubbles in a liquid. The collapse produces intense heating and high pressures resulting in the potential to erode or fragment surfaces. Cavitation may be induced using ultrasound. A sol is a suspension of solid particles that are small enough to be dispersed in the liquid without dissolving or settling. Ultrasonic cavitation has the potential to reduce silica particles to the appropriate sizes to form a sol. An analysis of existing literature predicts the most effective conditions to produce a silica sol via ultrasonic cavitation. To increase the potential for getting particles small enough to form a sol (5 to 100 nanometers), the initial particle size should be at least as large as the cavitation bubbles produced. At a potent insonication frequency of 20 kilohertz, the particles should be at least 200 microns in diameter. Degassed water at 40° Celsius under one atmosphere of pressure is a good subject to insonicate. The pH of the water may be adjusted with aqueous ammonia to produce a more stable sol.

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In this document, I will discuss the feasibility of using ultrasonic cavitation to reduce the size of silica particles to form a sol for sol-gel processing into silica glass. My research focused on determining the appropriate method of experimentation and materials needed to produce a silica sol from ultrasonic cavitation.

This report consists of four sections. The first section, the prologue, introduces a lay audience to cavitation and sol-gel science by defining terms, providing a relevant scientific history, and defining the experimental problem. The second section is an analysis of existing literature related to the experimental problem. It discusses the experimental conditions most likely to produce the desired silica sol. The next section presents the results of some relevant, preliminary experimental work performed at The University of Alabama at Huntsville (UAH). The final section reiterates my conclusions from the analysis section and recommends an experiment design.

PROLOGUE

This section defines technical terms of this study, presents a relevant scientific history, and defines the experimental problem.

Technical Terminology

Cavitation is defined as the formation, growth, and implosive collapse of bubbles in a liquid. The collapse produces intense heat and high pressure for a very short time. As a result, cavitation has the potential to erode or fragment surfaces. A common example of this damage may be found on eroded propeller blades of some marine craft. One way that cavitation may be induced is using ultrasound, which spans the frequencies from 15 kHz to 10 MHz.

A sol is a suspension of solid particles that are small enough to be dispersed in the liquid without dissolving or settling. A gel is a network of solid particles often acquired by partially removing the liquid from a sol. Ultrasonic cavitation has the potential to reduce silica particles to the appropriate sizes needed to form a sol. A silica sol can then be used in sol-gel processing to make various types of glasses and thin films.

The sol-gel process generally involves partially removing the liquid from a sol, which forms a gel, then drying and densifying the gel to produce a solid. Work in sol-gel science is recorded as early as 1864 when Thomas Graham prepared gels of silica (1: 2). In the 1940's thin films of silica had been produced and by the late 1950's the process was producing rear view mirrors for automobiles (2: 1). Around 1980, the sol-gel process began being used to create many new optical materials. Current research specific to sol-gel silica glass focuses on improving the quality and properties of silica glass for use in ultraviolet holography, precision optical imaging devices, and coatings for eyeglasses.

One great advantage of using the sol-gel process over normal processing is the reduced heat required to produce a dense solid. Normal processing requires a temperature of 1750^o Celsius (C) to fuse silica as opposed to 1100^o C by sol-gel processing (2: 1). Sol-gel silica glass is an important optical material because of its superior quality over other types of silica glass in the following areas: low expansion coefficient; ultraviolet, near infrared, and visible wavelength transparency; and absence of cations. In addition, it has a lower hydroxyl content and is more dense, providing a higher refractive index (1: 7).

Cavitation History and Background

The history of cavitation begins in 1894 when John I. Thornycroft and Sydney W. Barnaby investigated the cause of vibrations in a British destroyer.

They concluded that large bubbles were created by the propeller and imploded from water pressure. In 1917, Lord Rayleigh was commissioned by the British navy to study this phenomena known as cavitation. His mathematical analysis showed that pressures on the order of 10,000 atmospheres (atm) were created on the implosion of a spherical cavity. In 1927, Alfred L. Loomis began to study sonochemistry, the effects of soundwaves on liquids resulting from cavitation. Sonochemical experiments were sporadic until the 1980's, when high intensity ultrasound generators could be produced inexpensively. Sonochemistry started being used for catalysis of chemical reactions, increasing the reactivity of powders, and driving metal particles together at high speeds so that they melt on collision (3: 80).

Since 1917, scientists have tried to measure the high temperature and pressure produced by cavitation. Because the size of the bubbles is on the order of 100 microns and the implosion typically occurs in less than one microsecond, precise measurements are difficult. Recent estimates of the temperature fall around 5,500^o C and pressure estimates vary from 500 atm to 4500 atm (4: 1439; 3: 80; 5: 230). Kenneth Suslick dramatically, yet realistically, described the potential of cavitation in the following way: "[f]or perspective. . . one can create the temperature of the sun's surface, the pressure of deep oceanic trenches, and the cooling rate of molten metal splatted onto a liquid-helium-cooled surface" (4: 1439).

Cavitation effects are the result of a bubble implosion from the pressure of the surrounding liquid. The cavitation bubble is produced during the rarefaction cycle of an acoustic wave. A rarefaction is a low pressure region. The rarefaction region is created when molecules are compressed and then pulled apart. This activity may be visualized by the events in Figure 1, which illustrates the displacement of molecules in time due to a longitudinal wave. A transducer

compresses molecules A and B; B then moves toward C. The transducer then returns to its original position and to the left as A decompresses and follows. The kinetic energy from the initial collision produces subsequent collisions toward the right. After collision, the low pressure, or rarefaction, region then pulls these molecules to the right. The transducer then moves toward the right to collide with A and begin a new cycle. One cycle is complete when all molecules are in their initial position (6: 10-11).

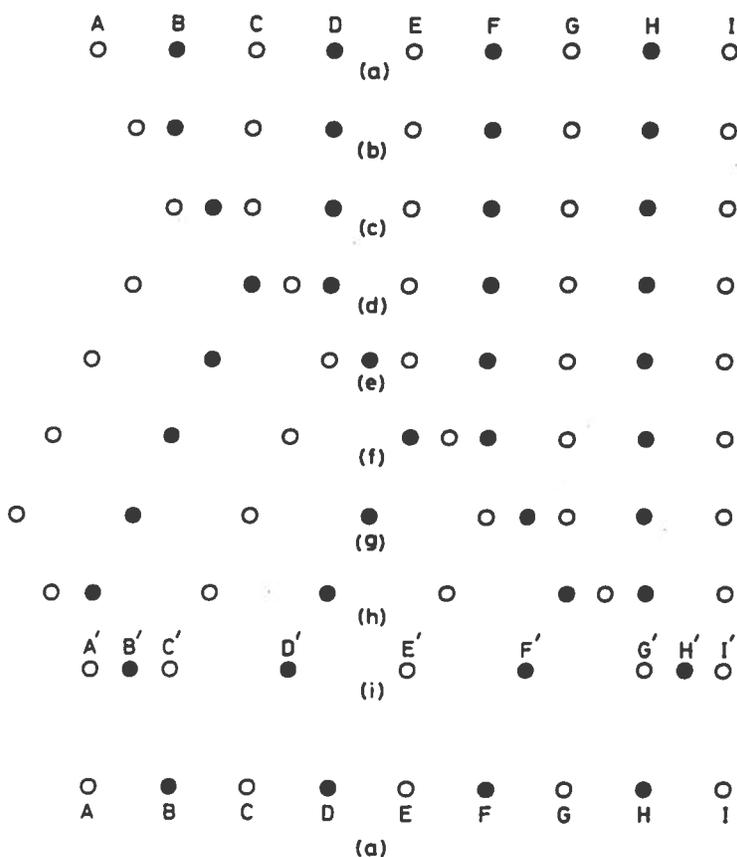


Figure 1 Displacement of Molecules Due to a Longitudinal Wave.
Source: Mason, T.J. Sonochemistry: the uses of ultrasound in chemistry. 1990, 10.

Suslick notes that the rarefaction pressure is not solely responsible for the production of a cavity. For instance, in ultrapure water the negative pressure required to produce cavities between molecules is predicted to be over 1000 atm. The most powerful ultrasound generators produce a negative pressure of 50 atm. Suslick asserts that gas-filled crevices in small particles reduce the tensile strength of the liquid so that only a few atmospheres of negative pressure is required for cavitation (3: 80).

It is worthwhile to note that in the previous and following discussions the words bubble, cavity, and void are used loosely and to some extent interchangeably. These three words represent a continuum with differences in the amount of vapor present in a spherical or nearly spherical shell. A void lacks vapor. A cavity may have some vapor, and a bubble usually acquires sufficient vapor to rise up through a liquid if it does not implode first.

Experimental Problem

The current and probably most conventional method of making a silica sol is by hydrolyzing, adding water to, a silicon alkoxide. A silicon alkoxide is a molecule with a silicon atom at the center surrounded by several alkoxy groups (oxygen plus multiple hydrogen and/or carbon molecules). The reaction is catalyzed by adding either an acid or a base depending upon the desired product, to be discussed later. For example, hydrolysis of tetraethoxysilane, TEOS, removes the alkoxy groups and replaces them with hydroxyl groups. $\text{Si}(\text{OC}_2\text{H}_5)_4$ becomes $\text{Si}(\text{OH})_4$. Loss of alcohol and water yield SiO_2 particles.

Milling is another, much older method of producing a sol. Milling involves rotating a jar containing ceramic balls so that the balls tumble and fragment the particles. In 1920, Victor Lenher reported that "if quartz [a type of silica] is ground sufficiently long, it can be so finely divided that it will go into colloidal solution

when treated with water" (7: 392). As simple as it seems, milling is a science with results dependant upon many variables and can be very time consuming (2: 40-41). Ultrasonic cavitation offers the potential for a method of producing a stable silica sol without expensive chemical reactions or time consuming milling.

In 1927, W.R. Richards unsuccessfully attempted to disperse glass particles in water using high frequency sound waves (8: 1727). He used a frequency of 300 kHz which, as discussed later, may be too large to produce optimum cavitation effects. The use of ultrasonic cavitation to reduce the size of silica particles beckons experimentation; I have not seen where anyone has published results on the topic.

The following sections of this report examine the literature describing the mechanisms behind the eroding effects of cavitation, discuss experimental considerations for producing optimum effects, and report the results of some simple experiments. The experimental considerations attempt to answer several questions: What particle size distribution is needed to produce a stable sol? What energies/pressures are available from the implosive collapse of the bubble? What is a good liquid to irradiate with ultrasound? What pH level of liquid is needed to produce a stable sol? What initial silica surfaces or shapes are subject to most fragmentation?

REVIEW OF LITERATURE

I begin this section by reporting the optimum size of silica particles to produce a sol. I then proceed with a discussion of the characteristics of ultrasonic cavitation to produce particles of this size.

Optimum Particle Size

A silica sol is a colloidal suspension of particles ranging from 5 nanometers (nm) to 100 nm in diameter. Particles larger than 100 nm tend to settle and particles less than 5 nm tend to aggregate due to Ostwald ripening (9: 345,317). Ostwald ripening is a pH and temperature dependant process in which very small particles dissolve and are deposited on larger particles (9: 175).

Commercial sols, which must be very stable for shipping, contain particles ranging from 5 nm to 60 nm (9: 415-417, 517). A distribution of sizes between 5 nm and 100 nm seems advantageous. Iler notes that for producing thin films, a sol of particles measuring 100 nm in diameter cracks less than a sol with finer particles; however, it remains soft. If a distribution of sizes is used, the smaller particles should fill in the cracks between larger particles thus producing a stronger, more dense film (9: 370). In conclusion, a distribution of particles between 5 nm and 100 nm can produce a stable sol capable of forming desirable products through subsequent sol-gel processing.

Mechanism of Cavitation Damage

In 1917, Lord Rayleigh produced the first mathematical model of the pressures developed in a liquid due to a collapsing cavity. He calculated the instantaneous pressure as a cavity wall hits a solid surface to be near 10,300 atm (10: 98). From ensuing research, two separate mechanisms of erosion caused by cavitation are known to exist and produce high pressures at liquid-solid interfaces--the shock wave and the liquid jet.

The existence of a liquid jet, produced by a collapsing cavity, was theoretically and experimentally proven in the 1950's and 1960's. In the presence of a wall, an initially spherical bubble undergoes an asymmetrical collapse due to a disturbance in the flow of the liquid. As depicted in Figure 2, the bubbles

"become elongated in the direction normal to the wall; then they tended to flatten and form an inward moving jet on the side of the bubble opposite the wall"(11: 284). Photographic studies have shown that extended surfaces or a bubble of equal size or larger may sufficiently perturb the bubble to produce jetting (12: 263-267).

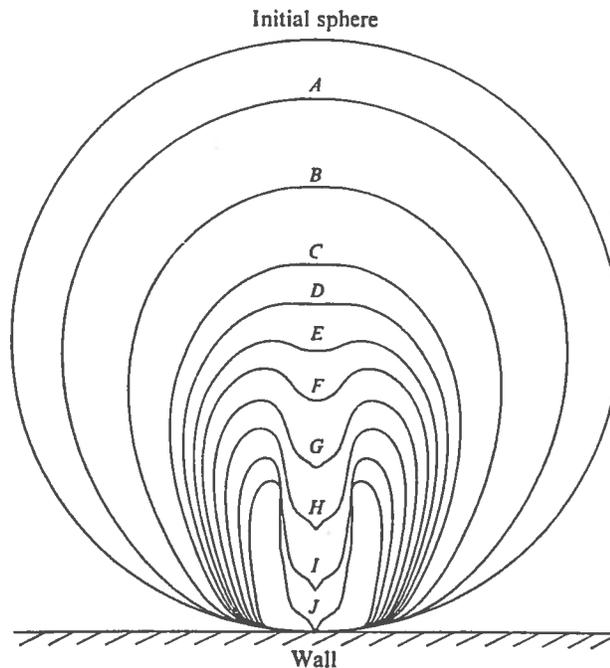


Figure 2 Asymmetrical Bubble Collapse in the Presence of an Extended Surface

Source: Plesset, Milton; Chapman, Richard B. Collapse of an initially spherical vapour cavity in the neighbourhood of a solid boundary. *Journal of Fluid Mechanics*. 1971; 47(2): 287.

Measurements of the jet velocity vary from 80 m/s to nearly 200 m/s (12: 263, 265). These measurements agree with estimates by Plesset and Chapman, who produced two simulations of an initially spherical bubble collapsing near a solid boundary. In the first, the bubble was touching the wall, as in Figure 2. In the second, the distance was one-half the initial radius of the bubble. Jet speeds were estimated to be 130 m/s and 170 m/s, respectively. Due to attenuation, the

first case is thought to be the more potentially damaging. The resulting pressure was approximated using the water hammer equation,

$$P = CV\rho \quad (\text{Eqtn. 1})$$

where ρ is the density of liquid, C is the speed of sound in the liquid, and V is the jet velocity. The resulting pressure may be as large as 2000 atm in water at 1 atm ambient pressure; however, the duration of stress lasts only about 10^{-7} seconds. As a result, the real damage mechanism may be the stagnation pressure, which is the pressure exerted by the jet when its velocity is zero. The duration of this pressure lasts longer and the magnitude is estimated to be 800 atm (11: 283-290).

The shock wave was discovered before the jet and is known to produce greater pressures. Jet production requires the presence of another surface at least as large as the bubble itself. At an insonication frequency of 20 kHz, the collapsing bubble size is about 150 microns (13: 1067). If the solid particles are not larger than the bubbles, then jets are not formed and any damage or erosion is a result of shock waves (14: 285). Pressures from shock waves have been measured to be about 3000 atm, and may peak to 4500 atm (15:1068; 5: 730). According to Plesset, the shock wave pressure attenuates according to $1/r$ where r is the distance from the bubble center (16: 243). Therefore, as with the liquid jet, the solid surface must be very close to the collapsing bubble for damage to occur.

Although these pressures seem impressive and are capable of damaging some surfaces, Neppiras states that the pressures due to the jet "are hardly great enough to cause erosion in resistant metals" (5: 230). Rayleigh's equations show that a typical bubble collapsing in water, at an ambient pressure of one atm, theoretically produces a potential energy of 4.2 ergs. Doktycz reported energies only in excess of 0.12 erg, based on the fusing of certain metals upon collisions produced by cavitation (13: 1068). Beginning with a silica particle 0.01 cm in

diameter using the reduced free surface energy of silica in water of 50 ergs/cm², Seeley calculated that a particle could not be reduced below one micron in diameter with a potential energy of 4.2 ergs (17: 18-19). Fortunately, experiments of Gutierrez and Henglein indicated that ultrasonic cavitation "does not lead to a gradual crushing of smaller particles but to a splitting-off of very small fragments" (18: 260). These fragments are the desired particles needed to produce a sol.

Threshold Energies and Associated Pressures for Cavitation

One way to think of cavitation is to visualize it as cold boiling. Boiling occurs, that is bubbles are created in a liquid, when the vapor pressure of the liquid is greater than the atmospheric pressure. During insonication, acoustic pressure is added to the vapor pressure. (Insonication is the process of irradiating with ultrasound.) With sufficient acoustic pressure, cavitation bubbles are produced during the negative pressure cycle. Therefore, the acoustic pressure, P_A , must be greater than the difference between the atmospheric pressure, P_H , and the vapor pressure, P_V (6: 16). The acoustic pressure may be thought of as the rarefaction pressure

$$P_A > P_H - P_V \quad (\text{Eqtn 2})$$

For example, the vapor pressure of water at 20^o C and under an ambient pressure of one atm is 17 Torr. One atm is equal to 760 Torr. From equation 2, P_A simply needs to be greater than 743 Torr. The required intensity threshold may be determined using P_A

$$\text{Intensity} = P_A^2 / 2 c \rho \quad (\text{Eqtn 3})$$

where ρ is the density of the liquid and c is the velocity of sound in the liquid, which is 1500 m/s for water. From equation 3, the required intensity is 0.3 W/cm², which is easily produced by ultrasound generators (6: 12, 16).

Optimum Insonication Frequency

A bubble requires a finite time to grow. If the time required is less than the rarefaction cycle, then cavitation is not produced. Therefore, cavitation becomes less likely with increasing frequency. Most sonochemical reactions are performed between 20 kHz to 50 kHz for optimum cavitation effects (6: 14-18). A frequency of 20 kHz, just above the human hearing capacity, allows one cycle to occur every 50 microseconds giving the bubble time to grow to 150 microns to 170 microns. This frequency produces strong cavitation effects evident from other experiments (19: 56; 13:1067; 4: 1440).

Insonication Duration

Based upon previous tests performed by Suslick, results from cavitation effects did not increase significantly beyond 15 minutes of insonication (19: 57). In lab work performed at UAH, high temperature super conductors were insonicated for up to 6 minutes, upon which no significant increase in results were observed (17: 7.). Therefore, an insonication duration of 10 to 15 minutes should be sufficient to produce the desired results.

Ambient Pressure

The ambient pressure has little effect on cavitation dynamics according to Hunter, Hickling and Plesset. Numerical calculations based upon the collapse of a spherical bubble under different external pressures showed the velocity of the wall collapse approaches infinity at the same rate (16: 243). Neppiras and Noltingk noted that under certain conditions, a standing pressure of two atm produced the maximum fluid pressure, but decreased at higher standing pressures. However, they noted that only limited conclusions could be drawn

from their results (20: 1036-1037). Based upon these results, an ambient pressure of one atm is recommended.

Liquid Choice

The choice of liquid is important to produce optimum cavitation effects. For best results, the liquid should have "low viscosity, high surface tension, low vapor pressure, and high sound speed" (21: 165-166). High thermal conductivity increases the collapse velocity; however, low thermal conductivity increases the average collapse pressure. Water fits these criteria, including having low thermal conductivity, and is known to produce violent cavitation (22: 238). The weight reduction of an extended surface insonicated in water is several times greater than the weight reduction from most organic liquids (21: 165-166). In addition to producing violent cavitation effects, water is known to reduce the surface energy of silica. The surface energy is the energy needed to create a new surface area from a particle. The presence of water reduces the energy needed to rupture a silicon-oxygen bond by nearly a factor of twenty, thus allowing easier cracking or erosion (23: 125). As a final consideration, water is inexpensive and easy to handle.

Two types of water may be used to produce cavitation, tap and degassed. Tap water contains dissolved gases and gas bubbles which serve as nuclei in bubble formation. As the name suggests, degassed water has had most of its gas removed. The gas in tap water lowers the required intensity threshold to produce cavitation and makes the bubble harder to collapse, possibly producing greater pressures upon collapse (24: 5). The drawback to tap water is that gas around a solid particle may cushion or cause greater attenuation of the pressures produced by bubble collapse and reduce the effects.

An alternative to water is aqueous ammonia. Since an ammonia molecule is about the same size as a water molecule, it has a similar effect on silicon-oxygen bonds. Ammonia is slightly more viscous than water but has a slightly lower thermal conductivity. As discussed later, the pH of a liquid is important and ammonia is a good reagent to adjust the pH. A good, inexpensive, versatile solvent is water with sufficient aqueous ammonia added to achieve a desired pH.

Temperature of Liquid

Temperature affects cavitation by changing the required threshold intensity. A temperature increase may increase the vapor pressure, lower the viscosity and/or surface tension of the liquid(6: 18). Flynn reports that "erosion will increase to a maximum and then decrease" in response to an increase in ambient temperature(21: 165): "in water at 50^o C, the weight loss by cavitation erosion on aluminum plates is four times greater than at 20^o C" (21: 165): Lorimer reports that for toluene erosion rate decreases for temperatures beyond 60^o C (25: 120). The vapor pressures of water and toluene at different temperatures are very close. Flynn reports that the maximum cavitation activity at a surface will occur when the vapor pressure of the liquid is between 35 and 80 mm Hg (21: 165). From the previous results, the optimum temperature for erosion (for water or toluene) appears to be between 40^o C and 60^o C.

pH-Sol Stability

Although the mechanism for producing a silica sol or gel via ultrasonic cavitation is drastically different from other mechanisms, control of the pH of the liquid is important to the stability of the sol for all mechanisms. For instance, many silica sol-gels have been produced using alkoxides as a silica source. As an example, tetraethoxysilane, TEOS, is dissolved and hydrolyzed with acids or

bases to produce a sol. Acid hydrolysis eventually yields clear gels probably composed of linear chains. Base hydrolysis yields cloudy gels (26: 504). Particle size reduction via ultrasonic cavitation should also depend on the correct pH level to help prevent aggregation, which occurs with very small particles (less than 5 nm in diameter). In either mechanism, the pH level of the liquid controls the ionic charge concentration around a particle and affects interparticle behavior. The particles throughout the liquid develop surface charges. Surrounding ions of opposite charge are attracted to the ionic layer on a particle while ions of the same charge are repelled. This double layer of ions separates and disperses the particles in the liquid. (This is known as the electrical double layer theory of which a detailed discussion is beyond the scope of this report.) The isoelectric point is a pH level at which the particle has no surface charge. At the isoelectric point particles may collide with one another and join, or aggregate, causing an increase in particle size (2: 15).

The stability behavior of silica sols partially contradict the double layer theory. Between pH 7.5 and 10.5 the particles possess a negative surface charge which prevents undesired aggregation (9: 331). However, at the isoelectric point, pH 2, the silica particles should theoretically aggregate quickly; instead, sufficiently small particles produce a relatively stable sol (9: 324). The sol is said to be relatively stable because it will gel very slowly at pH 2. Gelling is a type of aggregation in which the particles are linked together in chains and there is no increase in the concentration of silica in the medium. Therefore, the sol does not produce a precipitate as in other types of aggregation. Gelling occurs in silica sols at pH 7.5 and below. Gelling occurs most quickly from pH 5.5 to pH 6.5 and, surprisingly, very slowly at the isoelectric point of pH 2. Figure 3 shows the effects of pH in the colloidal silica-water system (9: 367). Since silica solubility increases above pH 10.5, two pH ranges exist in which a sol can be made. A very

stable sol can be produced between pH 7.5 and 10.5 and a slow gelling sol can be produced at pH 2.

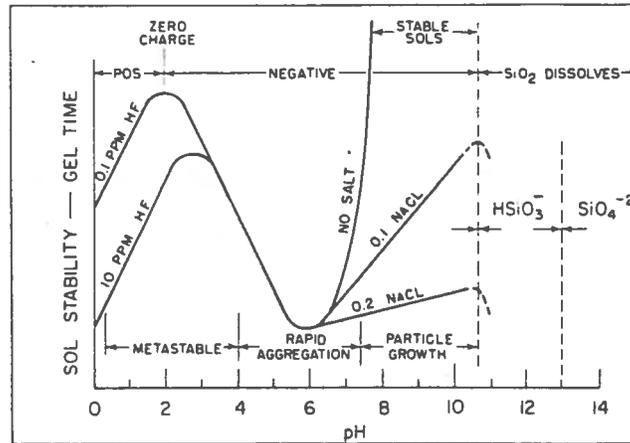


Figure 3 Effects of pH in a Silica Sol

Source: Iler, Ralph K. *The chemistry of silica: solubility, polymerization, colloid and surface properties, and biochemistry*. New York: John Wiley & Sons, Inc., 1979, 367.

Starting Particle Size and Concentration

As I noted earlier, the surfaces of silica particles to be insonicated should be larger than the diameter of the bubbles. Particles of this size, including extended surfaces, will help to produce asymmetrical bubbles producing water jets and greater potential for erosion. For a frequency of 20 kHz, the initial size of silica particles should be 200 microns or larger in diameter.

No data was found reporting optimum concentration of the solid to be insonicated. In similar experiments using other solids, concentrations varying from 0.1 g of solid per 50 ml of solvent to 1 g of solvent to 15 ml of solvent were reported (19: 56; 18: 259). Since no significant chemical reactions are expected to occur, silica may be added liberally and subjectively.

RELEVANT EXPERIMENTAL WORK

At UAH, Seeley has performed two simple experiments which I would like to report. He used a Heat Systems Engineering XL-2015 Sonicator, which operates at 20 kHz and has a probe with a cross sectional area of 1.3 cm^2 . He insonicated beach sand, which is about 95 percent silica, for approximately five minutes in water. The water turned cloudy and remained somewhat cloudy after being filtered and then standing for two weeks. Since silica particles larger than 100 nm tend to settle within a few days it is likely that some silica was fragmented into particles small enough to be dispersed as a sol, along with some of the impurities in the sand.

In another experiment an extended surface of glass was insonicated three times for 5 minutes each time in tap water. The glass fractured into several smaller macroscopic pieces and enough microscopic pieces to make the water cloudy. Like the first experiment, the water remained cloudy after standing for several days. These results appear to contradict the results obtained by Richards, probably because Richards insonicated glass at such a high frequency that the bubbles did not have time to grow large enough to produce violent cavitation fragmentation.

CONCLUSIONS/EXPERIMENT DESIGN

As stated, I have not seen any published work on using ultrasonic cavitation to reduce the size of silica particles to form a sol. From the previous analysis, I believe ultrasonic cavitation has the potential to fragment surfaces thus producing particles small enough to form a silica sol. I have stated several experimental conditions that I believe would contribute to maximum fragmentation

due to violent cavitation. The only problem that I foresee is that of yield since the amount of silica sol actually produced by insonication may be low. The ratio of silica mass to liquid volume for producing the greatest yield is unknown. At this point, given the simplicity of the experiment, it is best to perform the experiment using the conditions discussed in the previous analysis. I propose that an experiment testing the feasibility of using ultrasonic cavitation to produce a silica sol be performed under the following conditions. Figure 4 shows the experimental apparatus setup.

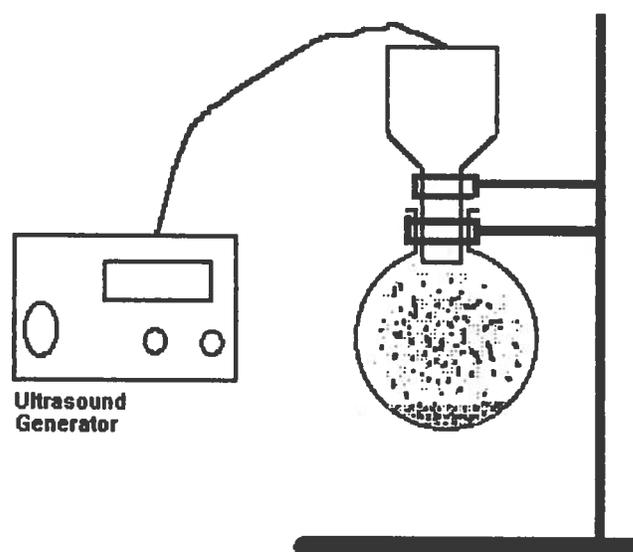


Figure 4 Experimental Apparatus

1. Place approximately 10 g of granulated silica particles at least 200 microns in diameter in a 300 ml round bottom flask.
2. Fill the flask with degassed water at 40° C.

3. Add aqueous ammonia (NH_4OH) dropwise to raise the pH to about 8.0.
4. Place the ultrasonic generator horn into the flask opening and insonicate for 10 minutes at an ambient pressure of one atm. (Note: A water bath may be needed to maintain the temperature between 40°C and 60°C . An alternative to the water bath is insonicating for two periods of five minutes, instead of ten minutes, allowing the water temperature to return to 40°C between the periods.)
5. Separate the liquid from the remaining silica and filter.
6. Allow the filtered liquid to stand for several days to precipitate particles larger than 100 nm and to determine the stability of the sol.

As a final note, the purpose of this project was to investigate a new option for making a silica sol. The option does seem feasible and, therefore, after further testing, may eventually be used as a preliminary step in sol-gel processing. Sol-gel technology is a potential tool for many applications, including precision optics and thin films. My interest in the subject developed from experiments at UAH, in which Seeley used sol-gel technology to try to produce thin film superconductors. As a physics student and an aspiring optometrist, I am interested in producing high refractive index optical lenses and a new, efficient method of applying thin films to optical lenses (ie. anti-glare or scratch resistant films) with sol-gel technology. As mentioned, the quality of sol-gel silica glass is known to be superior to that of silica glass produced by other methods. The use of ultrasonic cavitation to produce a silica sol appears to be more efficient than other methods discussed.

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