Sonochemically-Induced Nanoparticle formation in Microgravity

Topic Area: Sonochemisty

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Table of Contents

Introduction

Sonochemistry is a process in which ultrasound is used to achieve high energy chemistry. When an acoustic field is applied to a liquid, bubbles within the liquid undergo a process known as acoustic cavitation. Acoustic cavitation consists of three stages: Nucleation, bubble growth, and collapse. During nucleation, bubbles are formed within the liquid due to the negative pressure of the applied acoustic field. These bubbles then go through a growth process in which they absorb dissolved gases within the liquid. When the bubble reaches a given size it can achieve resonance, at which point it can efficiently absorb energy from the sound field. This causes the bubble to grow rapidly during a single acoustic cycle (2). At this stage, inertia dominates the system and causes the bubbles to collapse violently. Energy is concentrated within the bubbles as they implode. Compression of the gas within the bubble as it collapses generates extreme heat and pressure (4000K and 200 atm respectively) (1). *See Figure 1*.

The rapid expansion and collapse of the bubble results in rapid heating and cooling rates which provide a unique environment for chemical reactions to occur. Of particular interest to our study, when a metallic colloidal system is sonocated, H and OH radicals are formed within the local hotspots of the bubbles (1,2). These free radicals may then react with the metal colloids to form nanosized metal particles (nanoparticles).

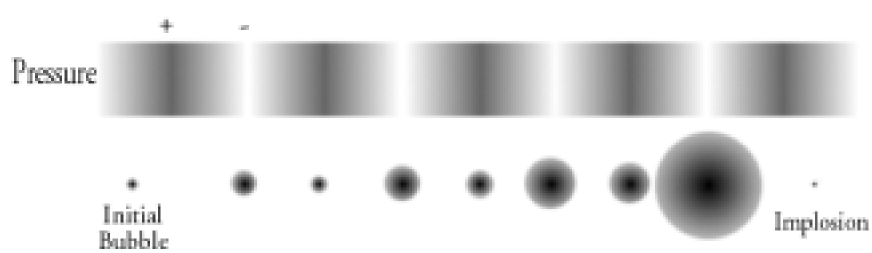


Figure 1. The initial bubbles absorbs dissolved gas from the surrounding Liquid and grows. Once large enough the bubble resonates and absorbs acoustic energy. It quickly grows over the course of one acoustic cycle until it can't sustain itself and it implodes.

Sonochemistry and Gravity

The formation of nanoparticles is directly related to the cavitation field. This cavitation field consists of hundreds or thousands of oscillating bubbles. Each individual bubble is a possible source of sonochemistry providing that the bubbles collapse is energetic enough to initiate chemical activity. It is known that in a 1g environment at least some of the bubbles generate pressures and temperatures high enough to form nanoparticles. However, buoyancy may limit the rate of formation of these nanoparticles. This is because gravity causes bubbles to translate upward (float). As these bubbles move through the liquid, the drag force distorts their spherical shape. As a consequence, the bubbles' collapse may not be spherical, in which case the amount of energy focused at the center of the bubble will be decreased. In zero gravity, however, the absence of buoyancy may effect the dynamics of these cavitation bubbles. We propose that this absence of buoyancy will result in a higher percentage of bubbles undergoing spherical collapse, resulting in an enhanced yield of nanoparticles. Partial support for this hypothesis comes from experiments done by Matula regarding single bubble sonoluminescence. Matula reported 11-30% increase in light intensity emitted by a single bubble while in zero gravity as apposed to 2g. A part of that increase in light intensity may be due to greater temperatures within the bubble since the compression is more spherical.

We hypothesize that gravity may effect the rate of sonochemical reactions in another way. It has been observed that during the sonocation process, bubbles close to the ultrasonic horn coalesce to form larger bubbles. These bubbles are to large to absorb the acoustic energy but instead reflect the energy back towards the horn. This process, known as acoustic shielding, prevents smaller bubbles from absorbing a significant amount of acoustic energy. We propose that buoyancy contributes to bubble coalescence near the tip of the horn. Thus when the sonocation process is carried out in micro gravity there will be fewer bubbles that coalesce. As a result the bubbles will remain small and will be distributed through out the colloidal system. This will result in a much more efficient production rate of nanoparticles. In summary, the effects of buoyancy on cavitation bubbles may limit the formation of nanoparticles in a sonochemical apparatus. In the next section we describe a proposed experiment to test our hypothesis.

Experimental Setup

Figure 2 shows the diagram of our apparatus. We will begin setup for the experiment at The University of Washington. First we will prepare several 20 ml samples of Palladium Chloride ($PdCl_2$) and ethanol in a weak acid solution. These samples will then be stored for transportation to Houston. Upon arrival to Houston we will fill the sonocation chamber with our sample. The chamber will then be fastened to the ultrasonic horn using an O-ring and steel fasteners. A gas purge port will be used to remove the overhead air. The chamber will also contain an electric thermometer connected to our lap top and analyzed by the lab view program. This program will record the temperature of our sample during the duration of our experiment. The acoustic horn will heat our sample during it's operation. Our desired operating temperature is 20 degrees Celsius, so we will be placing chemical cooling packs within a coolant chamber around the sonocation chamber in order to cool the sample down from any heating from the horn. Since our proposed experiment is to test percent yield of nanoparticle creation in micro gravity, our experiment will only be ran during the 20 seconds of reduced gravity. An accelerometer will connected to the lap top and lab view will record the acceleration. Lab view will give the ultrasonic horn power only while in micro gravity. The lab view program will also record the duration of which power will be given to the horn. This information will be used later to mimic our experiment in a 1g environment.

The same sample will be used for both flights and analyzed back at the university. We will then perform our experiment again at The University of Washington in a 1 g environment. Our sample will again be analyzed and the results from micro gravity will be compared to those of the 1g experiment. The next process will discuss our analysis techniques in detail.

Once our experiment has been completed we will analyze the products back at the University of Washington. The analysis will be done using the technique of titration. During the formation of Pd nanoparticles Hydrogen radicals formed within the bubble reduce Pd^{+2} ions in the solution resulting in $(Pd)_n$ nanoparticles and hydronium (H+) ions. An acid base titration will be used to detect the concentration of these hydronium ions. This concentration of hydronium ions will be used to find the mass of Pd^{+2} ions reduced to nanoparticles using the equation $2H + Pd^{+2} \rightarrow nPd + 2H^{+}$. The same analysis will be done on the 1g sample and the yields will be compared.

Flight Plan

While in flight, our job will be to monitor the experiment. However no actual measurements or control will be done by any one member of the team. Instead the computer will simply control when the horn is turned on or off. It will be our job then to make sure that the program is functioning properly and no faults occur within our system. If the program fails, the horn may then be operated by hand.

Proposed manifest for flights

Flight 1: Justin and David
Flight 2: David and Justin

On the Ground

1. Load chamber with prepared sample and secure to horn.
2. Secure apparatus to base plate and airplane.
3. Turn on computer and open Lab View program.
4. Test accelerometer and thermometer.

In Flight / Pre-parabola

1. Activate chemical cooling packs and secure in coolant chamber.
2. Begin data acquisition.
3. Monitor program and apparatus for malfunction.

In Flight / Parabolic maneuvers

1. Continue monitoring program and apparatus

Post Flight 1

1. Power down all equipment
2. Remove apparatus from plane
3. Store for next flight

Post Flight 2

1. Power down all equipment
2. Remove apparatus from plane
3. Prepare and store sample for transport back to Washington.

Structural Analysis

Our experimental setup consists of the laptop, a power supply, spill container, the acoustic horn and sample, and an accelerometer. Each of these will be bolted to an aluminum base plate (24 inches wide by 44 inches long by 0.5 inches thick) This base plate will be secured to the aircrafts 20 inch. Center with six steal bolts. We begin our structural load section with an analysis of this base plate.
The safety factor for the bolt hold down configuration must be greater than 5. Using the NASA supplied figure of 5000 pounds per bolt. The safety factor considering a possible 2.5 g acceleration is....

$$\frac{5000\text{lbs} \times 6}{200\text{lbs} \times 2.5} = 60$$

This factor is much greater than the required safety factor of **5**. Thus our bolts will not fail in tension. Any shearing force on the bolts would be due to the 9 g's acceleration of a forward crash loading. The shear stress in a bolt is given by

$$T = (\text{ P/bolt })/\text{bolt*area}$$

P is the force on a bolt.

For the six bolts holding the base plate under a 9g loading yields

$$T = \frac{(200\text{lbs} \times 9)}{(\pi/4)(.5)^2 \times 6 \text{ bolts}} = 1.5 \text{ ksi}$$

The shear stress for steal is at least **58 ksi**; therefore the bolts have a sufficient safety factor for shearing. We will now analyze the possibility of the bolts ripping through the base plate given a 9 g acceleration. This calculation is given by.....

$$\sigma = P/t*d$$

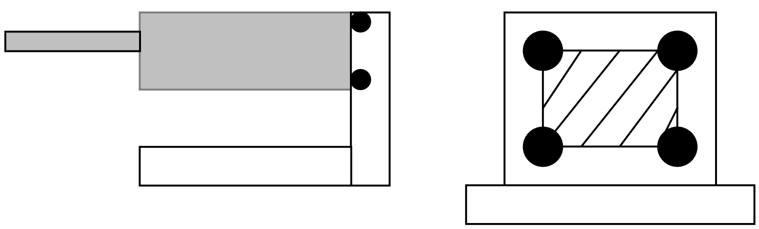
P: P/bolt is a force
t : is the thickness of the plate
d : is the diameter of the bolt

$$\sigma = \frac{200lbs X 9}{(.5)^2 X 6bolts} = 1.2 \text{ ksi}$$

The shear stress maximum for Aluminum is **27 ksi**; therefore the safety factor is more than sufficient.

The same analysis applies for all bolts securing the apparatus to the base plate. Since the entire system is only 200 lbs and 4 bolts will be used to hold each device down to the base plate. Calculation using the full 200 lbs and 4 bolts yields compliant safety factors of: **σ =1.8 ksi , *T= 2.3 ksi*** These factors are well within the safety limitations.

The acoustic horn will be secured to the base plate via an aluminum angle plate. There will be four steal bolts securing the horn to this angle plate....



These four bolts must comply with the safety factor of 5 for 5000 lbs per bolt. The horn is approximately 20 lbs, and must withstand a 9 g forward crash load.

$$\frac{5000lbs X 4}{20 lbs X 9} = 111$$

This calculation shows that the bolts will not fail in tension, and comply with the safety factor. The shear stress on these bolts would be due to a 6 g downward loading. The shear stress for the four bolts is given by...

$$T = \frac{(20lbsX6)}{(\Pi/4)(.5)^2 X 4 bolts} = .15 \text{ ksi}$$

Since the ultimate shear stress for the steal bolts is 58 ksi. We are well within the safety factor.

Finally we must calculate whether these four bolts will rip through the aluminum angle plate. The angle plate is (.5) inches thick and must withstand a 9g's forward crash load. The bearing stress is calculated as before......

$$\sigma = \frac{\mathbf{20lbs X 9}}{\mathbf{(.5)^2 X 4 \text{ bolts}}} = \mathbf{.18 \text{ ksi}}$$

This calculation shows that indeed the bearing stress for the bolts is well within the safety factor of 27 ksi for the aluminum plate.

All calculations used in this structural analysis were taken from Mechanics of Materials by R.C. Hibberler.

Equipment Descriptions

Name: Lap top computer
Purpose: Data acquisition and experimental control
Power Requirements: 115 V AC, 60 Hz, 1.5 amps
Weight: 6 pounds

Name: Accelerometer
Purpose: measurement of acceleration
Power Requirements: N/A
Weight: 3 pounds

Name: Acoustic Horn
Purpose: To generate acoustic field and induce sonochemical reactions.
Power Requirements: 400 W from power supply.
Weight: 20 pounds

Name: Power supply
Purpose: To provide power to the ultrasonic horn.
Power Requirements: 115 V AC, 60 Hz, 1.5 amps
Weight: 30 pounds

Name: Spill container
Purpose: Safe storage of spilled solution
Power Requirements: N/A
Weight: 1.2 pounds

Name: Sonocation chamber
Purpose: Contain sample during experiment.
Power Requirements: N/A
Weight: <1 pound

Name: Coolant chamber
Purpose: To secure chemical cold packs
Power Requirements: N/A
Weight: <1 pound

Hazard Analysis

Our sample of palladium chloride presents no in-flight hazard. Palladium chloride, as described by the current MSDS, only poses as a hazard when solid. We will prepare our aqueous solution at The University of Washington so no handling of $PdCl_2$ in solid form will be required. Palladium chloride undergoes thermal decomposition at 500 degrees Celsius and releases chlorine gas. Although extreme temperatures are generated within the cavitation bubbles, the solution itself undergoes minimal heating due to the horn. Temperatures of the solution will not exceed 90 degrees Celsius so the danger of thermal decomposition is NOT present.

The possibility of a spill is highly unlikely. The quantity and concentration of $PdCl_2$ is small and will pose no danger in the event of such a spill. A spill will be cleaned up using a moist towel, which will then be stored in an air tight container.

Out Reach Program

We realize the importance of communicating our results to others. Therefore we will be presenting our research findings to local high schools to encourage youth to get involved in the growing fields within science and technology. We will also establish and maintain a website of our research. This website will contain our proposal, notes from our flight, and our analysis. We will continue to update the sight with our ongoing research in an effort to get others involved in researching topics such as sonochemistry. We also hope to present our findings to faculty and students of the University of Washington within the chemistry and physics departments.

Agreement to Comply

We agree to all program requirements and will respond promptly to any request for additional information regarding further details or safety issues of this experiment.

Signed______________________

And

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Apparatus Diagram

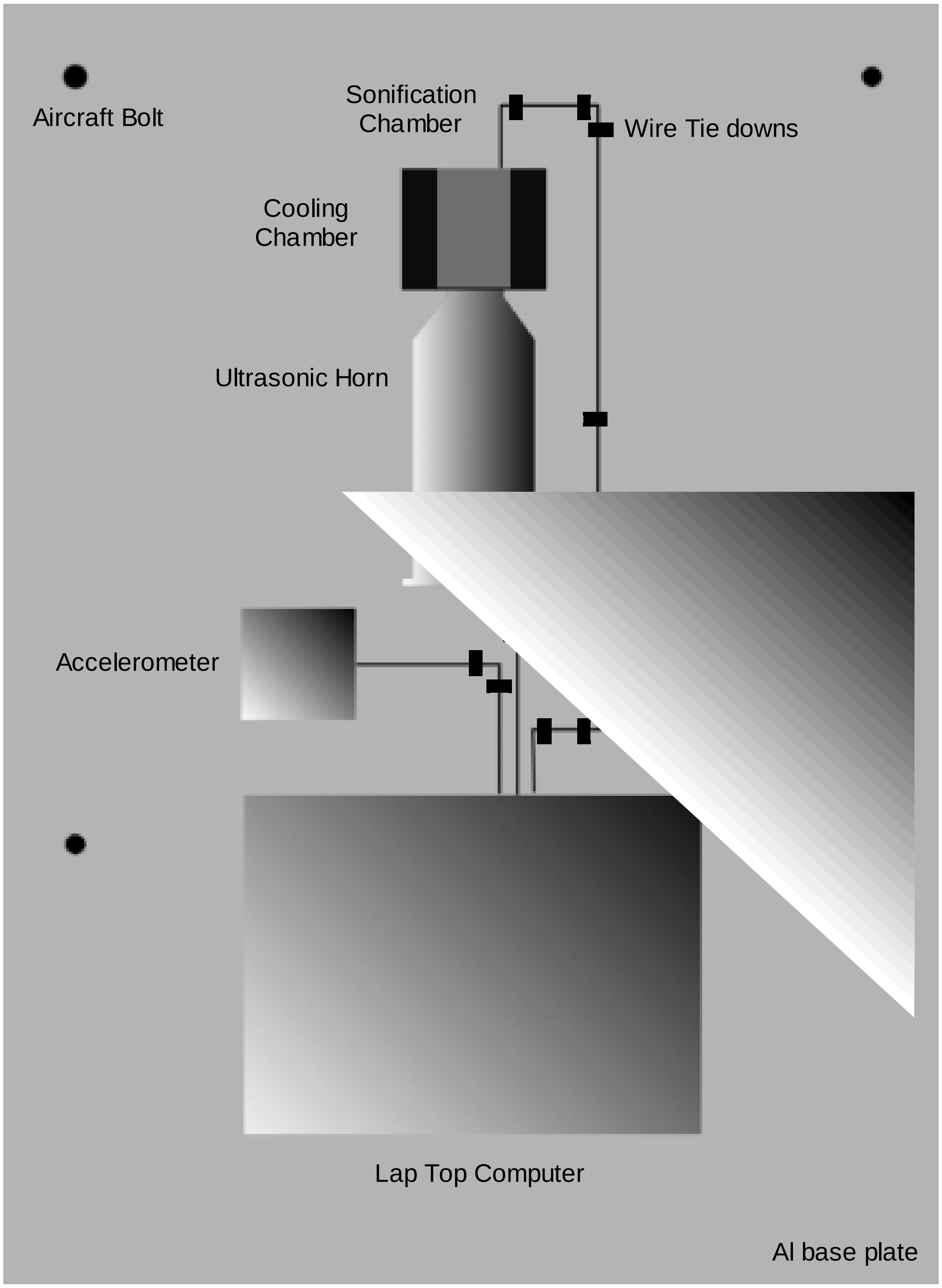

Figure 2. Our experiment will be done using a Lab View program on a lap top. The program will monitor the gravitational acceleration using the accelerometer. During micro gravity the program will turn on the ultrasonic horn. The program will also monitor the temperature of the sample. The .37 inch Al base plate will be held down with 4 aircraft bolts. All equipment will be bolted to the base plate and all wires will be tied down.