An experimental study of particle-bubble interaction and attachment in flotation

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ABSTRACT

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The particle-bubble interaction is found in industrial applications with the purpose of selective separation of materials especially in the mining industry. The separation is achieved with the use of bubbles that collect particles depending on their hydrophobicity. There are few experimental studies involving a single interaction between a bubble and a particle. The purpose of this work is to understand this interaction by the study of a single bubble interacting with a single particle. Experiments were conducted using ultra-pure water, glass particles and air bubbles. Single interactions of particles with bubbles were observed using two high speed cameras. The cameras were placed perpendicular to each other allowing to reconstruct the three-dimensional position of the particle, the bubble and the particle-bubble aggregate. A single size of particle was used varying the size for the bubbles. It was found that the attachment of a particle to a bubble depends on its degree of hydrophobicity and on the relative position of the particle and the bubble before they encounter.
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<tr>
<td>KAUST</td>
<td>King Abdullah University of Science and Technology</td>
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<tr>
<td>TPC</td>
<td>Triple phase contact</td>
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Chapter 1: Introduction to particle-bubble interaction

1.1 Overview

Interaction of different elements is found all around. An interaction can be understood as the influence that several elements have on one another. There are many examples of interactions such as how the water shapes the landscape and how the planets move in space by the influence of their gravity. Understanding how an interaction works can lead to control such interaction for something useful. There is a process that is used in the industry to separate materials called flotation. This process uses the interaction of bubbles with particles to selectively separate a desired material in the form of particles. Controlling the interaction allows to improve the process. In flotation, the particles are immersed in a liquid and bubbles are passed through it. Particles are a minute quantity of some material that if they happen to be close enough to a bubble, an influence of the bubble towards the particles and vice versa will take place. To understand the interactions taking place in the flotation process, a single interaction between a bubble and a particle needs to be analyzed. This interaction can have different outcomes depending on some conditions and parameters. The interaction can have as an outcome a slight change in the trajectories of the elements or a collision.
1.2 Interest in studying the topic

The particle-bubble interaction is currently found in many material-separation processes in the industry. Analyzing a simplified experiment of the flotation process, using one bubble and one particle, sheds light on their interaction. The analysis of flotation becomes more complicated when there are many particles and bubbles involved. Nevertheless, making such simplified experiment would allow to better understand the interaction process since there have not been many attempts of such a simplified analysis [1]. It is technically difficult to analyze one single interaction experimentally as both the bubble and the particle are in motion. This single particle-bubble experiments are needed for expanding the knowledge in the experimental part of the topic. Experimental data could lead to validation of the existing theory. The possibility to contribute to the available knowledge in this topic makes it interesting.

1.3 Motivation

For the investigation of the particle-bubble interaction, there have been few studies that were done based on experiments involving one single bubble and one or more particles, but in most of the experiments the bubble is kept from moving and the particle is falling freely on the bubble surface. The other type of experiments consisted of allowing a bubble to rise and having the particle in a fixed position. These experiments provided insight into the investigation but what is missing is that in the real interaction both, the bubble and the particle,
would be in free motion. Earlier there was an interesting experiment that involved a particle that was translated downwards by means of an automated translator while bubbles were rising and interacting with the particle [2]. This experiment represented better the real flotation process.

There is the opportunity to make an experiment that resembles the real process by having the bubble and the particle in free motion.

The particle-bubble interaction is involved in many processes that are used for separating materials, varying from the collection of coal and minerals of value to the recycling paper industry [1]. It is motivating to do research that can help to understand the separation process better so that improvements can be done in the current technology and techniques employed for the separation of materials involving the particle-bubble interaction.

1.4 Purpose and objectives

The purpose of this present work is to explore the interaction of one single particle and one single bubble in a fluid analyzing the whole interaction process to understand better the particle-bubble attachment. The experiment is representing the real process that happens in the industry by introducing the ideal case of having only one to one interaction. This interaction would require the particle to fall within the fluid just by gravity, and the bubble to ascend just by its buoyancy. The fluid will be water since most of the flotation process are
done based on water with some chemicals added. The objectives that are set to accomplish this ideal case analysis are outlined next:

- Summarize the available information about the particle-bubble interaction
- Create an experimental set up for conducting experiments in the ideal case
- Conduct experiments and analyze the results obtained
- Summarize the work and highlight findings in a thesis

These are the objectives representing the specific goals that need to be accomplished so that the purpose is fulfilled. Each one of the goals has its own challenges, but the experimental part is certainly the most challenging.
Chapter 2: Flotation process

2.1 Description

Material separation is important in the industry and, as it was mentioned before, in some of the separation processes that exist in the industry, there is interaction of particles and bubbles. This interaction is what makes the separation possible in the flotation process. This process works with the presence of particles and gas bubbles interacting in a liquid where the particles are attached to the bubbles and then carried up to the surface [3]. Usually the process of flotation is done in what are called flotation cells where a swarm of bubbles is generated to interact and collect the material particles present in the liquid phase where the bubbles are ascending.

What is important for flotation is that there will be particles that are more likely to attach onto bubbles depending on the properties of the particles. The properties vary depending on the material that the particles are made of. It seems that the most important factor for the attachment of the particles onto bubbles is the hydrophobicity of the particle surface, as one study showed that only the hydrophobic particles adhere to the bubble surface [4]. This is true when the liquid phase, where the interaction is taking place, is water. The flotation process can therefore separate hydrophobic particles from non-hydrophobic particles.

To consider the surface of the particle to be hydrophobic, the contact angle of water on the particle’s surface should be greater than 90 degrees [5]. Sometimes


the particles are naturally hydrophobic or sometimes they might need some treatment that would increase their likeliness to attach onto bubbles i.e. increase their hydrophobicity. In some industries, there is a need for controlling the surface behavior of the particles which is made possible by the use of reagents [6]. This promotes a more controlled separation of the materials as there will be particles that will attach onto the bubbles and others that will not, based on the use of the reagents.

There are many factors that affect the flotation process in the different industries as its use varies from one industry to another. This is mainly due to the different materials that are handled by each industry, which requires a tailored flotation process for each industry.

2.2 History of flotation

The flotation process has been a separation technique that has been used for more than 100 years, especially in the mining industry to extract the valuable minerals [7]. It is also called froth flotation in the industry. The process of froth flotation originated in Australia in the early 20th century [6]. Froth flotation concentrates the hydrophobic particles on the surface of the liquid in the form of a froth. Usually reagents are used in froth flotation to increase the material separation efficiency of the process [6]. Some of the reagents increase the hydrophobicity of the particles composed of specific materials, facilitating their separation by the bubbles.
For the first decades following its introduction, the flotation process was mostly used in the mining industry until it was adapted to other industries due to the high separation efficiency, simplicity of use, maintenance and cost effectiveness [8]. The oil industry and recycling paper industry are some of the industries that the flotation process was adapted to later on [9]. The industrial applications of the flotation process will be discussed in another section of this chapter. Currently the use of the flotation process is widespread and commonly used as an efficient technique for separating most minerals in the world [10]. The flotation process keeps on being adapted to more applications up to this day, for example to treat industrial waste water. Some of the available methods for treating industrial waste water have limitations. The adaptation of the flotation process has the potential to improve the water treatment processes [11].

The flotation process has been known for quite some time but the interest in the governing sub-processes in the particle-bubble interaction has just been studied for around 50 years [1]. There is still the need to understand better the process of flotation. It has been reported that the particle-bubble attachment process that has been investigated less than the collision of particles and bubbles [8]. It is the attachment what separates the hydrophobic particles from the non-hydrophobic ones. A better understanding of the flotation sub-processes could lead to expand its usage, improve the current technology and adapt it to more applications in the future.
2.3 Types of flotation

The process of flotation has been divided into two main types: mineral flotation and plastics flotation. The only distinction between the two is the relative size of the particles that are to be floated with respect to the bubbles. In the case of mineral flotation, the particles that will be floated are smaller than the bubbles and in the case of plastics flotation, the particles are simply larger than the bubbles [9].

The standard and traditional type of flotation is mineral flotation, where the particles are usually very small compared to the bubbles. The bubble size in mineral flotation for mineral processing is between 600 to 2000 micrometers in diameter and the particles that are needed to be floated vary from 10 to 150 micrometers [11]. There have been attempts to describing the process of mineral flotation. Sometimes the main focus is on the different efficiencies of the process (collision, collection and attachment efficiency) where the attention is on the overall behavior of the whole process and some other times the focus is on the particle-bubble interaction in the small scale by describing it with mathematical models. The first analysis of the interaction process was published in 1948 and it discusses the kinetics of the process [12]. More models were developed later for the calculation of the efficiencies corroborating them with experimental data.
In the case of the plastics flotation, the plastics particles are bigger than the bubbles and they are by nature hydrophobic [13]. This hydrophobicity is what allows the bubbles to attach onto the plastic particles and vice versa.

One of the challenges in plastics flotation consists in separating plastics among themselves by type. The use of reagents is needed to make some of the plastics more likely to attach bubbles so that flotation can efficiently separate them [13]. There has been less investigation on plastics flotation as it has not been used as much as mineral flotation in the industry. The available information on plastics flotation is mostly experimental data that does not provide a fundamental study of the process [2]. The models that have been developed for mineral flotation that were developed for the interaction of small particles and bigger bubbles generally are not suitable for the case of plastics flotation [2].

2.4 Steps in the process

To understand the interaction of particles and bubbles, a single particle interacting with a single bubble has to be analyzed. The particle-bubble interaction can lead to two outcomes; they both collide or they do not collide. In a few words, what happens in the process is that the particle approaches the bubble, then a thin liquid film forms between the two and starts to drain until the liquid film ruptures and forms a particle-bubble aggregate. This simplified explanation of the process assumes that the particle collides and attaches to the bubble.
To identify the particles that collide from those that do not, there is a concept called the grazing trajectory. In the case of mineral flotation, the grazing trajectory it is the path described by the particle that collides with the bubble and had the largest relative distance. The relative distance is the horizontal distance from the bubble’s centroid to the particle’s centroid. The grazing trajectory has a critical relative distance related to it. In theory, the particles that have a smaller relative distance than the critical relative distance will collide with the bubble. Those particles that have a larger relative distance than the critical one, will not collide.

The same concepts apply to plastics flotation. The difference is that the grazing trajectory is described by the bubbles instead of the particles. When there is no collision, the particle’s trajectory is changed by the ascending bubble or in the case of plastics flotation, the bubble’s trajectories are affected by the particle. Two possibilities exist when a collision happens: there is attachment between the particle and the bubble or there is no attachment. It has been reported that the quality of the particles’ surface does not affect the collision [2]. This would imply that the collision would not be affected regardless if the particle is hydrophobic or not. The steps involved in the interaction will be explained later.

The collision is the first step in the particle-bubble interaction process. The particle-bubble interaction steps are collision, attachment and stability [9]. Other
authors have defined the steps to be collision, attachment and detachment [14]. Nevertheless, the detachment does not always happen as the attachment can result in a stable particle-bubble aggregate. It is therefore preferred to use stability instead of detachment as the third step of the interaction process since stability is more comprehensive.

The steps in the particle-bubble interaction process are applicable to both types of flotation. It is worth mentioning that the collision itself does not guarantee attachment, since the particles can collide with the bubble and slide on the surface without attaching. The particles can also bounce off the bubble due to the deformation of the bubble’s surface.

2.4.1 Collision

The collision is the step when a particle and a bubble approach themselves for their encounter until a very small distance that is of the sub micrometer order [14]. This distance is defined to be the zonal boundary of the long-range hydrodynamic forces and the interfacial forces interacting between the particle and the bubble [14]. For mineral flotation, three zones around the bubble have been identified, where different forces arise in each one of them [15]. These zones are not discrete and they rather overlap one another. In the outer zone, the long-range hydrodynamic force field around the bubble and the fluid mechanics of the particle are the governing forces [14]. The particle passes through this zone when it is in the collision step.
The three zones are showed in Figure 2.1, where zone one that is the outer most zone. Zones two and three form part of the attachment step of the particle-bubble interaction process.

![Figure 2.1: Zones around the bubble.](image)

In Figure 2.1, Zone 1 is the outermost zone where the particle passes as it gets closer to the bubble. Zone 2 is the intermediate zone. This zone’s shape is affected by the wake behind the bubble. Zone 3 is the closest zone to the bubble where the interfacial forces affect the interaction.

The surface forces that act between the particle and the bubble include the van der Waals force, the electrical double layer and hydrophobic forces [16]. The surface forces can be measured using atomic force microscopy (AFM) for different particles. There are some studies showing that the hydrophobic particles have the presence of surface forces that hydrophilic do not have using the AFM technique [17]. The electrical double layer and van der Waals forces are very important for colloidal particles. For larger particles, these forces are still
present but they become important in close proximity of the surface of the particle.

2.4.2 Attachment

Attachment is a the most important step of the process concerning the separation of the hydrophobic particles from those that are hydrophilic. The attachment step can be divided into three elementary steps [18]:

- Thinning of the intervening liquid film
- Rupture of the intervening liquid film and formation of a triple phase contact (TPC) nucleus
- Expansion of the TPC to form a stable wetting perimeter

The attachment has a critical parameter related to it called induction time. The induction time is the time that it takes for the three elementary steps to occur in a given interaction of a particle and a bubble [14]. Sometimes the induction time is also called the attachment time. The elementary steps are explained below:

1. Thinning of the intervening liquid film.

   After collision, an intervening liquid film forms and starts to thin reaching a critical thickness just before the rupture of the film. This is considered to be the first elementary step.

   Going back to the description of the zones around the bubble in figure 2.1, zone two is where the thinning of the intervening liquid film starts. There are forces resulting from the concentration gradient of ions on the bubble
surface that help with the thinning of the liquid film in zone two. The ion concentration gradient is the result of the flow around the bubble and it is related to the electric field intensity giving rise to forces of an osmotic nature [15]. The electric field leads to the presence of electrophoretic forces that either attract the particle to the bubble surface or repel it from it [19]. The ion concentration gradient depends on the surface mobility of the bubble. A mobile surface has no resistance in its motion (no tangential stress). An immobile surface is one that acts as if it was a solid surface having the no-slip boundary condition [20]. The surface mobility is affected by the presence of impurities in the liquid that are taken by the bubble on its ascension to the surface. In the case of the flotation process, the impurities can come from the reagents used for improving the collection efficiency of the process when separating materials.

Zone three, that is also part of the first elementary step, is characterized by the influence of the forces that depend on the thin film thickness. These forces come from what is known as the disjoining pressure. The disjoining pressure is composed of three terms. One term is related to the overlap of the ionic atmospheres of both surfaces, another term is the specific attraction of the molecules from the liquid film and the particle and the last term depends on the particle’s surface hydrophilicity [15]. When the disjoining pressure is positive, it means that the particle is being pushed
away from the bubble’s, and when it is negative, the particle is being attracted towards the bubble surface.

Analyzing this elementary step from the point of view of the forces acting on the particle that is approaching the bubble surface, there are several forces that influence the process. These forces are surface, drag, gravitational, inertial and buoyancy forces. The surface forces act in the radial direction with respect to the bubble’s center using a polar coordinate system, and they are the characteristic forces of this first elementary step [18]. Among the surface forces the hydrophobic force is included. This force has not yet been completely understood but, it is known that, the more hydrophobic the particle, the stronger the hydrophobic force is [21]. The contact angle of water on the particle surface can be used to quantify the hydrophobicity of the particle, the bigger the contact angle, the more hydrophobic the particle.

The drag force can have many components among which the steady drag force is of more interest for the attachment process. The steady drag force acts in the radial direction and prevents the particle from getting closer to the bubble [18]. The buoyancy force is an upward force created by the presence of the particle immersed in the liquid and is equal to the weight of the liquid displaced by the particle’s volume. The gravitational force comes from the particle’s weight and the inertial force comes from the
mass of the particle. The inertial force sometimes causes the particle to deviate from the streamlines around the bubble. These forces are shown in Figure 2.2.

![Diagram of forces acting on a particle](image)

**Figure 2.2:** Forces acting on the particle.

In Figure 2.2 $F_{dp}$ represents the particle's drag force, $F_{bp}$ the particle's buoyancy force, $F_s$ the surface forces, $W_p$ the weight of the particle and $F_{ip}$ the inertial force.

2. Rupture of the intervening liquid film and formation of a TPC nucleus.

The rupture of the liquid film is not totally understood, there are some theories that try to explain what is the mechanism that causes this rupture. Some of the mechanisms that are thought to cause the rupture are growing capillary wave fluctuations, rapid instability due to the fluctuation in the density within the film and rapid instability due to unadjusted balance of the surface energy [18].

The formation of the TPC nucleus has a critical radius once it forms. There is a theory that relates the critical radius of the nucleus to the critical
thickness of the film. This theory is based on the free energy balance of the intervening liquid film before and after the rupture [18]. According to the theory, the formation of a TPC nucleus with a radius smaller than the critical radius predicted by the theory is thermodynamically impossible. Using this theory for particles that have surfaces with different degrees of hydrophobicity, shows that hydrophobic surfaces have a smaller critical radius than less hydrophobic surfaces [18]. This means that TPC nuclei can form easier with hydrophobic surfaces based on a thermodynamic analysis of the surface energy balance. The predictions of this theory agree with the previously stated premise that hydrophobic particles are more prone to attach onto bubbles in the flotation process.

3. Expansion of the TPC to form a stable wetting perimeter

Once the TPC nucleus is formed, an expansion of the TPC will take place. The TPC expands fast initially after the nucleus is formed and the velocity of expansion is the greatest at the beginning and slowly decreases as the TPC attains a stable wetting perimeter [18].

One of the driving forces in the expansion is the capillary force due to the surface tension of the air-liquid interface.

2.4.3 Stability

When the TPC expands attaining the stable wetting perimeter is when the stability step begins. The union of the particle and the bubble is called an
aggregate. This aggregate can be stable or unstable. The stability of the particle-bubble aggregate is determined with the tenacity of the aggregate. The tenacity is the summation of the forces that prevent the separation of the particle from the bubble. The forces that contribute to the tenacity are the capillary force, hydrostatic pressure force and the buoyancy force [14]. These forces are called adhesive forces and they act on the triple phase contact line (TPCL) of the aggregate. The capillary force comes from the surface tension. The hydrostatic pressure force is the result of the pressure exerted by the liquid surrounding the aggregate on the area enclosed by the TPCL. The buoyancy force is the weight of the volume of liquid displaced by the volume of the particle. Figure 2.3 shows the forces acting on the TPCL.

![Figure 2.3: Forces acting on the TPCL of a particle-bubble aggregate.](image)

In the figure 2.3, the adhesive forces are the capillary $F_c$, the hydrostatic pressure $F_h$ and the particle buoyancy $F_{bp}$ forces. The detaching forces are the particle weight $W_p$ and the turbulent inertial force $F_{ti}$. 
The other forces that influence stability are the detaching forces that promote the separation of the aggregate. The most important detaching forces are the particle weight and the turbulent inertial forces [14]. When the tenacity of the aggregate is larger than the summation of the detaching forces, the aggregate is stable. This is certainly desirable for the flotation process. When the detaching forces are greater than the tenacity, the aggregate is unstable. The turbulent inertial forces are sometimes the result of the dynamic conditions present in the flotation cells used in the industry [14].

2.5 Industrial applications

The process of flotation is used in many industries for the selective separation of materials. It is used to recover valuable minerals and coal present in ores, separate pollutants from waste water, separate plastics for recycling, de-inking of paper pulp for recycling as well, among other applications [1]. It is also applied for the separation of oil sands [2], de-oiling in heavy oil exploration, extraction of bitumen from oil sands and removal of fine particles in domestic and industrial waste water [9]. The flotation process was also tested for recovering oil from a sludge, proving that it could be a possible separation technique for this purpose [22].

The first industrial application of flotation was in the mining industry where it is still used. The process used in the mines for applying flotation consists of first crushing the ores to make particles smaller than 200 micrometers so that the
minerals are liberated, then they are mixed with some medium that is usually water. After, reagents are used to modify the mineral particles’ hydrophobicity so that they can attach to the bubbles generated in the flotation cells [23]. The usual bubble size employed for mineral flotation in the mining industry used for the separation of minerals is of 600 to 2000 micrometers in diameter [11]. The usual size of the mineral particles that need to be floated is between 10 and 150 micrometers in diameter [11]. In the mining industry, flotation has high separation efficiency, is cost-effective and the equipment that employs requires simple operation and maintenance [8].

Reagents are important for the flotation process as they are used to control the conditions inside the flotation cell. There are six main types of reagents: frothers, collectors, modifiers, activators, depressants and flocculators [6]. Frothers are used to control the size of the bubble and the stability of the froth. Collectors are used for making the particles hydrophobic as they are surface-active reagents. Activators promote the adsorption of collectors into a type of mineral particle. Depressants prevent the adsorption of the collector on some mineral particles. Modifiers control the flotation environment and flocculants help to dewater the flotation concentrates [6].

The process of flotation is not only used for particles that are smaller than the bubbles, but is also used for plastic particles that are bigger than the bubbles. Plastics flotation is being studied as a solution for separating the different types
of plastics for their further processing and recycling [13]. Most of the plastics are already hydrophobic and to implement flotation for their separation would require the addition of reagents that renders some types of plastics hydrophilic to have a selective flotation [13]. In this case, the use of depressants will be needed to make some of the plastics hydrophilic. Some of the plastics commonly used are polyvinyl chloride, polycarbonates, polyacetal and polypropylene ether [11].

The different applications of flotation require different sizes of bubbles. The size of the bubble can vary depending on how the bubble is formed. For the generation of bubbles in flotation cells, there are some conventional techniques used and they are listed in the following list with their main usage [11]:

- Electro-flotation: used to remove ink, oil and pigments from water.
- Dispersed or induced air flotation: used for mineral processing and oil-water separation.
- Dissolved air flotation: used for treatment of waste water effluents and very fine mineral particles.

There are other emerging techniques that are not widely used but they have potential for different applications. These emerging techniques are: nozzle flotation, column flotation, centrifugal flotation, jet flotation and cavitation air flotation [11].
2.6 Flotation efficiencies

For a successful particle recovery from a sludge in flotation the three steps of the particle-bubble interaction must take place. For each one of the steps, an efficiency (or probability) can be assigned depending on the overall behavior of the interaction of particles and bubbles. In the case of the collision step, a collision efficiency is defined to be ratio of the actual rate of collisions to the ideal rate of collisions [14]. For the attachment step, an attachment efficiency can be calculated as being the rate of particles that attach to a bubble divided by the rate of particles that collided with the bubble. The attachment efficiency depends on the success of the three elementary steps needed for attachment. The stability efficiency (or stabilization probability) is the probability of a particle-bubble aggregate to remain that way by the tenacity during the stability step.

The multiplication of the three efficiencies gives the collection efficiency that is also called the flotation probability [7]. This overall efficiency can be used to measure and evaluate the performance of a flotation process.
Chapter 3: Experimental

3.1 Particles and fluid

In the froth flotation process the particles are of different sizes and irregular form. For the experiments of this work, the particles employed were spheres. These spheres were soda lime glass spheres (Cospheric SLGMS 1000-1180um). The size of the spheres varies is in the range of 1000 to 1180 micrometers in diameter. According to the manufacturer more than 90% of the spheres are within the size range.

The particles were given different treatments to achieve various degrees of hydrophobicity. The types of particles that were used to make experiments were the following:

- Low hydrophobicity
- Smooth hydrophobic
- Rough hydrophobic
- Superhydrophobic particles

Each one of these types followed a different treatment and preparation.

The fluid used for the experiments was ultra-pure water at 25 degree Celsius. The water was freshly dispensed from a Milli-Q® Academic purification system (Millipore corporation) just before the experiments.
3.2 Particle treatments

There were four types of particles for the different experimental sets.
Nevertheless, there are more than four particle treatments as one of the treatments was only used for comparing the influence of the surface roughness on the attachment.

3.2.1 Treatment 1: Low hydrophobicity

This treatment was used to obtain the low hydrophobic particles. The steps are the following:

1. Cleaning the particles. The particles were deposited in a little clean glass container with acetone. The container was placed for one minute in an ultrasonic cleaner (Branson 2510). After that, the acetone was removed from the container leaving only the particles inside. Ethanol was poured into the container with the particles. The container was placed for one minute in the ultrasonic cleaner. The ethanol was removed after the ultrasonic cleaning. Isopropyl alcohol was poured into the container. The container was placed in the ultrasonic cleaner for one minute. The alcohol was removed right after, but there is some alcohol remaining in the container. The particles were fully dried with the aid of pressurized nitrogen gas applied directly to them.

2. Washing the particles. The particles were transferred into a clean glass container. Ultra-pure water was poured into the container with the
particles. The container was placed in the ultrasonic cleaner for a minute. After that, the water was removed using a syringe without extracting the particles. This step was repeated two times.

3. Wetting the particles. The particles had to be placed in a small glass container and ultra-pure water was added on top of them in droplets with a syringe. Some of the particles would stay in the bottom of the container, but others would remain in the surface. To sink the particles at the surface, droplets of ultra-pure water would be added on them until the particles would sink. Sometimes letting the droplets to fall from a higher level would work better.

4. Removing bubbles from the particles. When all the particles are at the bottom of the container, they usually have bubbles attached to their surfaces. To remove the bubbles, the container is placed in the ultrasonic cleaner for one minute. This process removes the bubbles from the surface of the particles.

5. Placing the particles on the funnel. A syringe connected to a capillary is used to move the particles from the container to the funnel where they are released for the experiments. The capillary has an inner diameter of 1.5 mm so that the particles fit loose inside. The capillary is introduced into the container and it is placed over one of the particles. The syringe piston is then pulled to extract a particle with some water from the container.
Then the capillary with the syringe is moved rapidly to the funnel, placing the tip of the capillary where the particle must be. The syringe piston is pressed slowly to push the particle out of the capillary.

3.2.2 Treatment 2: Smooth hydrophobic

The silanization of the particles is another surface treatment that makes the surfaces hydrophobic. The process consists of the deposition of a silane reagent that adheres to the surface of the particles. The silane reagent used was Trichloro (1H, 1H, 2H, 2H perfluoroocetyl) silane 97% (by Aldrich). The steps for this treatment are the following:

1. Cleaning the particles. The particles were deposited in a little clean glass container with acetone. The container was placed for one minute in the ultrasonic cleaner. After that, the acetone was removed from the container leaving only the particles inside. Ethanol was poured into the container with the particles. The container was placed for one minute in the ultrasonic cleaner. The ethanol was removed after the ultrasonic cleaning. Isopropyl alcohol was poured into the container. The container was placed in the ultrasonic cleaner for one minute. The alcohol was removed right after, but there is some alcohol remaining in the container. The particles were fully dried with the aid of pressurized nitrogen gas applied directly to them.
2. Plasma-cleaning. Once the particles were dry, they were transferred into a clean beaker of 80 milliliters of capacity. The beaker was placed in the plasma cleaner (Harrick Plasma PDC-002) for three minutes.

3. Deposition of the silane reagent. The beaker with the particles was placed in a desiccator that was inside a fume hood. 10 microliters of the silane reagent were placed on the sides of the beaker in the form of two or more droplets using a pipette. The tip of the pipette has to be washed with water abundantly after usage so that it can be discarded properly. Care has to be taken to avoid the silane reagent drops to make contact with the particles at the bottom of the beaker. A parafilm piece was used to cover the top of the beaker. After that, the desiccator was closed and the air was evacuated from it. The particles were left for 40 minutes in vacuum to promote the deposition of the vapor phase of the silane reagent on their surface. All this step was done under the fume hood using personal protective equipment.

4. Removal of excess coat. After the vapor deposition, the particles are sticky and it is impossible to use them in that way for the experiments. To remove the stickiness, the particles are placed in a clean small container with ethanol. The container was closed and shaken manually for around 30 seconds. The ethanol was removed after. The step was repeated two times.
5. Final drying. After removing the ethanol, the particles were dried with the aid of pressurized nitrogen gas. Once dry, they were placed in a clean glass container.

6. Trapping the particles. Water is placed in a small container. One particle is placed on the surface of water and it is sucked using the capillary-syringe device mentioned in step five of the previous treatment. Quickly the capillary is placed on the bottom of the container making sure that the particle is trapped inside the capillary.

7. Removing the bubbles from the particles. Water is contained inside the syringe and is pushed out through the capillary. The particle remains trapped inside the capillary with the bottom of the container. The water is forced to pass around the particle through a small area, removing the bubbles from the surface of the particle. This step may be repeated several times until the particle looks clean from bubbles.

8. Placing the particles on the funnel. The particle is transferred from the small container using the capillary-syringe device. The particle is taken out of the capillary by pushing water out of the syringe. The particle should remain inside the water at all times.

3.2.3 Treatment 3: Rough hydrophobic

This treatment allows to obtain the particles coated with Ultra Glaco. The steps are the next ones:
1. Roughening the surface of the particles. The particles were roughened individually using a two sand paper pieces (FEPA P 1000). One piece is used on top and the other on the bottom of the particle. The process consisted in rolling the particle on the sand paper in a circular fashion. This would be done until the surface of the particle would look cloudy and opaque.

2. Cleaning the particles. The particles were deposited in a little glass container with acetone. The container was placed for one minute in the ultrasonic cleaner. After that, the acetone was removed from the container leaving only the particles inside. This was repeated three times. After the acetone cleaning, ethanol was poured in the container with the particles. The container was placed for one minute in the ultrasonic cleaner and then the ethanol was removed. After the ethanol, isopropyl alcohol was used in the same way as the ethanol. After removing the isopropyl alcohol, the particles were fully dried with the aid of pressurized nitrogen gas.

3. Plasma-cleaning. Once the particles are dry, they were transferred into a clean small beaker. The beaker was placed in the plasma cleaner for three minutes.

4. First surface coating. The coating agent used (Ultra Glaco by Soft 99 Co.) was added to the particles covering their surface. The Ultra Glaco coating
agent dries quickly when exposed to the atmosphere. With the particles used, it takes more time for the Ultra Glaco coating to dry in areas between the particles. This is the reason why the drying process was aided with the use of pressurized nitrogen gas.

5. First heat treatment. When the Ultra Glaco coating has dried, the particles were placed inside a furnace at 80 degrees Celsius for 10 minutes. After that, the particles were removed from the furnace and let to cool down at room temperature.

6. Second surface coating. Another Ultra Glaco coating was applied on the particles in the same way as before.

7. Second heat treatment. The particles were placed for 10 minutes at 130 degrees Celsius inside the furnace. After the 10 minutes, they were taken out and let to cool down at room temperature.

8. Follow steps 3, 4 and 5 from treatment 1.

3.2.4 Treatment 4: Superhydrophobic particles

For having superhydrophonic particles, the steps used are the following:

1. Cleaning the particles. The particles were deposited in a little clean glass container with acetone. The container was placed for one minute in the ultrasonic cleaner. After that, the acetone was removed from the container leaving only the particles inside. Ethanol was poured into the container with the particles. The container was placed for one minute in the
ultrasonic cleaner. The ethanol was removed after the ultrasonic cleaning. Isopropyl alcohol was poured into the container. The container was placed in the ultrasonic cleaner for one minute. The alcohol was removed right after, but there is some alcohol remaining in the container. The particles were fully dried with the aid of pressurized nitrogen gas applied directly to them.

2. First surface coating. The particles were transferred into another clean container. Glaco Mirror Coat “Zero” (by Soft 99 Co.) was added to the particles making sure that it covers all their surface. The Glaco coating was fully dried using pressurized nitrogen gas.

3. First heat treatment. The particles were placed inside a furnace at 80 degrees Celsius for 10 minutes.

4. Second surface coating. A second coating of Glaco Mirror Coat “Zero” is done on the particles in the same way as the first surface coating.

5. Second heat treatment. The particles were placed for 10 minutes at 130 degrees Celsius inside the furnace. After the 10 minutes, they were taken out and let to cool down at room temperature.

6. Follow steps 6, 7 and 8 of Treatment 2.

3.2.5 Treatment 5

This treatment was not used for the main sets of experiments. The purpose of this treatment is to analyze the effect of the surface roughness on the attachment
of bubbles. The steps are repeated from some of the other previous treatments.

This treatment starts is the same as treatment three except that step number one is skipped.

3.3 Particle characterization

The surface of the particles need to be characterized to compare their hydrophobicity. The particles used for the experiments included those prepared by the first four treatments. To compare their hydrophobicity, the particles were placed carefully on the surface of ultra-pure water. A photo was taken of the particle on the water surface. In Figure 3.1, the particles with the different treatments are shown.

![Figure 3.1: Treated particles and slides.](image)

In Figure 3.1, the panels (a-d) are water drops lying on treated slides. Panels (e-h) are treated particles on a water surface. In this same Figure, the treatment for the
particles and the slides is the following: treatment 1 for (a) and (e), treatment 2 for (b) and (f), treatment 3 for (c) and (g), and treatment 4 for (d) and (h). The hydrophobicity of the particles’ surface will try to keep the particle out of the water. Therefore, the most hydrophobic particle is in Figure 3.1 (h) as it is the one that floats the most on the water.

Glass slides were prepared using the same treatments to represent the surfaces of the particles. The glass slides went through the same preparation steps as the particles. After, an ultra-pure water drop of ten microliters was places on the slides. The images of the drops on the glass slides are shown in Figure 3.1 (a) to (d).

The more hydrophobic the surface is, the more the water drop reduces contact with the glass slide. The glass slide with treatment 4 is the most hydrophobic.

The hydrophobicity of a surface can be quantified with the contact angle. This is difficult to measure on the surface of the particles. Instead the contact angle was measured on the glass slides using a plugin on the ImageJ software called drop analysis made by [24]. The measured angles appear in table 3.1.

<table>
<thead>
<tr>
<th>Surface treatment</th>
<th>Advancing contact angle</th>
<th>Receding contact angle</th>
<th>Contact angle with standard deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Treatment 1</td>
<td>54.3°</td>
<td>21.9°</td>
<td>38.1° ± 17.9°</td>
</tr>
<tr>
<td>Treatment 2</td>
<td>110.1°</td>
<td>86.4°</td>
<td>98.2° ± 13°</td>
</tr>
<tr>
<td>Treatment 3</td>
<td>117.6°</td>
<td>79.6°</td>
<td>98.6° ± 21.3°</td>
</tr>
<tr>
<td>Treatment 4</td>
<td>174.9°</td>
<td>158.5°</td>
<td>166.7° ± 10.3°</td>
</tr>
</tbody>
</table>
Table 3.1: Contact angles for treated surfaces.

These contact angles are just an estimation for the contact angles of the particles. The values obtained from the measurements are in good agreement with the qualitative observations on the hydrophobicity of the particles in Figure 3.1. The hydrophobicity of the particles goes down as they are exposed to the ambient humidity present in the laboratory. This was confirmed by measuring the contact angle of freshly hydrophobized particles and particles that had 5 days after their surface treatment. The contact angle of the freshly hydrophobized particles was larger than the particles that were hydrophobized five days before. It is because of this that the particles were immediately used after their treatment.

3.4 Bubbles

The bubbles used in the experiments were always air as in the flotation process used in the industry. Nozzles were used for the generation of individual bubbles. Depending on the desired size of bubble, the nozzle would be different. The nozzles were made from borosilicate glass capillaries. The capillaries were modified using a puller machine (Micropipette puller P-1000 from Sutter Instrument Company). The machine heats up a small section of the capillaries and pulls it while the glass is malleable until it splits it. This would create a tapered capillary with a very tiny tip. The tapered tip is cut making a nozzle. The smaller the tip’s cross section of the nozzle, the smaller the bubbles that it can
create. The nozzles are tested in ultra-pure water by blowing air through them and measuring the size of the released bubbles. If the bubble needed to be bigger, then the nozzle’s tip would be cut again and tested.

The empirical procedure of making the nozzles would be to cut the tip of the nozzle by small distances and test it after every cut. This would allow to attain the targeted size of the bubbles with more precision. For the bubbles that were bigger than the particle, adding Ultra Glaco to the nozzle would help to release individual bubbles. This was done by wetting the nozzle’s tip with Ultra Glaco. The Ultra Glaco was dried with pressurized nitrogen gas leaving the tip hydrophobic. No heat treatment was used. The Ultra Glaco coat would make the air phase prefer to stay inside the nozzle. This would allow to release individual bubbles with the appropriate pressure inside the nozzle.

3.5 Cameras and lenses

Two cameras were used for recording the interaction of the particles and the bubbles. The Vision Research Phantom v710 color camera was used as one of the cameras. The v710 camera has a Complementary Metal Oxide Semiconductor (CMOS) sensor. The sensor allows to have a frame size of 1280 by 800 pixels recording at 7500 frames per second with a 12-bit pixel bit-depth. The other camera was a Photron FASTCAM SA5. The SA5 has a CMOS sensor that records in the monochromatic scale with a pixel bit-depth of 12-bit. It can record with its full frame size of 1024 by 1024 pixels at 7000 frames per second.
The experiments were recorded with a frame size of 512 pixels by 800 pixels. The frame rate was of 7500 frames per second and the exposure time was of 100 microseconds. The lenses adapted to the cameras were the Leica Z16 APO lenses (Leica Microsystems). The zoom provided by the lenses was slightly adjusted depending on the experiments.

The software used to control the cameras was the Photron FASTCAM Viewer for the SA5 camera. For the Phantom v710 camera was the PCC 2.7. After an experiment was done, the video taken would be trimmed and saved using the same software. The Photron FASTCAM Viewer software was useful for doing the pixel correlation for both cameras when processing the videos.

3.6 Experimental set up

The equipment that was needed to record the experiments included the two cameras with the microscope objectives adapted to them (see Figure 3.2). For the triggering system, a laser diode (THORLABS CPS635R), a photodetector (THORLABS PDA10A-EC), a beam reducer and a mirror were used. To control the signal from the triggering system, a digital delay generator was used (Stanford Research Systems DG645). A trigger box served to power an electric motor. The motor was used to either release bubbles or particles by driving a syringe piston or by pulling a string that would release the particles. For the illumination, two metal halide illumination systems (Sumita LS-M352) were placed to provide the recording area with proper lighting. Other mechanical
components employed were jacks, micrometer translating stages, posts, joints, sliders, rails, among others. Figure 3.3 shows the main components of the experimental arrangement in the laboratory.

Figure 3.2: Experimental arrangement diagram.
3.7 Triggering system

The particle-bubble interaction happens in a fraction of a second. One of the important challenges is capturing the interaction inside the area that the cameras are recording. This requires to control the release of the particles and the bubbles, as well as the cameras. The triggering box powers an electric motor that drives a syringe piston to push air. The syringe is connected to the nozzle and the air is released in the form of bubbles. This trigger box is controlled by a signal that comes from the digital delay generator.
The initial signal comes from the photodetector that detects the laser diode light. When the laser light is blocked, the photodetector sends the signal to the digital delay generator. This device triggers the cameras and the trigger box. The laser light is blocked when the particle falls. This is the case when the release of the bubble depends on the particle because the falling particle blocks the laser light and triggers everything else. This arrangement for triggering is called arrangement one and it is shown in Figure 3.4 (a).

Figure 3.4: Arrangements for the triggering system.

For some experiments, it is needed that the bubble triggers the system. This is because small bubbles take a longer time to rise. This creates the need to delay the release of the particle to capture the interaction in the recording area of the cameras. In this case, the bubbles are generated using a simple syringe full of air connected to the nozzle. The syringe is pushed until a single bubble comes out of the nozzle. The laser diode module along with the photodetector are relocated just above the tip of the nozzle as represented in Figure 3.4 (b). When the bubble
is released it cuts the laser light. In this arrangement, the release of the particle is by mechanical means.

For this second arrangement, the particle is kept from sinking in the tank by a rod that prevents it from moving inside the funnel. The particle has to be preloaded into the funnel in advance. A piece of plastic string connected to the rod is pulled by the driving motor that is controlled by the trigger box. When the trigger box receives the signal, the motor pulls the plastic string. When the plastic string is pulled, the rod moves and the particle falls. A delay has to be set when triggering the cameras and the mechanical release mechanism. This second arrangement is called arrangement two and it is shown in Figure 3.4 (b).

3.8 Conducting experiments

Before conducting the experiments all the particles that were going to be used were previously prepared. The preparation of the particles was always done just before doing the experiments and it is explained by steps in another section.

Once the particles were ready, the final alignment and calibration of the cameras would be done. Following this, several experiments would have to be run to set the delays in sending the signals to the equipment in the delay generator. This is needed to make sure that the interaction of the bubble and the particle is happening in the area where the cameras are focused.
3.8.1 Alignment and calibration

The alignment of the cameras would be done using a quartz rod immersed in the container. The cameras’ resolution would be set to be the same. The control of the cameras was done from the software that they have in the computers. The rod would be found in the recording areas of both cameras. The zoom provided by the Leica lenses would be changed until the rod would appear to be of the same size for both cameras and then it would be changed to attain a very similar pixel correlation for both cameras. The height of the cameras was then adjusted using the rod in several positions until both cameras would be recording at the same height. A snapshot is taken having the quartz rod focused in the middle for both cameras. With the snapshot (see Figure 3.5) the pixel correlation could be obtained for each one of the cameras. This is the equivalence of the size of a pixel in the video to the real distance. A typical pixel correlation was 12.5 micrometers per pixel.

![Image](image.png)

Figure 3.5: A cropped image used to find the pixel correlation.
3.8.2 Release of bubbles

The bubbles were released by two methods. The first was using the motor that would drive the piston of a syringe pushing air into the nozzle. This would be useful for releasing bubbles larger than the size of the particle. Individual bubbles were released with repeatability. This release method was used in conjunction with the triggering arrangement one shown in Figure 3.4 (a) when the particle is used to block the laser light for triggering the cameras. Depending on the size of the nozzle, the parameters in the trigger box would have to be adjusted to release individual bubbles.

The second method was to manually use a syringe connected to the nozzle and push air out of it. This method was particularly useful for the generation of bubbles smaller than the size of the particle. The key to release one single bubble at a time was to control the piston of the syringe carefully. Empirically, what worked best was to quickly push the piston of the syringe and then release it. This is basically giving a peak pressure on the air inside the nozzle. Most of the times it would require several attempts to release one bubble. This empirical method resulted repeatable, allowing to make individual bubbles as small as 200 micrometers in diameter. The method was used only when the bubble would trigger the cameras, that is, using the triggering arrangement two (see Figure 3.4 (b)). The particle would be placed in advance on the funnel and then it would automatically be released after the bubble had passed through the laser light.
Chapter 4: Data analysis and results

4.1 Image processing

The important information to extract from the videos included the dimensions of the particle, the dimensions of the bubble and their centroids. In order to extract this information, several codes were developed. The software used for this purpose was MATLAB. The analysis required to process the video by analyzing individual frames. Each frame would be obtained from the video file. With the use of the image processing toolbox of MATLAB, filters and algorithms were used to convert the frame into a binary image. An extracted frame is shown in Figure 4.1 (a) and its binary image containing only the shapes of the particle and the bubble as seen Figure 4.1 (b).

![Figure 4.1: A video frame with its binary image.](image-url)
To obtain the shapes of the particle and the bubble as they were needed, the parameters of the algorithms were constantly modified. This depended especially on the illumination of the recording area as the parameters are sensitive to the intensity of the pixels in the original frame. From the binary image is easier to find points at the edge of the bubble or the particle that allow to detect their shape. One of the codes was developed to find these points at the edge by evaluating the intensity values of the pixels in the image. These evaluations were done along lines that start radially from a manually-selected point inside of the shape. Along each line, one point can be found at the edge of the shape. 20 lines were used to find 20 points along the edge of the shape of the bubble or the particle.

The points were used to fit an ellipse in the case of the bubbles (see Figure 4.2) or a circle for the particles. The fitting technique that the codes used was the linear least squares for both the circle and the ellipse. The code used for fitting an ellipse is found in Appendix A and for fitting a circle in Appendix B.
Figure 4.2: Ellipse fitted to a bubble shape.

The functions used to fit the ellipse or the circle give the location of the centroid and the parameters that define the shape. With the parameters, the volume of the bubble and the particle can be calculated. The relative position between the bubble and the particle can be obtained evaluating the videos from both cameras using the location of the centroids. Figures showing the initial positions of the particle and the bubble were constructed to analyze the outcomes of the interaction.

Trajectories can be reconstructed from the centroids of the particle, the bubble and the particle-bubble aggregate separately. The location of the centroids is obtained for consecutive frames from the video to calculate the trajectory of the objects. Velocities along the trajectories can be obtained in three dimensions. When the particle is attaching to the bubble, the geometrical centroid of the particle-bubble aggregate is changing. For this reason, the codes were used
before and after the attachment and independently for each one of the objects.

An example showing the centroid of a particle-bubble aggregate is shown in
Figure 4.3 (c) and the trajectory of the particle-bubble aggregate is shown in
Figure 4.3 (d).

Figure 4.3: Trajectories of a particle, a bubble, and an aggregate.

Figure 4.3s (a) and (b) show the movement of both the particle and the bubble in
the videos taken from the cameras. Figure 4.3 (a) is the view of the SA5 camera
and Figure 4.3 (b) is the view of the v710 camera. In Figure 4.3 (d), the y-z plane
view is obtained from the video recorded by the v710 camera and the x-z plane
view is obtained from the SA5 camera video. The trajectory of the aggregate
happens in a main plane that is rotated about the z axis. The trajectory points are
projected on the main plane (with direction x’) and on another plane
perpendicular (with direction y’) as it can be seen in Figure 4.3 (e). These
projections allow to visualize that most of the trajectory points lie on the main
plane.
4.2 Particle and bubble aggregates

The different treatments for the particles give different degrees of hydrophobicity. The degree of hydrophobicity is related to the contact angle of the water on the surface. The contact angle changes the way the aggregate looks once the particle and the bubble are attached. In Figures 4.4 (a) to (d), the particle-bubble aggregates with rough hydrophobic particles are shown with varying bubble size. In Figures 4.4 (a) and (b) the aggregate sinks while in Figures 4.4 (c) and (d), the aggregate floats.

![Figure 4.4: Particle-bubble aggregates of rough hydrophobic particles.](image)

Figure 4.5 (a) to (c) shows the stable particle-bubble aggregates for superhydrophobic particles with different bubble sizes. In Figure 4.5 (c) a floating aggregate is shown where the particle is almost completely inside the bubble due to its hydrophobicity.
4.3 Particle-bubble interaction analysis

The interaction of a particle and a bubble can be analyzed in many different ways. In this present work, it is desired to explore the influence that the relative position between the two of them has on the interaction. The surface treatment is known to play a determinant role in the interaction as it varies the hydrophobicity of the particles. The degree of hydrophobicity determines the readiness of a particle to attach onto a bubble.

4.3.1 Influence of relative distance

The relative distance (d) is the horizontal distance of between the centroid of the particle and the centroid of the bubble before the interaction as graphically seen in Figure 4.6 (a). Figure 4.6 (b) was used to make a model for predicting the terminal velocity of the particle-bubble aggregate.
For a determined surface treatment, the relative distance of the particle and the bubble has different outcomes such as attachment and no attachment between them. The outcomes are classified in cases. The defined cases are listed below.

- **Case 1:** Stable attachment. Once the particle and the bubble form an aggregate, they remain attached during the observed recording.
- **Case 2:** Unstable attachment. Attachment happens, but the particle detaches from the bubble.
- **Case 3:** No attachment.

The analysis was first done for each one of the surface treatments. The results are presented in the form of two-dimensional plots that show the position in x and y of the particle with respect to the bubble. The bubble centroid is located in the origin of the plot and the figures in the plot indicate the position of the particle.
The plots are equivalent to see the physical experimental arrangement from the top view. Figure 4.7 shows the orientation of the origin with the cameras.

![Figure 4.7: Cartesian coordinate system showing both cameras.](image)

The plots were done using a normalized distance in $x$ and $y$ of the position of the particles. This normalized distance is the real distance in $x$ and $y$ divided by the sum of the characteristic radius of the bubble and the radius of the particle for each one of the experiments. This means that when the normalized distance is more than 1, the particle and the bubble cannot touch each other.

In the plots, a circle is drawn that indicates the largest radial position of the interaction that resulted in a stable attachment. The radius of the circle is the largest relative distance for which a stable attachment was observed in that set of experiments. The circle radius normalized by the sum of the bubble radius and the particle radius is called the critical radius $r_c$. It would make sense to think that most of the collisions happening within this circle will yield attachment, but sometimes this is not the case.
For the rough hydrophobic particles, the resulting plots for the different size of bubbles are shown next in Figures 4.8, 4.9, 4.10 and 4.11.

\[ \text{Oh} = 1.15 \pm 0.22 \times 10^{-3} \]

Figure 4.8: Rough hydrophobic particle interactions with \( r_b \approx 0.22 \text{ mm} \).
Figure 4.9: Rough hydrophobic particle interactions with $r_b \approx 0.32$ mm.
Figure 4.10: Rough hydrophobic particle interactions with $r_b \approx 0.7$ mm.

\[ Oh = 17.57 \pm 2.11 \times 10^{-3} \]

Figure 4.11: Rough hydrophobic particle interactions with $r_b \approx 1.4$ mm.

From the previous figures, it is observed that the bubble size influences the outcome of the interactions by observing the size of the attachment region. The attachment region is the area enclosed by the circle drawn in the plots. This attachment region is the largest for the experiments in Figure 4.9. The larger the attachment region, the larger the possibility of attachment. As the bubble size increases, it is observed that the attachment region reduces. The changes in the attachment region depending on the bubble size, leads to the idea that there exists an ideal bubble size for having the largest attachment region. This bubble size would be close to 0.32 mm of radius.
For the low hydrophobic particles and the smooth hydrophobic, there is no attachment observed in any of the different experiments. The bubble sizes that were tested varied in the same range as in the experiments with the rough hydrophobic particles and the superhydrophobic particles.

The plots for the superhydrophobic particles are shown next in Figures 4.12, 4.13 and 4.14.

\[ \text{Oh} = 1.17 \pm 0.30 \times 10^{-3} \]

![Graph](image.png)

Figure 4.12: Superhydrophobic particle interactions with \( r_b \approx 0.2 \text{ mm} \).
Figure 4.13: Superhydrophobic particle interactions with $r_b \approx 0.31$ mm.

Oh = $3.47 \pm 0.56 \times 10^{-3}$

Oh = $18.75 \pm 4.22 \times 10^{-3}$
Figure 4.14: Superhydrophobic particle interactions with $r_b \approx 1.07$ mm.

### 4.3.2 Influence of bubble size and treatment

In the experiments, the bubble size is believed to play an important role. To describe the conditions for the bubble, the Weber number can be used. This number takes into account the velocity of the bubble and its size. The Weber number used is defined as:

$$We = \frac{\rho_w v_b^2 r_b}{\sigma},$$

where $\rho_w$ is the water density, $v_b$ is the bubble velocity, $r_b$ is the bubble characteristic radius and $\sigma$ is the surface tension of the air-water interface.

The dynamics of the particle need to be also taken into account. This can be done using the Reynolds number based on the particle. The Reynolds number used is defined as:

$$Re = \frac{\rho_p v_p r_p}{\mu},$$

where $\rho_p$ is the density of the particle, $v_p$ is the velocity of the particle, $r_p$ is the radius of the particle and $\mu$ is the dynamic viscosity of the water.

Is possible to combine both numbers by using the Ohnesorge number. This dimensionless value can describe better the conditions for each experiment and it allows to compare the effect of the bubble size on the interaction. The Ohnesorge number based on the two other dimensionless numbers is given by:

$$Oh = \frac{\sqrt{We}}{Re}.$$
The critical radius was plotted against the Ohnesorge number for the experiments with rough hydrophobic and superhydrophobic particles as seen in Figure 4.15.

Figure 4.15: Critical radius for two particle types with varying bubble size.

For the rough hydrophobic particles, it seems to be a value of the Ohnesorge number for which the critical radius is maximum. The Ohnesorge number changes mainly by the bubble size. Having an Ohnesorge number for which the critical radius is the largest, means that a certain bubble size corresponds to it.

For the superhydrophobic particles, the behavior is different. It seems that there is a tendency for the critical radius to increase as the Ohnesorge number increases.
4.3.3 Influence of surface roughness

The effect by the surface roughness on the attachment is investigated. A comparison between rough and smooth particles was done using the same hydrophobizing agent for both. The rough particles were obtained using Treatment 3 and the smooth particles were obtained using the Treatment 5. The relative positions of the interactions are shown in Figures 4.16 and 4.17.

Figure 4.16: Relative positions of the interactions for smooth particles.
Figure 4.17: Relative positions of the interactions for rough particles.

There is no attachment observed in the smooth particles while the smooth particles show several cases of attachment. It is believed that the surface roughness promotes attachment because the Ultra Glaco adheres better on the rough surface or because the rough surface facilitates the attachment as air is kept on the rough surface due to the pinning force. This force can be very strong and is capable of keeping a TPCL fixed to a rough surface [25].

4.4 Terminal velocities

After the attachment, the particle-bubble aggregate is formed. The aggregate reaches a terminal velocity sometime after the attachment. The terminal velocity is believed to depend on the size of the bubble and the particle. The schematic of
Figure 4.6 (b) was used to find the expression that allows to calculate the terminal velocity.

### 4.4.1 Influence of bubble size

The aggregate terminal velocity is analyzed based on the dimensionless bubble size $r^* = \frac{r_b}{r_p}$, where $r_b$ the bubble characteristic radius and $r_p$ the radius of the particle. A plot of the terminal velocity magnitude against the normalized bubble radius is shown in Figure 4.18. We developed a model to estimate the terminal velocity that is explained later in the next section.

![Figure 4.18: Terminal velocity of the particle-bubble aggregates.](image)

### 4.4.2 Terminal velocity model

The terminal velocity of an aggregate depends on the bubble and the particle size for the conditions of the experiments. Based on the forces acting on both the

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1 The model was developed with the help of José Federico Hernández Sánchez
particle and the bubble, we obtained a simple and useful model for predicting the terminal velocity.

We consider the buoyancy force for the bubble and for the particle acting opposite to the gravity. The buoyancy force of the bubble is $F_{bb} = (\rho_w - \rho_{air})V_b g$, where $\rho_w$ is the water density, $\rho_{air}$ is the air density, $V_b$ is the volume of the bubble and $g$ is the gravity constant. We ignore the drag force on the bubble as its surface is mobile and the drag force is minimal. We consider the buoyancy force of the particle $F_{bp} = \rho_w V_p g$, where $V_p$ is the volume of the particle. The drag force on the particle is $F_{dp} = \frac{1}{2} \rho_w v_p^2 C_d A$, where $C_d$ is the drag coefficient and $A$ is the reference area that corresponds to the particle. The weight of the particle has also to be considered and it is $W_p = \rho_p V_p g$, where $\rho_p$ is the density of the particle.

At terminal velocity, the sum of forces acting on the aggregate are zero so $0 = F_{bb} \pm F_{dp} + F_{bp} - W_p$. The sign on the drag force of the particle is positive if the aggregate is sinking and negative when it is floating. This sign convention has to be taken into account as the terminal velocity has a direction. Using the force balance on the aggregate at terminal velocity, substituting each force with their variables in it and doing some simplification steps we obtain:

$$v^2 = \left(\frac{8}{3}gr_p \frac{\rho_p}{C_d} \right) \left( \pm \left\{ \frac{\rho_p}{\rho_w} - 1 \right\} - \left\{ 1 - \frac{\rho_{air}}{\rho_w} \right\} \left( \frac{r_b}{r_p} \right)^3 \right),$$

where $v$ is the terminal velocity of the aggregate. The sign convention is the same as for the drag force. The negative values of the equation give the positive values
of the velocity and vice versa. A prediction of the terminal velocity using this model can be seen in Figure 4.18.

4.5 Attachment and detachment

The attachment process varies depending on the type of treatment used for the particles. Two phenomena were observed with the superhydrophobic particles. One phenomenon was the generation of a satellite bubble due to the formation of the TPCL. The phenomenon was only found for the smallest size of bubble interacting with a superhydrophobic particle. A typical satellite bubble generation is displayed in Figure 4.19.

Figure 4.19: Satellite bubble generation during the expansion of the TPCL.

In Figure 4.19 (a), the bubble is in close proximity to the superhydrophobic particle. In Figures 4.19 (b) and (c), the capillary waves can be seen on the surface of the bubble and in Figure 4.19 (d), the satellite bubble generated is shown. This satellite bubble is generated by the mechanism described in [26]. This mechanism consists in the concentration of the capillary waves in one part of the
bubble where they amplify and create the satellite bubble. This satellite bubble generation was observed in 11 experiments for the same bubble size.

Another phenomenon observed only for superhydrophobic particles was a droplet generation inside the bubble. When the attachment happened, a minute amount of water was trapped in the form of a dimple that later became a droplet. This phenomenon was observed only in two experiments. The droplet is not seen to coalesce with the surrounding water, it remains inside the bubble for the whole recording. Images from the two experiments are shown in Figure 4.20 (a) and (b) where a droplet is shown inside the bubble for two different experiments.

Figure 4.20: Water droplet inside a bubble of a particle-bubble aggregate.

The formation frame-by-frame of the water droplet is seen in Figures 4.21 (a) to (d). Figure 4.21 (b) highlights the dimple on the particle surface that later becomes a droplet.
Figure 4.21: Droplet generation inside the bubble from a dimple.

The water droplet can be seen to be separated from the particle surface in Figure 4.21 (d).

In some of the experiments the attachment was unstable. This means that the particle detaches from the bubble. For these cases, a neck forms between the bubble and the particle after attachment. The neck becomes thinner as the bubble tends to separate from the particle until the bubble pinches-off. A small bubble remains on the particle surface as a consequence of the previously formed neck as it can be seen in Figure 4.22 (j). This was observed for all the bubbles pinching-off.
Figure 4.22: Bubble pinch-off from a particle.

Figure 4.22 shows the whole detachment process. In Figures 4.22 (a) to (e), the whole detachment is observed and in Figures 4.22 (f) to (j) a close up of the bubble pinch-off is presented.

4.6 Collision efficiency comparison

There is a simple way to calculate the collision efficiency by taking into consideration the grazing trajectory of the bubble. According to [2], the collision efficiency is given by:

\[ E_c = \left( \frac{x_0}{r_p + r_b} \right)^2 \]

where \( x_0 \) is the relative distance between the particle and the bubble for the grazing trajectory. This can be used to compare it with the obtained results to the predicted collision efficiency based on the sample of experiments that are available. \( x_0 \) can be approximated with the critical radius for the rough hydrophobic particles as when a collision happens, it results in attachment. The
comparison was done for the rough hydrophobic particles and the results appear in Figure 4.23.

![Graph showing predicted and observed collision efficiency.]

Figure 4.23: Predicted and observed collision efficiency.

It is noticeable that the model that predicts the collision efficiency is not very suitable for the experiments that were done. This is explained by the fact that the model considers only the geometry of the bodies. In the case of the rough hydrophobic particles, there are hydrophobic forces that attract the particle to the bubble and this increases the collision efficiency as it can be seen for the largest values of the Ohnesorge number. Another variable is that the number of experiments for each set is limited and to obtain the correct efficiency.
5.1 Ideal bubble size

Evaluating the same treatment for the particles with different bubble size, it has been showed that the bubble size influences the attachment region for the same surface treatment. This attachment region is a circular area centered in the bubble characterized by the critical radius. The critical radius is compared for the various sizes of bubbles using the Ohnesorge number (Figure 4.15). It can be seen that the behavior of the critical radius for the rough hydrophobic particles has an apparent maximum value for some value of the Ohnesorge number. This means that there is a bubble size for which the critical radius is the largest. Increasing the critical radius increases the attachment probability. This knowledge can be applied to flotation processes where the material that is desired to be floated has similar characteristics to those of the rough hydrophobic particles.

For the same particles, we can observe a decrease in the critical radius with increasing bubble size after the largest critical radius. This decrease is attributed to the induction time. For the larger bubbles, their surface deforms more as the Weber number increases with bubble size. This deformation does not allow the liquid film to drain during the contact time. Instead of pushing the water from the liquid film, the bubble deforms and less pressure is applied on the liquid film. This leads to have no attachment.
It is important to note that the largest critical radius for the rough hydrophobic particles happens for aggregates that sink. The purpose in the flotation process is to float some material. In order to float particles with the same bubble size as those from the experiments, the particle density should be less.

Analyzing the bubble size influence on the critical radius for the superhydrophobic particles, we notice a different behavior. In this case, the critical radius correlates positively with the bubble size. This facilitates the attachment for large bubbles. The induction time for the particles with this treatment is believed to be shorter than the rest of the treatments due to the hydrophobicity of the particles.

The surface roughness has an important effect on the attachment. A comparison shows that the attachment was not possible for smooth particles while rough particles with the same hydrophobizing agent. Another example that supports this claim is that even though treatment 2 (smooth hydrophobic particles) and treatment 3 (rough hydrophobic particles) give a very similar degree of hydrophobicity, the particles with treatment 2 do not attach for the same conditions. In these two treatments, the roughness is the key difference among the particles.

5.2 Terminal velocity

The terminal velocity of a particle-bubble aggregate depends on the bubble size, particle size and the liquid characteristics as the model presented in Chapter 4
depends on those parameters and is in good agreement with the experimental measurements.

5.3 Collision description

The collision of the particle and the bubble is not a simple interaction as there is influence of the hydrodynamic flow around the two objects that start to influence one another as they approach. Besides this factor, during the collision, the bubble surface deforms and some kinetic energy goes to the bubble deformation. When the particle and bubble are close, they start to decelerate due to the influence of their respective velocities fields around them. This deceleration included the influence of the added mass to both of the objects as the fluid surrounding them has to be accelerated with them. The added mass is especially important to consider for the bubble as it is the mass of water of half the volume of the bubble. Therefore, the mass of the bubble and the particle has to include the added mass, so their masses will be equivalent masses. The added mass for a sphere is

$$M_{\text{added sphere}} = \frac{2}{3} \pi r^3.$$

The collision of the particle and the bubble can be modeled as an inelastic collision of two spheres where is possible to account for the loss of kinetic energy with a restitution coefficient that is the fraction of kinetic energy after the collision as

$$C_{\text{restitution}} = \frac{E_{\text{initial}}}{E_{\text{final}}}.$$  

In this collision, the momentum has to be conserved, as well as the energy with the restitution coefficient. This is expressed in the following equations:
\[ m_b v_{b1} + m_p v_{p1} = m_b v_{b2} + m_p v_{p2} \]

and

\[ C_{\text{restitution}} \left( \frac{1}{2} m_b v_{b1}^2 + \frac{1}{2} m_p v_{p1}^2 \right) = \frac{1}{2} m_b v_{b2}^2 + \frac{1}{2} m_p v_{p2}^2 \]

where \( m_b \) is the added mass of the bubble, \( m_p \) is the mass of the particle plus its added mass, \( v_{b} \) is the velocity of the bubble and \( v_{p} \) is the velocity of the particle where the subscripts 1 and 2 represent before and after the collision respectively.

This system of equations can be simplified by assuming that one of the objects remains in rest while the other is approaching at a certain velocity.

5.4 Future work

The hydrophobicity of the particles’ surfaces is believed to have influence on the terminal velocity of the aggregates as the aggregate shape changes for the same bubble and particle size. The aggregate shape may change the drag force on the aggregate, thus influencing the terminal velocity. This is especially important for immobile bubble surfaces as the drag cannot neglected for those bubbles. The bubble surface becomes immobile with the presence of reagents in the slurry.

The bubble-bubble coalescence is similar to the phenomenon observed for the super hydrophobic particles where a satellite bubble is generated. The surface of a superhydrophobic particle inside the water behaves like a bubble when the attachment happens. This is attributed to the air layer on the particle surface that makes the attachment process possible. More experiments and investigation is needed to have more insight into this observed phenomenon.


APPENDICES

Appendix A: Ellipse fit MATLAB script

%% FINDS AND PLOTS AN ELLIPSE BASED ON A DEFINED NUMBER OF POINTS

% THIS NUMBER OF POINTS CAN BE DEFINED IN THE VARIABLE numberoflines

close all; clear all; clc;

video=VideoReader('7500_100_18.avi');

startingframe=3;

frameoriginal=read(video,startingframe);

% frameoriginal=rgb2gray(frameoriginal);

frame=imsharpen(frameoriginal,'Radius',7,'Amount',3);

threshold=.15;

frame=edge(frame,'Prewitt',threshold);

frame=imfill(frame,'holes');

% imshow(frame);

% [x0, y0] = getpts;

% index=sub2ind(size(frame),y0,x0);

% frame=imfill(frame,index);

frame=bwareaopen(frame,100);

% imshow(frame);
length=130; % Size of the line to survey for finding the edge of the shape
threshold=.5;
figure(1)
imshow(frame)
[x0, y0] = getpts;
intensityvalues=impixel(frame,x0,y0);
xposition=zeros(length,1);
averagevalues=zeros(length,1);
imshow(frame); hold on
numberoflines=20;
for h=1:1:numberoflines
angle=2*h*pi/numberoflines;
for n=1:1:length
xposition(n,1)=round(cos(angle)*n)+x0;
yposition(n,1)=y0-round(sin(angle)*n);
intensityvalues=impixel(frame,xposition(n,1),yposition(n,1));
end
averagevalues(n,1)=(intensityvalues(1,1)+intensityvalues(1,2)+intensityvalues(1,3))/3;
end
derivate = abs(diff(averagevalues));

I = find(derivate > threshold);

I = round(min(I));

point(h,1) = xposition(I,1);

point(h,2) = yposition(I,1);

for k = 1:length
    plot(xposition(k,1), yposition(k,1), 'r+', 'MarkerSize', 1); hold on
end

end

%% PLOT THE LINES FOR FINDING THE EDGE OF THE ELLIPSE AND THE POINTS THAT WERE FOUND TO BE ON THE EDGE

% plot(point(:,1), point(:,2), 'r+', 'MarkerSize', 100, 'Color', 'b'); hold on

%%
x = point';

[z, c, a, alpha] = fitellipse(x);

imshow(frameoriginal); hold on

y = 1:1:800;

plot(z(1,1), y, '.', 'Color', 'b'); hold on

plot(z(1,1), z(2,1), 'r+', 'MarkerSize', 100, 'Color', 'b'); hold on

plotellipse(z, c, a, alpha, 'g--');
Appendix B: Circle fit MATLAB script

%% FINDS AND PLOTS A CIRCLE BASED ON A DEFINED NUMBER OF POINTS

% THIS NUMBER OF POINTS CAN BE DEFINED IN THE VARIABLE numberoflines

close all; clear all; clc;

video=VideoReader('7500_100_17.avi');

startingframe=1;

frameoriginal=read(video,startingframe);

% frameoriginal=rgb2gray(frameoriginal);

frame=imsharpen(frameoriginal,'Radius',8,'Amount',3);

threshold=.1;

frame=edge(frame,'Prewitt',threshold);

frame=imfill(frame,'holes');

% imshow(frame);

% [x0, y0] = getpts;

% index=sub2ind(size(frame),y0,x0);

% frame=imfill(frame,index);

frame=bwareaopen(frame,100);

imshow(frame);

%%
length=80; % Size of the line to survey for finding the edge of the particle
threshold=.5;

figure(1)

imshow(frame)

[x0, y0] = getpts;

intensityvalues=impixel(frame,x0,y0);

xposition=zeros(length,1);

averagevalues=zeros(length,1);

imshow(frame); hold on

numberoflines=20;

for h=1:1:numberoflines
    angle=pi+h*pi/numberoflines;
    % angle=2*h*pi/numberoflines;

    for n=1:1:length
        xposition(n,1)=round(cos(angle)*n)+x0;
        yposition(n,1)=y0-round(sin(angle)*n);

        intensityvalues=impixel(frame,xposition(n,1),yposition(n,1));

        averagevalues(n,1)=(intensityvalues(1,1)+intensityvalues(1,2)+intensityvalues(1,3))/3;
    end
derivate=abs(diff(averagevalues));
I=find(derivate>threshold);
I=round(min(I));
point(h,1)=xposition(I,1);
point(h,2)=yposition(I,1);
for k=1:length
    plot(xposition(k,1),yposition(k,1),'r+', 'MarkerSize', 1); hold on
end
end
plot(point(:,1),point(:,2),'r+', 'MarkerSize', 100,'Color','b'); hold on

[xc,yc,R,b]=circfit(point(:,1),point(:,2));
imshow(frameoriginal); hold on
circle(xc,yc,R);