Tailored bio-chemical solutions on Unmanned Aerial Vehicles

A thesis submitted in partial fulfillment of the degree Doctor of Philosophy

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March 2019
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Abstract

The development of Unmanned Aerial Vehicle (UAV) systems in the field of biochemistry and medical transport is an emerging field of research. The availability of such systems offers to advance optimal medical care through improved timeliness in diagnosis in particular for procedures that require bio-chemical processing. This thesis reviews the relevant literature on liquid characteristics and current UAV technologies. It then proceeds to develop novel methods to study liquid behaviour on superhydrophobic surfaces. These methods included research on formation of droplets, transport of droplets and stability of droplets on superhydrophobic surfaces. This was done with the objective of creating a practicable UAV system able to conduct biochemical applications. While this objective was not fully achieved, it revealed important insights relevant in the use of higher liquid volumes in vials and capillary tubes. Substantial progress was made in developing UAV based solutions that incorporated bio-chemical processes. Through the use of manoeuvres and modification of elements, bio-chemical processes such as mixing, and centrifugation were achievable on UAV systems.
Declaration

This thesis contains no material which has been accepted for the award of any other degree or diploma at any university or equivalent institution and that, to the best of my knowledge and belief, this thesis contains no material previously published or written by another person, except where due reference is made in the text of the thesis.
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Date: 04/03/2019
Publications


Acknowledgements

I would like to thank Tirthankar Vardhaman Mahavira for being my strength and my guide. I would also like to thank my supervisor, Associate Professor Tuck Wah Ng, for his mentoring and guidance, and my colleagues, Dr. Murat Muradoglu, Dr. Chun Yat Lau, So Hung Huynh, Dwayne Chung, and Alifa Zahidi, for their support and assistance in the project.

Heartfelt thanks to my mother and my wife for the emotional support.

This research was supported by Monash Graduate Scholarship, Faculty of Engineering Scholarship, Department of Education Joint Research Engagement scheme
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Chapter 1 Introduction

1.1 Background

There is understandable interest towards increasing use of point-of-care testing (POCT) for biomarker measurements with its associated advantages of reducing delays in diagnosis and treatment, minimal sample handling and eschewing the need for sophisticated equipment, ease-of-use with lower staff training burdens to maintain formal accreditation, as well as for opportunities for broad applications both in the clinical and nonclinical setting. POCT offers to bypass many steps where pre-and post-analytical errors most commonly arise in centralized laboratory-based testing, which include appropriate specimen collection, handling, transport and storage; instrument set up; testing; and result reporting. Although significant advances in POCT technologies in terms of improved test reliability and increasing spectrum of available POCT menus have been made, there remains issues of relative cost burdens, analytical and clinical performances compared to laboratory-based testing that continue to require sustained efforts to address. Clearly, the role of laboratory testing remains a core pillar for disease management where rapid diagnosis, timely and appropriate intervention are central to quality healthcare.

The transport of samples to the laboratory hence remains to be an important factor. It is strongly governed by the twin factors of timeliness (to ensure their viability) and cost, where the use of autonomous schemes have clear advantages. In hospitals, pneumatic tubes have long been implemented in this vein, to the extent that they are now indispensable to the function of most healthcare support systems. More recently, the use of drones, or Unmanned Aerial Vehicles (UAVs), has been explored for outdoor biological specimen delivery. There have been successful preliminary attempts in using UAVs for blood transportation. Utilising UAVs to conduct medical product transport is also being considered. With the rapid advancements in drone technology, UAVs promises to provide a more sophisticated and an effective alternative to the conventional sample transport methods.

While some instances of malfunctions have been highlighted as issues of concern, these glitches will likely be overcome through the use of more robust electronics technologies, thus enabling their potential use not only in remote venues but also in traffic congested
urban environments, where route planning strategies can only offer limited improvements in timeliness of sample delivery. The timeliness aspect can be further augmented if some parts of the pre-processing of samples are done en-route during transport.

In order to develop bio-chemical solutions in-flight using UAVs, it is pivotal to study the effect of transporting discrete volumes of liquid in the microliter range in biochemical analysis. The use of fluids in small volumes offers a viable approach to do so due to the advantages of limited contact with the solid substrate. Due to reduced sample contamination and loss and controlled transport, surveying liquid behaviour on superhydrophobic surfaces is a great way to understand how liquid samples will be affected during the transport on a UAV.

UAVs can be used to complete pre-analytical processing steps in addition to transporting the liquid samples. Appropriate mixing of samples and centrifugation are arguably two of the most common sample pre-processing steps for obtaining quality specimens for laboratory testing. In the case of blood collection into receptacles, proper mixing for homogenous dispersion of silica particles, separator gels, clot activators or anti-coagulants is vital for optimal downstream separation of serum or plasma. In the case of whole blood samples, it is vital to separate serum and plasma from contact with the cell components within a given time frame as some test analyte levels are significantly influenced by the time between collection and centrifugation.

It is to be noted that extensive research is conducted on transporting biofluids, in several countries. However, the extent to which the biofluids are affected during the transport has not been studied in detail. This could lead to poor transport efficiency and loss of quality of samples. Also, there is very little research on using UAVs to conduct bio-chemical processes in-flight.

1.2 Objectives

The main objective of this project was to design and develop UAV based solutions to advance biochemical applications. This is a two-part process. Firstly, it is pivotal to understand the biochemical processes. Secondly, UAVs must be developed such that they can successfully assist in providing solutions to complete these processes more efficiently. One of the most important components of developing biochemical applications is clear
understanding of behaviour of biofluids. This is achieved by studying liquid behaviour on superhydrophobic (SH) surfaces. Superhydrophobicity is characterised by the extent in which the surface repels water and quantified by the apparent contact angles above 150° at the liquid-solid interfaces.

To study how liquids would behave during transport on UAVs, it is important to study the behaviour of liquids under various factors. Central to this are efforts to minimise sample losses, the possibility of using microfluidic devices is to be considered. Other aspects like characteristics of bio-fluids, effects of parameters like temperature and altitude must also be studied to develop an efficient and practical solution.

With a sound basis on liquid transport efficiency, we made endeavours to design and explore novel bio-chemical solutions using UAVs.

1.3 Outline

The body of this thesis is organised as follows:

• Chapter 2: A two-part review of existing literature consisting of liquid characteristics and UAVs. The review of liquid characteristics comprised of surveying the current literature on biofluids, liquid receptacles, wettability, effects of temperature and altitude on liquids and stability of liquids. The review of UAVs consisted of surveying different types of UAVs, examining flight and control characteristics, the effects of gusts on UAVs and their applications in the field of biochemical transport and analysis.

• Chapter 3: In this chapter, we conducted studies on the behaviour of liquids on superhydrophobic surfaces. These included studying the formation of drops, transport of liquids on SH inclines and the effect of resonance of liquid bodies when contacting an SH surface. These studies offered important insights into the characteristics of liquids and the impact of different factors on the behaviour of liquids.

• Chapter 4: It is desirable to physically test the flight models and control strategies of small unmanned aircrafts (UA). A gust simulator that is able to operate within a wind tunnel, where airflow conditions can be closely controlled, is reported here. The simulator here
presents a convenient experimental platform to develop improved mini UA designs to account for the effect of gusts.

- Chapter 5: Innovative ways of conducting in-flight mixing and centrifugation of biofluids are reported in this chapter. It is shown here that flipping maneuvers conducted with quadcopters are able to facilitate complete and gentle mixing. This capability incorporated during automated sample transport serves to address an important factor contributing to pre-analytical variability which ultimately impacts on test result reliability. The ability to conduct en-route centrifugation of samples improves quality and timeliness in the pre-analytical phase. This is demonstrated here on a quadcopter whereby the propellers were adapted to house and apply centrifugal forces to sample-containing capillary tubes instead of incorporating a centrifuge

Chapter 2 Literature Review

2.1 Liquid characteristics

2.1.1 Biofluids

In this section, characteristics and mechanics of biofluids are studied. To create new devices in the biofluids field, understanding biofluids phenomena is very important. The fluids associated with a human body are called biofluids or body fluids. These include air, O₂, CO₂, water, solvents, solutions, suspensions, serum, lymph and blood. Biofluids can be excreted (such as urine or sweat), secreted (such as breast milk or bile), obtained with a needle (such as blood or cerebrospinal fluid), or develop as a result of a pathological process (such as blister or cyst fluid). One of the most commonly studied biofluids is blood due to its importance in medical diagnosis. The main function of blood is to transport energy and nutrients to all the living tissues and to remove the waste products from the body. Since ancient times scientist have been motivated to understand blood [1, 2, 3]. Due to its complex nature and the lack of tools to study it, the road to its fully understanding has been slow and still growing [4]. It is commonly known that blood in rest separates in different layers of fluid. An upper layer known as serum or plasma, a thin layer of leukocytes and platelets and a red blood cells (RBCs) layer on the bottom. Blood studies continue to develop in the early twentieth century.
Human Blood is a two-phase fluid system and consists mainly of an aqueous polymeric and ionic solution of low viscosity, the plasma, in which is suspended a 0.45-0.50 concentrated cellular fraction [5]. The plasma is a liquid phase mixture of metabolites, proteins and lipoproteins suspended in a salt solution composed mostly of water. The cellular fraction is a complex mixture of mainly erythrocytes, commonly known as red blood cells, but it also contains leukocytes (white blood cells) and thrombocytes (platelets). Nearly a 99% of the cellular fraction in blood is represented by red blood cells [6]. The complete set of blood components is usually referred as whole blood.

Plasma proteins play an important role on the hemorheological properties of whole Blood [7]. First, because even though blood plasma is 92% water, its viscosity at 37°C is nearly twice the viscosity of water at the same temperature and this is due mainly to plasma proteins. Second, plasma proteins (especially fibrinogen) causes red blood cells to stick together forming aggregates, like piles of coins, known as rouleaux. Rouleaux formation is important because it causes the viscosity of blood to be very dependent on the shear rate to which it is exposed [8].

The erythrocytes volume fraction on a blood sample is commonly referred as haematocrit. The normal range of haematocrit differs between men and women and it is 40% to 50% and 36% to 46% respectively. Leukocytes and thrombocytes together only comprise about 1% of the cellular fraction. This high concentration of RBC’s is the main reason that they are hemorheologically important, though physical and morphological properties of them also contribute to blood behaviour [4].

Human red blood cells have an unusual shape. They are anucleate biconcave discs [9] of about 7:8 m diameter and a thickness of 2:5 m at the thickest point and 1 m or less in the centre, this gives them the capacity to align with the direction of flow. The cellular membrane of a healthy RBC is flexible, which means, that it can change its shape and deform under different conditions. This is due to the elastic properties of its bi-layered composition and cytoskeleton. They also have the, previously mentioned, tendency to adhere together forming aggregates, also referred as rouleaux, this aggregates formation. Last, they contain a haemoglobin solution of high concentration which affects the speed at which they deform. All these properties of red blood cells act together to give blood a viscosity substantially higher than blood plasma and contribute to its non-Newtonian properties [10].

On the other hand, white blood cells and platelets hemorheological role is less significative, mainly due to its low concentration in blood in comparison with red blood cells. Even though
white blood cells are bigger in size and present viscoelastic properties by themselves, they play an important role in micro-circulation resistance [11]. However, since its volume concentration is approximately three orders of magnitude lower than red blood cells, its effects are less relevant in general circulation. In the case of platelets, they are much smaller than RBC’s and their volume in blood is even smaller than the leukocytes concentration [12].

Biofluid mechanics play a pivotal role to help us find or predict solutions for disorders and diseases. The World Health Organization (WHO) estimated around 17 million people die of cardiovascular diseases, mostly heart attacks and strokes, every year [13]. Also, the number of people with end stage of renal disease who receive renal replacement therapy is more than 1.4 million with an increasing rate of 8% per year [14]. This enormous rate of death and therapy for only two diseases demonstrates the importance of biofluid mechanics research.

Biofluid concepts apply to numerous systems in the human body. These include Ocular [15], synovial [16], cerebrospinal [17], cochlear [18], pulmonary [19], nasal [20, 21] and other [22]. A strong practical benefit of studying biofluid mechanics is that investigation of directly accessible biofluidic observables can lead to improved diagnostic capabilities as well as assessment and prediction of treatment outcomes [23].

A substantial amount of research has been done to study biofluid mechanics in the body. However, the mechanics of biofluids during transport have not been studied in detail.

### 2.1.2 Liquid Receptacles

In this section, small liquid receptacles and their importance in POCT systems is surveyed. During the last decade, bio-electronic industry has gain interest with intensive research performed in small portable devices capable of rapid detection and monitoring of lab scale processes allowing to perform diagnostic tests directly at the point of patient service. For portability, Point of care Testing (POCT) generally contain receptacles or containers that can only contain very small amounts of liquid [24]. This has an inherent advantage of minimisation of sample losses.

Smaller liquid receptacles can also decrease considerably the amount of blood sample needed from millilitres (from a vein) to microlitres (from a fingertip). Liquid receptacles in modern laboratories include droplet receptacles, capillaries and vials. They are generally classified on the basis of volume that be contained in the vessel. A droplet receptacle can generally contain
between 1 to 20 µL of the sample while capillary storage tubes can contain between 25 µL to 150 µL of a liquid sample. Volume of liquid in vials can range from between 1mL to 5mL.

Figure 2-1: Pictorial depiction of drops, capillaries and vials (Left to Right) [25, 26]

While it is often desirable to use droplets for sample transport and in-flight biochemical analysis to use the lowest possible volume, several factors play a role in the type of receptacle used for a particular system. The suitability of the receptacles can depend on adequacy of volume required to conduct analysis to get conclusive and comparable results, environmental conditions posed using UAVs amongst other factors.

2.1.3 Wettability

In this section, fundamentals of wetting and the classification of surfaces based on wettability are reviewed. When wetting occurs, an interface is created, and energy is required to break bonds between atoms or molecules on the liquid surface. Thus, there are fewer neighbouring molecules at the surface of a solid or liquid than those in the bulk. Such discrepancy results in atoms at the interface having surplus energy or surface tension, $\gamma$ [27]. Measured in N/m, it is equal to the tensile force required to create a surface per unit length.

Hydrophilic surfaces are water-loving surfaces. Hydrophobic and super hydrophobic (SH) are water-repelling surfaces. It means that the water does not stick to its surface. In hydrophobic surfaces, the contact angle of the droplet is less than 120 degrees and the roll off angle are more than 10 degrees. In SH surfaces, the contact angle can 150 degrees and the roll off angle is less than 10 degrees. Roll-off Angle is the maximum tilt angle that a solid can be tilted before a drop releases. This is most often measured using a tilting base option.
SH surfaces are surfaces with great water repellency with a water contact angle greater than 150° [28, 29]. The contact angle of an ideally flat and chemically homogeneous surface can be found using Young’s relation [30]

$$\cos \theta = \frac{\gamma_{SV} - \gamma_{SL}}{\gamma_{LV}}$$  \hspace{1cm} (Eq. 2.1.3.1)

where $\theta$ is contact angle ($^\circ$); and $\gamma$ is the interfacial surface tension while the subscripts S, L and V represent solid, liquid and vapour respectively. Representation of hydrophilic, hydrophobic and SH surfaces can be seen in Figure 2-2.

![Figure 2-2: Different surfaces defined based on contact angle](image)

Young’s equation does not take the roughness of the surface into the account. Wenzel [31, 32] accounted by introducing a correcting factor for the contact angle given as

$$\cos \theta^* = R \cos \theta$$  \hspace{1cm} (Eq. 2.1.3.2)

where $\theta^*$ is the corrected contact angle ($^\circ$); R is the Wenzel correction factor given as

$$R = \frac{A_{\text{rough}}}{A_{\text{smooth}}}$$  \hspace{1cm} (Eq. 2.1.3.3)

where A is the contact area between liquid droplet and the surface

Wenzel equation is based on the assumption that the droplet completely wets the surface. This is not always the case and a hydrophobic surface can sometimes remain partially non-wetting under a liquid droplet. To calculate the contact angle for such a non-wetting state, Cassie-Baxter equation
is used. It is given as

\[ \cos \theta^* = -1 + \varphi_S (\cos \theta + 1) \]  

(Eq. 2.1.3.4)

An illustration of Wenzel state and Cassie-Baxter can be seen in Figure 2-3.

These equations are used to explain the wetting phenomena and are often to quantitatively identify an SH surface.

In summary, Wenzel model predicts that contact angle can be increased with surface roughness while Cassie-Baxter equations [33] simply depicts the apparent contact angles can be higher even though it was less than 90° on the original smooth surface. Due to the characteristics aforementioned in this section, superhydrophobic surfaces serve as an excellent surface to study fluid flow due to their non-wetting behaviour.

2.1.4 Environmental effects on transported blood

In this section, how environmental factors like change in temperature and pressure affect the transported blood is studied. Due to the constant variation in altitude as UAVs transport blood from one location to another, it is important to study the environmental factors affecting the quality of blood. A major consequence of not understanding the effects could lead to poor blood quality and incomplete or incorrect diagnosis.

2.1.4.1 Temperature Effects

Blood components and their quality can be significantly impacted by changes in temperature. The United States Food and Drug Administration (FDA) guidelines during the early 1980s allowed the preparation of blood components at 20°C to 24°C within 8hrs of collection and a 5-day storage period for platelet concentrates (PCs) [34]. In Germany, the Armed Forces Blood Service in Koblenz collect up to 40000 whole blood donations from voluntary military and
civilian personnel of the German Armed Forces. The whole blood units are then stored at 20°C and sample tubes for laboratory investigation are stored at 4°C before being carried in a refrigerated vehicle overnight [35].

In Canada, as well as in many other countries, guidelines for the preparation of blood products allow blood banks to store whole blood (WB) units up to 24 hours before processing, provided that the blood is actively chilled to 22 ± 2°C. This is usually achieved by placing the whole blood bags under cooling plates filled with butane-1,4-diol, which rapidly cool fresh blood units from a post collection temperature of about 34°C to 22 ± 2°C within 2–3 hours [36, 37]. This rapid cooling maintains blood product quality, while normalizing temperature history of WB units before processing [38, 36]. The routine use of cooling plates represents an important operational challenge for blood banks, particularly when WB units are collected on remote sites [39]. Exposure to outside temperatures during transport from mobile collection sites limit the capacity of platelets to cool and to maintain the temperature of WB units between 20°C and 24°C. As a result, cooling plates for overnight storage of WB units have not been an ideal solution [40, 41].

Currently, the most commonly used blood product is fresh frozen plasma (FFP), which can be obtained from a plasmapheresis collection or donated whole blood (WB). FFP must be prepared and frozen within 6 or 8 hours from the time of collection, according to the usage of different anticoagulants for WB storage [42]. These requirements for FFP preparation could limit the number of WB units that can be processed into FFP because of the blood collections from blood-collecting vehicles. Whole blood stored overnight at 4°C can be used to manufacture plasma and concentrated red blood cells (RBCs) but is of little use to manufacture platelet concentrates because of the deleterious effect of temperature on platelet quality. Platelets do not tolerate refrigeration and disappear rapidly from circulation if subjected to chilling before transplantation [43, 44] After a conditioning step of several hours at 4°C, cooling plates must stand for several minutes at ambient temperature to allow their temperature to reach 14°C–16°C to prevent damages to platelets. Therefore, whole blood used for platelet concentrate separation is generally stored at room temperature. Currently, PC preparation according to buffy coat removal method is widely practiced [45] and is associated with significantly better platelet recoveries and less platelet activation immediately after preparation.
A study conducted in China [46] compared the quality of plasma prepared from whole blood that was processed and stored at different temperatures. These included preparing plasma from whole blood that was processed within 8 hours after collection and storage at 4°C, within 18-24 hours of storage at 4°C, or within 18-24 hours of storage at ambient temperature and quality of platelets from buffy coat processed either from fresh whole blood (PC1) or whole blood that was held overnight at room temperature (PC2). To understand the effects of variation in storage temperature and duration, the coagulation factors or clotting factors were compared. Compared to the FFP samples, the levels of coagulation factors FVII and FVIII in the FP24 (plasma frozen within 24 hours of blood collection) samples decreased significantly. The pH, Na⁺, LDH, and FHb levels differed significantly between FP24 and FFP. Compared to PC1, PC2 exhibited lower pH, pO₂, and Na⁺ levels, a higher platelet count, and increased pCO₂, K⁺, Lac, and CD62P expression levels [46].

Also, strict temperature control contributes to a better quality of the red cell concentrates [47]. However, in practice, temperatures may be less well controlled, and room temperature variations must be considered. Also, temperature excursions may occur during transport, during handling or if delays in transport occur. Especially on days with extreme temperatures, WB temperatures have been suspected to come outside the range 20°C–24°C, despite using butane-diol plates that have a protective effect against changes in environmental temperature.

A study conducted in Netherlands [48] investigated the quality of blood components was affected by small deviations (18°C–25°C) from the required temperature limits (20°C–24°C). It was found that the quantity of effector molecule 2,3-DGP significantly decreased when the temperature was above or below the ambient temperature. Also, red cell concentrates, and platelet concentrates exhibited higher initial lactate levels and lower pH at 25°C when compared with the concentrates at 18°C. It was concluded that the platelet and blood component quality is largely impacted when blood is exposed to varying temperatures [48].

### 2.1.4.2 Pressure Effects

Potential disadvantages of high external pressure on blood components include decreased deformability of RBCs and haemolysis caused by greater shear stress on erythrocytes. The shear stress is generated by the turbulent blood flow passing through the transfusion set or by the direct pressure applied to the cells in the blood bag [49]. Indeed, several studies indicated that the application of higher external pressure results in greater haemolysis of transfused blood [50, 51]. It is believed that the shear stress resulting from high external pressure may induce
haemolysis. External pneumatic pressure on a blood bag up to 300mm Hg may increase plasma-free haemoglobin, which is evidence of haemolysis [49].

The RBC deformability may also be reduced by shear stress exceeding physiological levels [52] With appropriate external forces, RBCs undergo large mechanical deformation without rupture and then are restored to their original shape when released [53]. Furthermore, the duration of storage can affect the function of RBCs. In a study conducted to assess the influence of storage on RBCs rheological properties [54], it was reported that the deformability of RBCs was normal on the fifth and seventh days of storage, but the deformability was significantly decreased on the 14th day of storage and remained low throughout the remainder of the storage period. Thus, externally applied high pneumatic pressure on RBCs and the storage period may affect RBC deformability and haemolysis due to increased shear stress, especially in aged blood.

2.1.5 Stability of liquid transport

2.1.5.1 Effect of liquid sloshing

Road tank vehicles are one of the most common means of transport for carrying a wide range of liquid cargoes. However, they are very frequently involved in rollover-related accidents causing serious harm to the public and the environment. Statistical data collected by Statistique Canada has shown that 83% of truck rollover accidents on highways are cause by tank vehicles [55]. In USA, a study has reported that the average annual number of cargo tank rollovers is about 37% of the total number of heavy vehicle highway accidents [56].

The reasons for vehicle rollover accidents include driver’s fatigue, poor weather conditions and liquid sloshing [57, 58]. Sloshing is the motion of the free liquid surface inside its container. It is caused by any disturbance to partially filled liquid containers. Depending on the type of disturbance and container shape, the free liquid surface can experience different types of motion including simple planar, nonplanar, rotational, irregular beating, symmetric, asymmetric, quasi-periodic and chaotic. When interacting with its elastic container, or its support structure, the free liquid surface can exhibit fascinating types of motion in the form of energy exchange between interacting modes. Modulated free surface occurs when the free-liquid-surface motion interacts with the elastic support structural dynamics near internal resonance conditions. Under low gravity field, the surface tension is dominant, and the liquid
may be oriented randomly within the tank depending essentially upon the wetting characteristics of the tank wall [59].

In order to understand the causes of liquid sloshing, it is important to estimate hydrodynamic pressure distribution, forces, moments and natural frequencies of the free-liquid surface [60]. These parameters have a direct effect on the dynamic stability and performance of moving containers. Generally, the hydrodynamic pressure of liquids in moving rigid containers has two distinct components. One component is directly proportional to the acceleration of the tank. This component is caused by the part of the fluid moving with the same tank velocity. The second is known as “convective” pressure and represents the free-surface-liquid motion. Mechanical models such as mass-spring-dashpot or pendulum systems are usually used to model the sloshing part [61].

A liquid’s motion inside its container has an infinite number of natural frequencies, but it is the lowest few modes that are most likely to be excited by the motion of a vehicle. Most studies have therefore concentrated on investigating forced harmonic oscillations near the lowest natural frequencies, predicted by the fluid field linear equations [59]. However, nonlinear effects result in the frequency of maximum response being slightly different from the linear natural frequency and dependent on amplitude. Nonlinear effects include amplitude jump, parametric resonance, chaotic liquid surface motion, and nonlinear sloshing mode interaction due to the occurrence of internal resonance among the liquid sloshing modes. The nonlinearities associated with free-surface motion inside moving containers are different from those nonlinear water waves in ocean and canals. The theory of nonlinear dispersive waves originated by Stokes [62] and solitary waves observed by Russell [62] is well documented in Debnath [63].

Analytical solutions are limited to regular geometric tank shapes such as cylindrical, and rectangular. The nature of sloshing dynamics in cylindrical tanks is better understood than for prismatic tanks [64, 65]. Sloshing phenomena in moving rectangular tanks can usually be described by considering only two-dimensional fluid flow if the tank width is much smaller than its breadth. Sloshing in spherical or cylindrical tanks, however, is usually described by three-dimensional flow. Tanks with two-dimensional flow are divided into two classes: low and high liquid fill depths. The low fill depth case is characterized by the formation of hydraulic jumps and traveling waves for excitation periods around resonance [59]. At higher fill depths, large standing waves are usually formed in the resonance frequency range. When hydraulic
jumps or traveling waves are present, extremely high impact pressures can occur on the tank walls. Impact pressures are measured experimentally [66].

The problem of liquid sloshing in moving or stationary containers remains of great concern to aerospace, civil, and nuclear engineers, physicists, designers of road tankers and ship tankers, and mathematicians. Civil engineers and seismologists have been studying liquid sloshing effects on large dams, oil tanks and elevated water towers under ground motion. Since the early 1960s, the problem of liquid sloshing dynamics has been of major concern to aerospace engineers studying the influence of liquid-propellant sloshing on the flight performance of jet vehicles, and new areas of research activities like the effects of sloshing in small containers on UAVs have emerged.

The modern theory of nonlinear dynamics has aided to uncover complex nonlinear phenomena. These include rotary sloshing, Faraday waves, nonlinear liquid sloshing interaction with elastic structures, internal resonance effects, stochastic sloshing dynamics, hydrodynamic sloshing impact dynamics, g-jitter under microgravity field, dynamics of liquid bridges, cross-waves, and spatial resonance [67, 68, 69, 70]. The dynamic stability of liquefied natural gas tankers and ship cargo tankers, and liquid hydrodynamic impact loading are problems of current interest to the designers of such systems. A liquid free surface in partially filled containers can experience a wide spectrum of motions such as planar, non-planar, rotational, quasi-periodic, chaotic, and disintegration. Other important contributions include the development of digital computer codes to solve complex problems that were difficult to handle in the past. The following methods are currently used to study the sloshing characteristics in tanks:

1) **The quasi-static (QS) method:** The cargo’s static moment at a specified point on a tank vehicle can be approximated by calculating the transient centre of gravity (CG) of the liquid bulk in the tank. Then, the liquid sloshing effect on tank walls can be analysed. It is convenient and simple to obtain liquid sloshing force using the QS method. However, the analysis results have poor accuracy [71, 72, 73, 74].

2) **The hydrodynamics method:** By theoretically analysing liquid flow characteristics in partially filled tanks, sloshing parameters can be acquired using basic hydrodynamic equations. Although the results so obtained are accurate, this method is complicated. Due to the limited studies on turbulence and the fact that the majority of flow can be
categorized as turbulence, a large number of liquid flow phenomena cannot be explained using this method [75, 76, 77, 78].

3) **The experimental method:** By building a test platform or using test tank vehicles, liquid sloshing phenomenon can be observed, and relevant parameters can be monitored by reproducing liquid sloshing [79, 80]. The experimental results will depend on the test devices used, the sensor accuracy, and the operation of the tests, and so forth. And the method requires significant human and material resources.

4) **Computer simulation:** Simulation software is used to simulate liquid sloshing and to obtain the values of a corresponding sloshing dynamic effect [81, 82].

5) **The equivalent mechanical model:** Here, mechanical models are used to simulate liquid sloshing [83] and widely used for its simplicity and accuracy. Until now, most of the researches using this method have focused on spacecraft tanks and other vertical tanks [83, 84, 85].

To date, many studies have been carried out on liquid flow and sloshing characteristics in larger containers or receptacles like tanks. However, the effects of liquid sloshing in smaller receptacles like vials have not been studied in detail.

### 2.1.5.2 Sessile Drop Resonance Effect

The vibration of liquid droplets and the resonance effect is an important aspect to study its effects on liquid droplet transport through UAVs. It is a topic that has numerous potential applications in both science and technology. In particular, the vibration of droplets on surfaces (sessile drops) has been shown to aid processes such as drop atomisation. Here, liquid drops are placed on a surface that is vibrated in such a way as to excite the resonant modes of the droplet. If higher order modes are excited with large amplitudes, small secondary droplets are ejected from the surface of the sessile drop [86]. One of the potential applications of the vibration of sessile droplets is that of the mixing of fluids where oscillating microlitre liquid droplets has been shown to accelerate the mixing process by about two orders of magnitude [87]. Other examples of potential applications include technologies such as inkjet printing, spray coating and fuel injection [88] where the production of drops of a well-defined size is required.
Recent studies have shown that [89, 90, 91] that the motion of droplets on surfaces can be controlled through vibration. The vibration of droplets on surfaces also provides a technique that allows for the simultaneous measurement of surface tension and viscosity [92]. Surface tension can be measured by examining the dependence of resonant frequency on drop size [93]. Viscosity of the liquids can be determined by considering the damping of the oscillations via measurements of the mechanical resonances [94]. Although these measurements can be carried out on levitated droplets [95], developing the technique for sessile drops has many advantages as the experiments are quick and simple to carry out using relatively low-cost equipment.

Numerous authors have derived theories that attempt to describe the behaviour of the vibration of sessile drops [96, 97, 89]. This work has been utilised to explore the potential of using sessile drop vibration as a tool for determining the properties of liquids. Oscillations in sessile droplets can be excited in several ways. Commonly, the oscillations are driven, for example by attaching the substrate, on which a drop sits, to a loudspeaker [89] or piezoelectric actuator [88]. Here, the resonant modes are determined by performing a frequency sweep and monitoring the change in shape of the drop. This technique drives the droplets at all frequencies simultaneously and with the same amplitude. It is also possible to excite resonance by perturbing the drop with a short impulse that provides broadband excitation. All oscillations apart from those that meet the criteria for resonance will decay away instantaneously leaving the drop ringing at its resonant frequencies. This technique has been utilised previously by Sharp et al. where a short puff of air was used to perturb the droplet [98].

### 2.1.5.3 Shear Driven Sessile Drop Shedding

Exposing shear flow to a sessile droplet which is placed on a substrate makes the droplet move. This movement is called shedding and only happens when the shear flow overcomes the droplet adhesion to the substrate. After the start of the droplet shedding, which is called the incipient motion, the droplet moves downstream along the surface [99]. Shear driven droplet shedding has various industrial applications such as in icing phenomena that occurs in air foils of an aircraft, wind turbine blades and power lines [100]. The knowledge can also be used in
enhancing the performance of oil recovery systems [101]. The droplets generally impact on the
surfaces and later on start to move along the surfaces due to air shear flow. Generally, on a
hydrophilic surface, the flow of droplets results in the formation of narrow streams of liquid
known as rivulets. These rivulets then propagate on the surface driven by shear forces and can
freeze when the ambient temperature is lower than water freezing temperature. Freezing can
cause significant damage to aircraft components. For example, in the case of airplane, ice
accumulation significantly reduces the aerodynamics efficiency of the airplane by increasing
drag and decreasing lift forces. In a multi-rotor UAVs, icing can lead to an intimidating deficit
in thrust and abrupt increase in power consumption that could result in catastrophic failure and
freezing near propellers can cause the aircraft to fail mid-air [102].

There are limited insights into fundamentals of air shear driven drop shedding in literature
especially on superhydrophobic surfaces. In a recent study [99], droplet incipient motion due
to low shear flow speed below 30 m/s was studied on different surface wettabilities. However,
there has not been much research into investigating droplet incipient motion at higher speeds,
close to those occur in aerospace applications. There have been some studies conducted to
assess the shear flow effect on both hydrophilic and superhydrophobic surfaces. Study of the
drop behaviour on superhydrophobic surface is an active research area due to the high water
repellence characteristics of these surfaces. As mentioned before, shedding occurs when the
drag force overcomes the adhesion forces. It is known that that both drag, and adhesion depend
on, contact angle of the surface (wettability), the size and shape of the droplet. Since the last
two parameters are also a function of contact angle, the surface wettability is highly important
in droplet shedding process [103].

In a recent study [104], the shear driven shedding behaviour of dingle droplet with high speed
was investigated on hydrophilic and superhydrophobic surfaces. It was found that high shear
flow speed caused the droplet to form rivulets while moving on hydrophilic surface. On the
contrary, on superhydrophobic surfaces high shear speed caused the droplet to deform to an
oval shape and subsequently easily detach from the surface. It was also shown that the contact
time of the droplet on the superhydrophobic decreased by increasing the Reynolds number.
The low adhesion of droplet to the superhydrophobic surface makes this kind of surface an
ideal choice for UAV applications [105].
2.2 UAV Systems

2.2.1 Types (Fixed-wing/Multi-rotors/Blimps)

Different types of UAVs are used for various operations. Some of the most common UAVs or remotely piloted aircrafts (RPA) include multi-rotor systems [106], fixed wing systems [107] while blimp systems [108] are used less extensively. A brief overview of these UAVs, along with their design, applications, advantages and limitations is provided in the following sections.

**Multi-rotor UAVs:** Multi-rotor UAVs are most commonly used small sized unmanned aircrafts. They are the easiest and cheapest option for getting a small payload into the sky. There are several configurations of a multi-rotor UAVs [109].Multi-rotors exist mainly in quadcopter (Figure 2-4), hexacopter, and octocopter configurations and generally have an even number of rotors for torque cancellation. Multi-rotors are almost exclusively small UAVs, powered by electric motors. They afford the advantages of Vertical Take-off and Landing (VTOL) flight, the ability to hover and loiter on station, agility, and a relative freedom from vibration, but at the expense of limited range, altitude, and endurance [110].

![Figure 2-4: DJI Phantom 4 Pro, Quadcopter UAV](image)

The missions for which VTOL aircraft (e.g., helicopters and multi-rotors) are designed generally do not dictate high airspeeds. Moreover, where endurance and range may be sacrificed to obtain other desirable attributes, such as low vibration or manoeuvrability, a multi-rotor platform may be selected over a fixed-wing platform. The ability to fly in any direction, rather than in only a straight line, negates the advantages of streamlining, and multi-rotor designs, consequently, do not incorporate fairings or fillets or any other strategy to reduce form or interference drag. Aerodynamically, multi-rotors are very inefficient since antennae,
wires, controllers, and motors protrude into the airstream. In comparison to the design of fixed-wing UAVs, the low-speed operational envelope and directional agility of multi-rotor aircraft render the consideration of drag less important in the design process.

Although the technology is improving all the time, multi-rotors are fundamentally very inefficient and require a lot of energy just to fight gravity and keep them in the air. With current battery technology, they are limited to around 20-30 minutes when carrying a lightweight camera payload. Heavy-lift multi-rotors can carry more weight, but in exchange for much shorter flight times. Due to the need for fast and high-precision throttle changes to keep them stabilised, it isn’t practical to use a gas engine to power multi-rotors, so they are restricted to electric motors. So, until a new power source comes along, it is only expected that very small gains would be made in flight time [112].

Multirotor aircrafts, particularly quadrotors, have been the most capable vehicles in terms of accessibility, manoeuvrability, capacity for onboard sensors, and applicability to a breadth of applications. Great research progress has been made across multiple areas, including the control of agile manoeuvres; planning and perception in unknown, unstructured environments; and collaboration in multiagent teams, sparking a surge of industry investment. The first half of 2017 saw $216 million in venture capital funding for drone companies [113], spanning such applications as infrastructure inspection, rapid package delivery, precision agriculture, disaster response, and choreographed performances.

**Fixed wing UAVs:** Fixed-wing aircrafts (Figure 2-5) typically have a foam shell holding electronics and are hand, or mechanically, launched. Fixed wing drones require a higher initial speed and the thrust to load ratio of less than 1 to initiate a flight [114, 115]. They can fly for much longer durations of time and cover larger areas due to their high cruising speeds. Usually these drones are used for mapping large areas or surveying. They are best applied with programmed flights. Rudder, ailerons and elevators are used for yaw, roll and pitch angles to control the orientation of aircraft. Figure 2-6 shows the force applied on fixed-wing aircraft. Fixed wing drones cannot hover at a place, and they cannot maintain their low speed.
However, fixed-wing aircrafts generally need a larger area for take-off and landing. Fixed-wing aircrafts are also unsuitable for applications that require the aircraft to hover or stay at one point.
for extended periods of time. The life expectancy of both multi-rotor and fixed-wing UAVs varies dramatically based on flight conditions, maintenance, and component fatigue.

**Blimp UAVs:** A blimp UAV is a lighter than air (LTA) aircraft system. Figure 2-7 depicts an autonomous blimp UAVs designed by Kobe University, Japan [117]. Blimp UAVs have some advantages over multi-rotor systems since they can fly with lower noise, have longer endurance, have higher energy efficiency and are safer to use [118]. Blimp UAVs do not require energy for generating lift since it is an LTA system, and only requires energy for its propulsion systems. As a result, blimp UAVs can last longer for the same amount of energy. Another distinctive advantage of blimp UAVs is that it can shut down its propulsion systems and drift when not required and can activate these systems periodically to maintain its position and this results in conservation of energy.

Figure 2-7: Blimp designed by Kobe University, Japan [117]

Blimp UAVs are also safer in the event of failure or malfunctioning. For instance, a blimp UAV would not immediately lose altitude if it lost power or control. Since blimp UAVs do not constantly need power or control input to maintain lift, a loss of control or a slight error would not instantaneously cause loss of altitude. Also, a blimp UAV does not move rapidly,
and adequate time would be available for human intervention to prevent a crash. In the event of being unable to prevent a crash, the low speed of the blimp would very likely cause the blimp to come down very slowly and have a gentle impact. Unlike a multi-rotor UAVs that loses power and crashes, a blimp UAV along with its payloads is more likely to be recoverable. [119].

Blimp UAVs can also carry larger payload for the same amount of energy. The operating costs of blimp UAVs would also be lower than that of multi-rotor systems as the helium required for lift generation can be reused for multiple payloads. These advantages make blimp UAVs a viable alternative to multi-rotor UAVs for applications that require longer flight endurance. However, Blimp UAVs travel at lower speeds compared to multi-rotor UAVs and fixed-wing UAVs and are generally very large in size. These render them unusable for applications that require fast missions in a compact area.

2.2.2 Flight Control

In this section, various methods of aircraft control and conduction of different aircraft missions are reviewed. The recent work done in advancing automated flight control in multi-rotor UAVs is also surveyed.

The operation of the aircraft ranges from full manual control, to stabilized or “remote control,” to automated flight profiles without direct flight path control. The level of automation in the flight mission is dependent upon several factors, including, but not limited to, the amount of repetitious aircraft movements required, aircraft proximity to other objects, and the dynamic nature of the mission.

**Manual Control:** Under manual control the operator has direct, unassisted control of the aircraft’s flight path. The control input is typically applied through a handheld console that allows the operator to make fine changes in aircraft pitch, roll, yaw, and throttle. The console can be configured to provide exponential control depending on the degree of input applied so that fine inputs can be made with small inputs and large inputs will result in exponentially larger commands. The operator may also have direct control over other aircraft subsystems such as flaps, landing gear, and brakes.

Manual aircraft control provides a skilled operator with precise control over the aircraft’s flight path and predictable outcomes to control inputs. However, manual control requires
extensive operator training and experience to accomplish effectively and safely. Due to the difficulty of manually controlling an aircraft, many operators that are capable of full manual control have spent a lifetime flying remote control aircraft as a hobby [120].

**Stabilized control:** Under stabilized control the operator has direct, assisted control of the aircraft’s flight path. This type of aircraft control typically routes the operator’s inputs from a handheld console through an autopilot onboard the aircraft that translates the direct inputs into desired outputs. Stabilized control allows the operator to maintain direct control of the aircraft’s position but reduces the need for fine control to ensure a fixed-wing aircraft returns to wings level or a Vertical Take-off and Landing (VTOL) aircraft returns to hover. Some VTOL aircraft are equipped with a magnetometer that maintains a single direction as “away” from the operator so that, regardless of the aircraft’s orientation, away, left, right, and toward the operator remains constant. Stabilized control greatly reduces the operator skill level required to effectively and safely control the aircraft while still providing dynamic control of the flight path. The majority of VTOL systems are capable of stabilized control and this has resulted in significant growth of the VTOL market due to the ease of aircraft operation.

However, stabilized control means that the operator must be able to see the aircraft clearly enough to determine the precise orientation of the aircraft in relation to the object(s) being observed. Applications that require repetitive, precise positioning of the aircraft over an area of interest, such as aerial mapping, are difficult to conduct from only the ground-level perspective of an operator. [121]

**Automated control:** Under automated control the operator has indirect, assisted control of the aircraft’s flight path. This type of control is typically conducted through a graphical software interface that provides an overhead view of the aircraft’s position overlaid on aerial or satellite imagery. The operator can usually plan the mission in advance through the software’s planning tools and upload commands to the aircraft during flight to alter the flight path. The aircraft’s autopilot determines the control surface and throttle inputs to position the aircraft on the desired flight path in a 3-D space, and the operator observes the behaviour of the aircraft to ensure that mission is conducted as desired.

Automated control requires the least amount of direct operator skill for aircraft control; however, the multitude of software interfaces for UAVs vary greatly in complexity. Some interfaces are designed to provide only basic functionality and may be custom tailored to a specific aircraft and therefore only need high-level inputs from the operator. Other interfaces
may require operator input for every possible variable in the mission and can take a
significant time to learn. Regardless of the interface, automated control can greatly increase
the efficiency and reduce the workload required for a mission. Repetitive flight paths, such as
orbits and mapping missions, are particularly well suited to automated control. [122]

Tremendous progress has been made in the design and development of automated flight
control systems, which can roughly be categorized as either man-made or bio-inspired. In the
former class, fixed-pitch rotorcrafts have become particularly popular for their mechanical
simplicity, but several more complex vehicles have also been developed. For example,
decoupled rotational and translational degrees of freedom can be achieved with variable-pitch
[123, 124] or omnidirectional rotor configurations [125]. Passive vehicle stability, the ability
to fly stably without active attitude control can be accomplished with the addition of
appropriately designed cages [126], lowering vehicles’ energy consumption and increasing
their robustness to failures of inertial sensors. The research efforts of the government,
industry, and academia have positioned quadrotores to become one of the first flying platforms
with full end-to-end autonomy between high-level task specification and motor actuation. To
understand the dynamics and control of a simple quadrotor system, mathematical modelling
and control algorithms of an automated quadrotor UAVs are studied [127].

The quadrotor (illustrated in Figure 2-8) is a vehicle with four propellers, each rigidly mounted at
a distance L from the center of mass. The front and back rotors rotate anticlockwise, while the
left and right rotors rotate clockwise. Modulating the rotor thrusts allows the vehicle to roll,
pitch, and yaw, which in turn induces translational motion.

Let $I = \{e_1, e_2, e_3\}$ be the inertial world frame. Let $C = \{c_1, c_2, c_3\}$ be an intermediate frame
after yaw rotation $\psi$ and $B = \{b_1, b_2, b_3\}$ be a body frame fixed to the vehicle. Let $x_Q \in \mathbb{R}^3$
represent the position of the vehicle’s center of mass relative to $I$, $R \in SO(3)$ represent the body-
to world-frame rotation matrix (the body-frame components of vector $B v$ can be translated into
The front propeller is on the right world-frame components $\mathbf{v}$ with $\mathbf{v} = R^B v$ and $\Omega \in \mathbb{R}^3$ represent the angular velocity expressed in B. The system’s dynamics evolve on $\text{SE}(3)$, with state

$$x = [x_Q^T \quad \dot{x}_Q^T \quad R \quad \Omega^T]^T$$  \hspace{1cm} (Eq. 2.2.2.1)

and the input is

$$u = [f \quad M^T]^T \in \mathbb{R}^4$$  \hspace{1cm} (Eq. 2.2.2.2)

The thrust magnitude, $f$, acts in the $b_3$ direction, and the moment, $M$, acts about the body-frame axes. These result from individual thrust forces from each rotor, $f_i$:

$$\begin{bmatrix} f \\ M \end{bmatrix} = \begin{bmatrix} 1 & 1 & 1 & 1 \\ 0 & L & 0 & -L \\ -L & 0 & L & 0 \\ -\mu & \mu & -\mu & \mu \end{bmatrix} \begin{bmatrix} f_1 \\ f_2 \\ f_3 \\ f_4 \end{bmatrix}$$  \hspace{1cm} (Eq. 2.2.2.3)
where $\mu$ is a constant representing the ratio of drag to lift force produced by each propeller. Let $m_Q$ represent the vehicle’s mass, $J$ represent its inertia tensor in $B$, and $g$ represent the positive gravity constant. The quadrotor’s equations of motion can then be derived:

$$\dot{x} = \begin{bmatrix} \frac{f}{m_Q} & 0 & 0 & 0 & 0 & 0 \\ Re_3 - ge_3 & 0 & 0 & 0 & 0 & 0 \\ R\tilde{\Omega} & 0 & 0 & 0 & 0 & 0 \\ J^{-1}(M - \Omega \times \Omega) & 0 & 0 & 0 & 0 & 0 \end{bmatrix}$$  \quad \text{(Eq. 2.2.2.4)}

The hat operator, $\hat{\cdot}$, maps a vector to a skew-symmetric matrix such that $\hat{x}y = x \times y$

During autonomous operation, the quadrotor must track desired trajectories $x_{\text{des}}(t)$. A significant control challenge is handling the quadrotor’s nonlinear and underactuated dynamics. Fortunately, it can be shown that the dynamics are differentially flat [128]; that is, there exists a set of flat outputs, $x^f$, such that the model’s states and inputs can be written as functions of $x^f$ and its higher derivatives [129]. Mellinger & Kumar [130] showed that there exists a diffeomorphism between Equations 1 and 2 and the vector:

$$\begin{bmatrix} x_Q^T & \dot{x}_Q^T & \ddot{x}_Q^T & \chi_Q^T & \psi & \dot{\psi} \end{bmatrix}^T$$  \quad \text{(Eq. 2.2.2.5)}

Thus, the flat outputs are

$$x^f = \begin{bmatrix} x_Q^T \\ \psi \end{bmatrix}^T \in \mathbb{R}^4$$  \quad \text{(Eq. 2.2.2.6)}

![Figure 2-9: Architecture of a hierarchical quadrotor controller](image)

This is a powerful observation. When planning trajectories in the full state space $x$, Equation 2.2.2.4 must be included as nonlinear constraints. However, any sufficiently continuous trajectories in $x^f$ can always be associated with a full state trajectory that satisfies Equation 2.2.2.4.
This facilitates the design of a hierarchical controller, illustrated in Figure 2-9. The superscript “des” references desired values calculated by a trajectory planner, while the superscript “c” (for “commanded”) references intermediate values calculated by the controller. The outermost position controller is typically a traditional proportional–derivative controller:

\[ F = -k_x(x_Q - x_Q^{\text{des}}) - k_v(v_Q - v_Q^{\text{des}}) + m_Q(x_Q^{\text{des}} + g e_3) \]  
\( \text{Eq. 2.2.2.7} \)

where \( k_x \) and \( k_v \) are positive gain matrices. From this,

\[ f = F \cdot R e_3 \]  
\( \text{Eq. 2.2.8} \)

To derive \( R^c \), the third body-frame vector can be defined as

\[ b_3^c = \frac{F}{\|F\|_2} \]  
\( \text{Eq. 2.2.9} \)

and a desired direction for the intermediate frame axis, \( c_1^c \), can be defined with

\[ c_1^c = \begin{bmatrix} \cos(\psi^{\text{des}}) & \sin(\psi^{\text{des}}) & 0 \end{bmatrix}^T \]  
\( \text{Eq. 2.2.10} \)

Assuming \( R \) is a \( z-x-y \) rotation matrix, axes \( b_1 \) and \( c_1 \) are coplanar, and the remaining body-frame vectors are

\[ b_2^c = \frac{b_3^c \times c_1^c}{\|b_3^c \times c_1^c\|_2} \]  
\( \text{Eq. 2.2.11} \)

\[ b_2^c = b_2^c \times b_3^c \]  
\( \text{Eq. 2.2.12} \)

Note that \( x b_3^c \times c_1^c \neq 0 \); that is, the thrust vector cannot be parallel to \( c_1 \). The desired orientation is then

\[ R^c = [b_1^c \quad b_2^c \quad b_3^c] \]  
\( \text{Eq. 2.2.13} \)

From here, an attitude controller commands an input moment, \( M \), to track \( R^c \), and an innermost controller calculates individual motor commands. In practice, each inner loop must run
significantly faster than its previous layer. Thus, the position control loop can optionally be executed on an external base station, but the speeds demanded of the attitude and motor controllers require them to run on onboard processors.

Proposed attitude controllers have commonly taken the form

\[ M = -k_R e_R(R, R^c) - k_\Omega e_\Omega(\Omega, \Omega^c) + M_{ff} \]  
(Eq. 2.2.2.14)

where \( k_R \) and \( k_\Omega \) are positive gain matrices, \( e_R \) and \( e_\Omega \) are error functions, and \( M_{ff} \) is a feedforward moment. In earlier works, the dynamics were linearized about the hover equilibrium and the attitude controller was implemented in Euler angles [131]. This linearization performs well for roll and pitch angles up to approximately 30°, corresponding to linear velocities of approximately 1.5–2 m/s; however, nonlinear controllers have largely eliminated these limitations.

For example, in a proposed geometric controller [132],

\[ e_R(R, R_c) = \frac{1}{2} \left( (\dot{R}^c)^T R - R^T R^c \right)^v \]  
(Eq. 2.2.2.15)

where \( v \) is the map from a skew-symmetric matrix to its vector representation. This defines the attitude error directly on the manifold SO(3) (i.e., Equation 2.2.215 returns the axis-angle representation of the rotation from \( R \) to \( R^c \)). Similarly, the angular velocity error is

\[ e_\Omega(\Omega, \Omega^c) = \Omega - R^T R^c \Omega^c \]  
(Eq. 2.2.2.16)

where

\[ \Omega^c = (R^c)^T \dot{R}^c \]  
(Eq. 2.2.2.17)

\( \dot{R}^c \) can be found by differentiating Equation 2.2.2.13 and substituting values calculated from differential flatness equations. Finally, the feedforward moment is defined as

\[ M_{ff} = \Omega \times J \Omega - J(\Omega \times R^T R^c \Omega^c - R^T R^c \dot{\Omega}^c) \]  
(Eq. 2.2.2.18)

where \( \dot{\Omega}^c \) can again be found through differential flatness. By expressing the control equations in a coordinate-free, singularity-free manner, this controller can guarantee almost global exponential attractiveness [132], and in practice, it can control the quadrotor at almost inverted
orientations. Yu et al. [133] presented a similar formulation but decomposed the attitude controller to track the fast-response roll and pitch angles before the slow-response yaw angle, and Brescianini et al. [134] presented a quaternion-based controller that also has singularity-free, globally asymptotic stability properties.

The design of the innermost motor controller is also crucial. This controller typically involves two steps. In the first step, the calculated inputs $f$ and $M$ must be translated into individual thrusts $f_i$. Assuming that $f_i$ can be achieved almost instantaneously, Equation 2.2.2.3 can simply be inverted. This assumption has proven practical for slow-moving vehicles; for high-speed flight, however, incorporating a first-order dynamic model for $f_i$ can noticeably improve performance [135]. Furthermore, in many works, when the calculated $f_i$ exceeds a set maximum, the thrust is simply clipped to $f_i = f_{\text{max}}$. However, this can result in unpredictable tracking errors. Thrust-mapping methods that deliberately reassign $f_i$ according to set criteria (e.g., prioritizing achieving the desired roll and pitch over yaw) can result in more robust behaviour [135, 136]. Faessler et al. [135] further showed that modeling $\mu$ as a variable dependent on $f_i$ also improves performance. Alternatively, nonlinear model predictive control schemes can be used to directly obtain outputs $f_i$ to track the desired $R^c$ and $\Omega^c$ [137]. Here, an optimal control problem is solved in a receding-horizon fashion to yield the motor thrusts at each time step. This formulation has the advantage that thrust and angular velocity limits can be directly incorporated into the optimization, but it is more computationally expensive.

In the second step, motor commands $\omega_{r,i}$ must be computed from forces $f_i$. A basic model relates

$$f_i = k_F \omega_{r,i} \quad \text{(Eq. 2.2.2.19)}$$

where $k_F$ is a motor constant found through calibration. Recently, however, researchers have begun to adopt a polynomial model [135, 138]:

$$f_i = k_2 \omega_{r,i}^2 + k_1 \omega_{r,i} + k_0 \quad \text{(Eq. 2.2.2.20)}$$

Bangura & Mahony [139] further proposed a hierarchical motor controller that explicitly models aerodynamic effects. A final challenge is maintaining safety and stability in the presence of unexpected disturbances. Small aerodynamic effects that are usually rejected as unmodeled disturbances become significant near surfaces (e.g., the ground or obstacle walls) and other vehicles [140]. In these cases, the effects must be explicitly modelled through techniques such as experimental identification of drag constants [141] or numerical calculation.
using a computational fluid dynamics solver [142]. Alternatively, Yao et al. [143] predicted future disturbances from past observations in flight, and Bartholomew et al. [144] predicted aerodynamic effects around obstacles using observed depth images and prior training data. For safety in the presence of mechanical failures, Mueller & D’Andrea [145] experimentally demonstrated controllers that enable stable flight even following the loss of one or two propellers.

These sophisticated models and nonlinear control strategies have enabled quadrotors to perform manoeuvres that fully utilize the systems’ entire range of motion, such as perching on vertical surfaces [146], a feat that would not be feasible with previous linearized controllers.

2.2.3 Weather impacts on UAV operation

In this section, impact of weather conditions on UAV operations is studied. Operating UAVs under poor weather conditions can result in reduced visibility, loss of communication and loss of control; all of which can lead to loss of aircraft. Additionally, these operators and observers of UAVs exposed to these conditions may impair their ability to see and control the aircraft [147]. To conduct safe and successful operations using UAVs, it is important to understand how different forms of weather affect the UAV operation. Weather hazards for UAV operations generally range from moderate, to adverse, to severe.

Moderate weather hazards are classified as those that result from a weather condition which can cause reduced visibility but otherwise do not harm the aircraft. Moderate weather conditions generally include dense fog, haze, glare and cloud cover. Adverse weather hazards are classified as those that result from weather conditions that have the potential to cause loss of communication, loss of control, poor aerodynamic performance and loss of operator’s ability to perform operations required to complete flight mission. Adverse weather conditions include strong winds, gusts, turbulence, heavy rain, solar storms, humidity, snow, ice and extreme temperatures. Finally, severe weather hazards are situations that can cause severe damage to or loss of aircraft and put the operator in a dangerous situation. These conditions include thunderstorms, lightning, hail, tornadoes and hurricanes. Flying in severe conditions is generally avoided by the UAV operators. As such, impacts of moderate and adverse weather conditions on UAV operations are reviewed in the following sections. [148]

Glare: Glare is a weather phenomenon that occurs in clear skies and can affect the visibility of an operator [149]. Firstly, it can hinder direct observation of the aircraft. In addition to the
difficulty of spotting a small aircraft on a bright day, looking too close to the sun can result in watery eyes and spotted vision. In preparation for a mission on a sunny day, visual observers should take care to pack sunglasses.

Secondly, UAV operations often require a user interface displayed on a monitor, tablet, or other screen that enables the operator to plan flight missions, send commands, trim and change control derivatives, or track the aircraft, while receiving telemetry or data from the payload. If the monitor is not bright enough, the reflection of the sun on these screens can overpower LCD brightness such that it can be difficult to see or send the correct information to navigate or control the aircraft. To mitigate this problem, the operator should shield the display from glare, either by operating from a shaded location or using a display hood.

**Wind, Gusts and Turbulence:** Wind and turbulence play the largest role in manned aviation weather accidents. A study conducted by the [150] on data from 2003-2007 on weather related aviation accidents showed that wind accounted for 53.6% of manned aircraft accidents, higher than any other factor by 35%. This number was even greater for smaller non-commercial manned aircraft. Regarding UAV accidents, 297 accidents involving UAVs were cited between October 2009 and April 2015 in the preliminary FAA report [151]. Only 3% of these incidents were related to weather and turbulence. These numbers do not give a clear picture of how wind conditions affect UAV operations since 60% of the data in this report comes from large military UAV, such as the Predator and Guardian, and give no sense of the numbers of unreported accidents for UAV.

Despite limited data for UAV accidents, it stands to reason that wind affects these lighter weight aircraft in a similar manner, yet more radically than large UAV or manned aircraft. The major ways in which affects UAV include reduced endurance, poor control and changed flight trajectory.

Strong winds have the capacity to affect the ground speed and flight path of an unmanned aircraft. Similar to larger manned aircraft, headwinds and tailwinds, respectively, increase or decrease the ground speed of UAV. Unlike typical manned aircraft, however, wind speeds can easily surpass the maximum speeds of UAV. Figure 2-10 shows a histogram of the maximum flight speeds listed for 92 different UAV compared to the Beaufort wind scale. This histogram shows that over half of the UAV surveyed have maximum speeds that are below the wind speeds one would find in a storm. In general, multi-rotor aircraft have slower maximum speeds
than those of fixed-wing aircraft, which make them more likely to struggle even in lower wind speeds.

Figure 2-10: UAV maximum speeds compared to Beaufort wind scale. The darker brown indicates overlap between fixed wing and multi-rotor aircraft /152/

Flying a multi-rotor or a fixed-wing UAV in a headwind with a greater velocity than that of the aircraft results in stationary or backwards flight. Furthermore, flying in a crosswind can cause the UAV to drift with the crosswind rather than at the intended heading. Strong winds may result in the UAV to be blown over a populated, dangerous, or unrecoverable area. It could also lead to the UAV being blown into an area or to an altitude where one can no longer see the aircraft. Moreover, winds are often associated with gusts that can easily be a factor of two greater than the sustained wind speed.

Wind gusts, wind shear, and turbulence all have the potential to reduce aircraft control. Aircraft control refers to the ability to manoeuvre the aircraft by use of pitch, yaw, and roll.
Pitch changes an aircraft’s angle of attack, yaw changes the aircraft’s heading, and roll rotates the aircraft around the axis of the fuselage Figure 2-11. These change the aircraft’s altitude, the direction the nose is pointed, and turn the aircraft, respectively.

![Figure 2-11: The effect of wind gusts on the pitch, yaw, and roll of an aircraft. Smaller arrows indicate smaller magnitude wind gusts](image)

Wind gusts are sudden increases in wind speed that typically last no longer than 20 seconds [153]. Horizontal gusts affect the yaw of a fixed wing aircraft by blowing against the rudder. For roll stability, most low-wing aircraft are designed with dihedral, where the wings are angled upward from the horizontal plane as shown in Figure 2-12. A horizontal gust can roll the aircraft by blowing underneath one of the wings, and because banking (rolling) an aircraft changes its direction of flight, horizontal gusts are particularly dangerous while flying near obstructions.

![Figure 2-12: Dihedral angle on an aircraft wing](image)

Vertical gusts can roll a fixed-wing UAV if there is a gradient in the gust’s magnitude from one wing to the other. For a fixed-wing or multi-rotor aircraft, a gradient between the front and the back of the aircraft can cause a pitching or diving motion (Figure 2-11). If the sudden pitch caused by a gust exceeds the critical angle of attack for the UAV, the wing will stall, meaning there is no longer enough lift produced by the wing to keep the aircraft flying. If the vertical gust is strong enough, this may even tip the UAV upside down. [155]
Wind shear and turbulence affect UAV in a similar manner to gusts. Wind shear refers to changes in wind speed or direction that often occur due to strong temperature inversions or density gradients [156], while turbulence refers to changes in density or air flow velocity that result in chaotic mixing motions in the air [157]. Because wind shear and turbulence are associated with sudden changes in airflow, their effect on aircraft control is unpredictable and difficult to compensate for. UAV generally fly at low altitudes where wind shear and turbulence are worsened by obstructions such as buildings, trees, or mountains. Additionally, from the ground to a height of approximately 50 m, strong vertical wind shear and small-scale turbulence are caused by varied atmospheric and land surface processes, convection, and surface roughness in the natural planetary boundary layer [158].

Wind reduces the endurance or flight time of an aircraft if it causes higher than expected current draw from the batteries. To fly at a constant ground speed in a headwind or crosswind, or to compensate for sudden changes in aircraft motion due to turbulence, the thrust produced by the motor must increase. Figure 2-13 shows the change in battery endurance that occurs when increasing the thrust of a typical UAV propulsion motor.

![Endurance v. Thrust for Different 3S Batteries](image)

Figure 2-13: Endurance (flight time) decreases by half its value as thrust of motor increases from 50% to maximum [152]
The endurance values calculated here assume a constant current draw and a linear relation to the battery capacity,

$$t = \frac{Q}{I} \quad (Eq. 2.2.3.1)$$

where $t$ is the endurance, $Q$ is the battery capacity and $I$ is the current draw. For example, a battery capacity of 2 Ah drained at 4 A should be able to produce half an hour of flight time. In reality, there would be less than half an hour due to the Peukert effect, which states that as the current draw increases, the battery capacity becomes less effective. The endurance calculation adjusted for the Peukert effect [159] is

$$t = \frac{R_t}{I^n} \left( \frac{Q}{R_t} \right)^n \quad (Eq. 2.2.3.2)$$

where $R_t$ is the battery hour rating (i.e. discharge time over which the capacity was determined) and $n$ is a constant discharge parameter dependent on the temperature and type of battery [160]. Since most multi-rotor UAVs have an endurance of 30 minutes or less, the rapid decrease in battery life due to windy conditions results in a much shorter flight than anticipated and may put the operator in a position where sighting or controlling the aircraft for a safe landing might be impossible before the battery capacity is exceeded.

**Temperature:** UAVs are generally operated between 253 K and 323 K. However, operating temperatures can sometimes range from 223 K to 343 K. Extreme temperatures can severely dent the aerodynamic performance as well as damage physical components of a UAV. Batteries are most commonly affected component of a UAV resulting from extremities in temperatures. Batteries generally used in UAV are nickel-cadmium (NiCad), nickel-metal-hydride (NiMH), and lithium polymer (LiPo). Because NiCad and NiMh batteries lose a significant portion of their charge daily and are heavier than LiPo batteries, LiPo batteries are the more common battery type used in UAV [161].

The standard operating temperatures for LiPo batteries are 253 K to 318 K. Although high temperatures rarely present a hazard during flight, continued charge or discharge at these temperatures will reduce the life cycle of the battery. On the opposite end of the spectrum, as a typical battery’s temperature decreases from about room temperature, the internal resistance increases and the chemical reaction that produces a current across the two battery leads slows considerably [161]. In other words, capacity of LiPo batteries drops significantly, as exemplified [162] in Figure 2-14.
In this graph, an 850 mAh LiPo battery was discharged from 4.2 V to 3 V at various temperatures. At -20°C (253 K), the battery lost 30% of its listed capacity. Flying in temperatures lower than this may cause the battery to alter its behaviour or stop functioning altogether. This altered behaviour is the main concern for the other electronics onboard the aircraft as well.

UAV airframes are generally made from wood, plastic, carbon fiber, foam and metal. If one uses a plastic in construction, low temperatures can cause the plastic to become brittle and crack under force of impact or when screwing down parts. For example, polypropylene becomes brittle at 253 K [163]. This material also has a tendency to deform in high temperatures. As a result, tougher plastics are more frequently used in construction, such as Polycarbonate or ABS for the airframe and Nylon for propellers.

**Humidity:** Humidity can affect UAV operations if the moisture in the air is condensed on the electronics used in a UAV. Water can damage the on-board electronics resulting in erroneous behaviour, loss of functionality or high amounts of heat output possibly leading to a fire. A short that results in an electrical fire is particularly dangerous when operating with LiPo batteries. These batteries release hydrogen as part of their discharge. If a LiPo battery is
damaged, the build-up in hydrogen combined with the sparks from a short can result in an explosion [164]. For this reason, LiPo batteries are contained in plastic housing and sealed with Kapton tape or other insulating materials to prevent damage to the battery [165]. This being the case, small amounts of water on the battery itself will most likely cause no harm. The danger comes from the potential for other electronics to catch fire near the battery. Maximum relative humidity specifications on UAV range typically from 50% to 100%. Humidity is a greater problem in the morning, when there are relatively lower temperatures and higher humidity levels. Relative humidity varies significantly with geographic location. In areas with high average humidity, it is particularly important to check humidity levels before flight and verify that electronics are watertight [166].

**Solar storms:** Solar storms, such as flares and coronal mass ejections (CMEs), disrupt GPS transmissions. Most UAV use GPS to determine the position and speed of the aircraft, provide altitude and location information for Earth observations, and in some autonomous systems, steer the aircraft [167].

GPS radio signals travel from a satellite through the ionosphere to the receiver on the GPS module of a UAV. GPS systems use a model to compensate for the ionosphere’s effect on the accuracy of position, but when solar flares occur, these models no longer approximate the average ionosphere correctly [168]. Solar flares interfere with the ionosphere by sending electromagnetic radiation and increasing the number of electrons present. In some cases, solar flares have such an impact on the ionosphere that the position is off by many meters or, in worst-case scenarios, the GPS receiver cannot locate the satellite signal at all. CMEs are an even greater hazard for GPS systems. CMEs eject particles from the sun that reach the Earth in three to five days. These particles can collide with GPS satellites and disrupt the electronics on board [169].

Much like atmospheric weather, solar storms are forecasted and monitored by NASA and NOAA. These forecasts are available online and reported to electric companies, airline pilots, and spacecraft operators. As such, UAV operators can account for solar storms during mission planning if they intend to rely heavily on GPS navigation systems.

**Fog, Clouds and Haze:** One of the most common payloads on UAV is an onboard camera. Dense fog, clouds, or haze reduce the distance a camera can see. Consequently, flying in these situations is dangerous as UAV may fly into buildings, manned aircraft, power lines, vehicles, and any other number of hazardous objects.
Fog is produced when the temperature of the air near the ground cools to the air’s dew point, that is, when saturation is reached (100% humidity) and clouds form [170]. Flying through clouds can result in condensation on the camera lens. Haze is where dust, smoke, or other particulates obscure the sky and most commonly associated with air pollution. Therefore, one is more likely to encounter haze in urban areas [171].

Some UAV use other sense-and-avoid technologies in conjunction with the on-board camera to detect motion and other objects in the area. Some of these include light detection and ranging (LIDAR), traffic collision avoidance system (TCAS), and automatic dependent surveillance broadcast (ADS-B). However, these do not solve many of the issues presented by fog, clouds and haze. LIDAR, for example, cannot penetrate fog and clouds, and is limited in dense haze. ADS-B and TCAS can sense other manned aircraft but cannot sense terrain or buildings. That being stated, an operator should take the necessary precautions when entering areas of dense fog, haze, or low-altitude clouds. [172]

2.2.4 UAV applications

The rise in drone deployment has increased exponentially in the last decade because of the introduction of multi-copters. As the use of drones has numerous benefits, one being low risk to human life, drones can be used for situations such as storm data collection, forest fire surveillance and battlefield reconnaissance. Local uses include surveillance of national parks to ward off poachers and gather information on wildlife ecology. Extensive research is being conducted on the use of drones for the delivery and transportation of clinical specimens, Point of Care (POC) devices and urgent medication in rural environments.

Drones have a large variety of applications and fields in which they can be employed.

- **Medical/Emergency** - any procedure that requires broad area search or information gathering for health, police, and/or firefighting personnel is placed in this category. Many issues are addressed here, including dispersal of medical supplies and humanitarian aid in natural disasters, hazardous materials and wildfire assessment and mitigation, missing persons response, as well as rally, riot, and protest monitoring [173]. Volcanic ash surveying and eruption damage assessment, search & rescue, and biological sampling or lab specimen transportation also fall under this category [174]. Displaced technology includes helicopters, handheld cameras, and satellites.
The use of drones to transport these samples and POC devices may aid in response time between the laboratories and doctors. An added advantage is that it is possible to transport medical samples for a patient in critical condition in inaccessible locations. Whereas previously a motor vehicle hindered by traffic and road accessibility would be required to transport the samples, a drone can now be used which does not have to follow geographical routes. With the use of UAVs, it is possible reduce the price of testing for patients as the testing fee covers transportation of the samples. This method may also improve the overall efficiency of the small parcel delivery system. [175]

**Atmospheric information collection:** Atmospheric sampling is one of the few applications for UAVs that does not relate directly to imaging or interacting with ground-based objects. Instead, atmospheric sampling involves the sensing or collection of airborne particulates or gases to identify the characteristics of the atmosphere. Atmospheric sampling has been performed for many years using manned aircraft and balloons, but UAVs bring a new capability to sample air more effectively and in regions that were previously more challenging to sample, such as extreme low and high altitudes [176]. UAVs can also assist in understanding weather patterns and forecasting by providing information about the temperature, wind speeds, humidity, and other variables at multiple altitudes. [177]

**Hazardous material detection:** Another type of atmospheric sampling is the detection of airborne hazardous materials. Detecting toxic substances in the air is critical in identifying events that may be hazardous to humans or the environment. Gas leaks are likely the most common hazardous substance that may become airborne and locating these leaks can be challenging because the gas may be odourless and colourless, while still being hazardous. UAVs are ideal for this application since no human is onboard an unmanned aircraft, and the aircraft can be flown through areas with known hazards to identify and quantify the substance and potentially locate the source. Gas leaks from chemical plants, petroleum pipelines, or other sources can be found using this method, and early research indicates that multirotor aircraft can even enhance currently available sensors due to the air movement of the rotors increasing the flow over the sensors. [178, 179]

- **Imaging and Recording** - these operations include any form of media, arts, or entertainment. Mainly these drones will be used for making movies, filming stunts, or recording televised news. Drone journalism provides low-cost local news coverage and weather assessment without endangering personnel using other transportation. Some current
technology and workforce that are displaced by UAVs in this field include helicopters for news coverage, storm chaser personnel, film movement rails or stands, and handheld video cameras. [180]

- **Infrastructure monitoring** - this category primarily addresses the assessment of structural components such as road integrity, bridge fatigue, powerline condition, mining safety, pipeline corrosion, rooftop inspection, and many more. This takes an at-risk group in the manned workforce and replaces, or supplements, them with machines. Difficult-to-reach areas are also part of this category and include analysis of wind turbine defects, property assessments/tax valuations/appraisals, insurance claims, oil spills, gas leaks, and industry accident notification. [181]

- **Monitoring/environmental surveillance** - this category encompasses wildlife conservation and environmentalism, habitat monitoring, endangered species tracking, and antipoaching operations. Drones are already being tested to capture heat signatures of black rhinos and aid in the arrest of illegal poachers [182]. The health of humpback whales is being assessed by drone collection of mucus expelled from their blowholes and later tested [183]. Another application is permafrost analysis in the Arctic to determine climate change. Drone image collection of specific areas for the purpose of intelligence analysis is another application of UAVs in this field. Examples of this include border patrol, traffic monitoring, collision avoidance in autonomous ships, and bacteria bloom detection in water systems [184]. Companies are exploring the possibilities of replacing helicopters, manned aircraft, and ground vehicles used to observe the environment with UAVs. [185]

- **Precision agriculture** - this category involves the analysis of crop health or surveying of land specific to agriculture and food products. These operations include monitoring livestock, surveying fields of crops, analysing irrigation and drainage systems, predicting crop yield, surveying the need for chemical and pesticide application, and detecting diseases in plants. Technology that is potentially displaced by implementing drones in this field include tractors, field sprayers, and sprinklers. [186]

- **Other** - all other UAV uses that cannot be classified in the categories above fall under “other.” This section includes UAVs for education, R&D, real estate, delivery, and more.
Chapter 3 Liquid on Superhydrophobic surfaces

3.1 Introduction

To efficiently develop UAV systems for bio-chemical applications, it is important to study the behaviour of liquid during transportation on a UAV. Poor understanding of liquid behaviour during UAV flights can alter the properties of bio-fluids transported and can render the sample unusable due to the possible inaccurate diagnostics data. Superhydrophobic surfaces serve as an excellent testing surface to study the characteristics of a liquid due to their non-wetting behaviour. In this section, the formation and transport of droplets on superhydrophobic surfaces is studied. The stability of a droplet impacted by using a superhydrophobic surface is also reviewed.

3.2 Transformation of drops on an SH incline

3.2.1 Background

Microfluidic systems offer the ability to greatly advance biochemical analysis [187, 188]. There is increasing awareness in the community today that continuous and closed - microfluidic flow devices are inherently difficult to integrate and scale due to the flow at any one location being dependent on the flow properties of the entire system. Discrete, independently controllable sample volumes [189, 190, 191], alternatively, permit the microfluidic function to assume a set of basic repeated operations, whereby one unit of fluid can be moved over one unit of distance, thus facilitating the use of hierarchical and modular approaches that then make up flexible and scalable system architectures with high fault tolerance capabilities. Moreover, because sample volumes can be controlled independently, such systems offer greater potential to be reconfigured whereby groups of units in an array can be altered to change their functionality, With the increasing ability to fashion surfaces exhibiting superhydrophobicity (SH), there is now substantial effort aimed at harnessing them as part of the microfluidic system for biochemical applications [192, 193, 194, 195]. The added attractiveness is the ability to transport liquid analytes or samples with lower loss and contamination, and recent reports of relative ease and cost in creating these surfaces [196].
One of the basic requirements in using drops in biochemical applications is their creation. The most well-known approach for the delivery of discrete volumes is via automated pipettes, although piezoelectric micro-dispensers are now also widely adopted [197, 198]. With the latter, there is a potential for damage of cellular material due to the acoustic forces developed [199]. Both technologies are understandably difficult and expensive to incorporate into a disposable microfluidic device itself, and even more so if the substrate is superhydrophobic since drop movement is rapid. Yet the underlying physics of discrete liquid dispensing systems involves intervening with the liquid continuum such that a pinch off occurs. When a jet (a stream of matter having a more or less columnar shape) is subject to surface energy changes, it will pinch off into a stream of drops [200, 201]. Such an approach will generally not be feasible for incorporation into microfluidic devices due to the relative long distances needed for transformation, and the high speed of the drops when they contact the SH surface [202, 203] which will lead to difficulties in control. A method that overcomes these limitations is presented in the following sections.

3.2.2 Numerical Modelling

A liquid drop that forms statically on a horizontal surface (Figure 3-1A) is often taken to be semi-spherical at low volumes and generally conforms to Young’s equation [204] that relates the equilibrium contact angle $\theta$ to the interfacial tension forces between liquid and solid, liquid and vapor, and solid and vapor phases, $F_{SL}$, $F_{LV}$, and $F_{SV}$ respectively, as

$$F_{SV} - F_{LV} \cos \theta + F_{SL} = 0$$

Eq. 3.1.2.1
Figure 3-1: Schematic description of the static model of a drop on a horizontal (A) and an inclined (B) SH surface

This equation assumes chemically homogenous, rigid, inert, atomically smooth surfaces which often cannot be physically attained. When these conditions cannot be met, the surface cannot have an equilibrium contact angle, leading to the contact angle as having hysteresis. When the hysteresis is time independent, it is often taken to be of the thermodynamic kind. If the surface is tilted by $\phi$ (Figure 3-1B) for instance, the advancing angle $\theta_a$ and receding angle $\theta_r$ depart from $\theta$ due to hysteresis, permitting the drop to stay on the surface despite the presence of a gravitational force $\rho V g \sin \phi$, where $V$ is the volume, and $\rho$ the liquid density. Such a situation has been widely investigated [205, 206]. For a drop that experiences small amounts of hysteresis, the limiting tilt needed to move the drop is given by [207]

$$\sin \phi = \frac{\gamma}{\rho g \left( \frac{96}{\pi V^2} \right)^{\frac{1}{3}} (\cos \theta_a - \cos \theta_r) \left[ (1 + \cos \theta_a)^{\frac{3}{4}} (1 - \frac{3}{2} \cos \theta_a + \frac{1}{2} \cos^3 \theta_a) \right]^{\frac{3}{4}} \left( \frac{\cos \theta_a + 2}{3} (1 - \cos \theta_a)^{\frac{9}{4}} \right)^{\frac{3}{4}}} \quad \text{Eq. 3.1.2.2}$$

for the surface tension being $\gamma$. From such a static model, one can cursorily conceive that the droplet volume will increase until gravity force exceeds the hysteresis retention forces, after which it will pinch off and roll down the incline. This leads to the obvious expectation that the volume of the discrete drops generated should be constant regardless of how fast the drop grows. An SH surface should meet the condition demanded of in EQ. (2) and can conveniently thus be made to be integral with the development of a biochemical analysis device with droplet transport capabilities. It should be noted that the hysteresis such as that depicted in EQ. (2) can be time dependent. In such a case, it is described as kinetic in nature.
Kinetic hysteresis is generally attributed to the surface being deformable, mobile, orientating, and when liquid does not penetrate the surface fully.

### 3.2.3 Experimental methodology

In the experiments, the SH substrate is a copper plate with a small hole of 1mm diameter drilled through, polished earlier to remove all visible scratches using silicon carbide electro-coated water-proof abrasive paper (KMCA, WET/DRY S85 P600). Prior to use, it was first cleaned using absolute ethanol, allowed to air dry, and then immersed in a 24.75 mM aqueous solution of silver nitrate (AgNO₃) for 1 minute to form the micro and nano structures. After this, it was rinsed with copious amounts of distilled water followed by absolute ethanol before being allowed to air dry. Once dried, it was immersed in a 1 mM solution of the surface modifier CF₃(CF₂)₇CH₂CH₂SH in absolute ethanol (ethanol with low water content) for 5 minutes. After removal, it was again rinsed with copious amounts of distilled water, followed by absolute ethanol, and then air dried. The SH plate was located on a rotary stage and tilted at 45° to the horizontal. Distilled water was delivered via a flexible tube and adaptor located at the bottom of the plate through a programmable peristaltic pump (NE9000 New Era). Videos of the drops forming on the SH surface were recorded using a high-speed camera (Fastec) at 250 frames per second. A second acrylic plate was made and tested in a similar way to provide surface effect comparisons.

### 3.2.4 Results and Discussion

The most evident observation from the video footages was that all drops did not have the delivery orifice at the center (as depicted in Figure 3-1B) but rather with the rear contact line coinciding with it (see Figure 3-2). Formation of droplets for flowrates under 8mL/min is defined as Regime I. This indicated a propensity of the advancing contact line to breach ahead of the rear contact line. Such a behaviour had been reported previously [208] although the amount of contact angle hysteresis shown at the rear line exceeded what was previously recorded on a SH surface [209]. Clearly, the orifice itself served as a strong pinning site for the rear contact line. Cursorily this appears to be consistent with reports of how surface discontinuities have been able to provide strong pinning for drops [210].
The orifice however does not have geometries that resemble a normal surface discontinuity. Hence, there is a need to consider the pinning mechanism more carefully (see Figure 3-3). It is well known the SH character is attributed to micro and nano scale surface structures that allow the drop to rest on pockets of trapped air leading to the Cassie wetting state. The surface tension forces holding the drop-in place however act only at the three-phase contact line [211]. When the rear contact line moves to the input orifice, this contact line no longer encounters a predominant Cassie state. One way to view this is to treat the orifice as one big well region (from the microscopic perspective) having a Wenzel wetting state in which the rear contact line resides at. This offers one rationale for the additional pinning. As walls of the orifice are perpendicular to the surface, it is also likely that additional hysteresis has been provided by an edge effect. The contribution of this effect has been reported [212] and has recently been usefully harnessed for improved transparency micro plating [213]. The increasing weight of the drop, due to the continuous delivery of liquid through the orifice into it, will cause the rear contact angle to reduce. Eventually, this will pinch the drop off from the liquid body in the orifice, causing the contact line to “jump” over the orifice. On making contact with solid however, a predominant Cassie wetting state is encountered which will quickly allow the drop to detach and travel down the surface. Clearly the added pinning allows more liquid to be delivered into the drop to allow it to grow in size before detachment. This is underlined by control experiments conducted in which drops flung from a pipette tip onto the surface were found to displace at smaller volumes on the incline than drops delivered from the orifice. The
former manner of drop delivery was of course random and not practicable in a biochemical analysis system.

Two distinct regimes of drop behaviour were uncovered. These were dictated by the flowrate of liquid into the drop. The first regime, which is conveniently denoted as I, occurs for flowrates below 8 mL/min. From the example in Figure 3-3, which falls within this regime, the drop was elongated, clearly a consequence of the infilling liquid and gravity tending to stretch it perpendicular to and along the incline respectively. The latter increased its influence when the drop grew in size. Figure 3-4 (a) – (c) are images of drops just prior to pinch-off at increasing flowrates which seemed to show length increase. The plot of the elongated drop length (the distance between the rear and front contact lines) against flowrate (Figure 3-5) confirmed this and indicated a somewhat linearly increasing trend. The influence of gravity in the process was attested to by the lengths exceeding the capillary length \( = \frac{\gamma}{\rho g} \) of water which is around 2mm. The increasing length with flowrate can be attributed to the dynamic effect of pressure in the liquid body pushing more strongly (with increasing flowrate) against the liquid-gas interface. That the resistance offered by the front contact line was lower than the rear then allowed the drop to elongate in response to this. When the drop detached from the surface, it could very quickly assume a spherical shape down the incline which allowed the volume to be approximated.
Figure 3-4: Images of the drop just prior to detachment at flowrates of (a) 2 mL/min, (b) 4 mL/min, and (c) 6 mL/min

Figure 3-5: Plot of the length of the drop just prior to detachment against the inlet flow rate

The plot in Figure 3-6 shows that volumes of the drops in regime I was almost independent of the flowrate. Hence, while the pressure in the liquid due to the infilling liquid was able to distort the shape of drop, the ability to detach from the surface remained controlled by the three-phase mechanics acting at the rear contact line. This is borne out by the advancing and receding contact angles measured which remained rather invariant (see Figure 3-7). The hysteresis of 50° was much larger than what would be expected on a superhydrophobic surface and was testament to the strong pinning that occurred at the rear contact line coincident with the inlet orifice.
Interestingly, in comparison with the previous situation of liquid delivered on higher energy surfaces \cite{214}, the rear-contact line could not pin indefinitely to form a rivulet here notwithstanding the low capillary number (CA) values from small flowrates used. Due also to the low energy of the surface, no “cornered” shape of the rear contact line could develop. This meant that no daughter droplets could form during detachment, making it favourable for biochemical applications in terms of preventing sample losses.
A curious behaviour of the liquid body happened in regime II (at flowrates in excess of 8mL/min). Here, an initial “lifting” of the front contact line from the surface could be observed (see Figure 3-8). This resulted in the liquid thread performing somewhat of an initial “wheelie”. Intriguingly, this analogy is apt from the view that the drop was able to grow out an almost separate component which then tended to flip itself upwards away from the surface (see Figure 3-8) due to the relatively high flow from the inlet orifice in that direction trying to overcome the effects of gravity. This was abetted by the strong pinning of the drop at the rear contact line which kept the non-grown out component on the surface. Since there was no increasing gravitation impetus to move the drop (specifically the non-grown out component) down the incline, the rear contact angle would not shift much towards the limiting receding value. As the grown-out section continued to increase in volume, alternatively, it became more affected by gravity, which then caused it to flip back downwards towards the surface. On contact with the surface, the drop became governed by the three-phase mechanics once again. This meant that it would be able to reach a state just prior to detachment with the surface (see Figure 3-9) as previous. It is noteworthy that the two in one drop shape could be retained even up to this point. It is noteworthy that the liquid body was able to assume a condition momentarily, in between the flipping up and down phases, in which it was aligned almost perpendicular to the surface (see fourth sequence from left of Figure 3-8). This presents an interesting situation where an elongated liquid could be suspended in air can be formed through dynamic inflowing of fluid. Some similarities and contrasts can be made with erstwhile studies on ferrofluid magnetic drops under an applied external magnetic field [215].

Figure 3-8: Pictorial depiction of drop formation at a flowrate of 9 mL/min (from left to right) on the inclined SH surface under regime II
In considering this regime, it was not surprising that the drops were elongated more with increasing flowrate due to the increased pressure in the liquid body (see Figure 3-5). What was unexpected was the seeming ability of the drops to assume greater volumes (Figure 3-6). This conundrum however has a ready explanation. During the process where the drop developed the “wheelie” behaviour from which a grown-out section could form, it (the grown-out section) was not subject to the action of three phase mechanics. Consequently, it could grow more freely to increase the overall volume of the drop in the time that it remained airborne. Once the grown-out section came back to contact the surface, however, the operation of three phase mechanics recommenced. At that stage, however, the drop would have attained a marginally higher volume than what it could sustain if the wheelie action had not occurred. While the front contact line was able to assume the limiting advancing contact angle state quickly (which was invariant across regimes), the rear contact line nevertheless did not have sufficient time to achieve the receding contact angle limit before detachment. This is evidenced in the plot given in Figure 3-7. The result is an approximate loss of 30° in the degree of hysteresis.

In some instances, the extent of separation of the grown out section from the original drop was so great that it formed an arched shape when it made contact with the surface as it flipped back due to gravity (see Figure 3-10). For this brief moment, one observes the action of water being bent without the use of an external source such as static electricity (which is commonly demonstrated in high school physics classes) or lasers [216]. From there, the drop could then quickly undergo significant shape changes like a very elastic soft solid to minimize surface tension. Despite these strong shapes changes, there was no breakup of the drop due to the low energy of the surface. Evidence of drops behaving as soft solids had been previously
demonstrated in another context [217]. It should be noted, however, that jetting commenced at flowrates above 12 mL/min, which then incapacitated discrete drop generation. There were also some small capillary waves generated on the drop that assisted in the dissipation of energy.

In order to provide contrast to the SH surface, the liquid behaviour on a similarly inclined acrylic surface is analysed (Figure 3-11). With this surface, a flowrate of 1 mL/min caused a rivulet with a bulbous front to form (a) until it reached the edge of the surface (b), where it accumulated until a discharge of material (c). Such a behaviour, when done on highly wetting glass surfaces previously, showed an ability to generate a strong circulation flow all the way back to the source [218]. As the remnant liquid is stretched, energy minimization caused the rivulet to break up and form smaller daughter droplets (d) – (f). Such a flow regime will be unfavourable in creating discrete drops, highlighting the usefulness in using a SH surface.
That the ability of the drop to be retained on the inclined surface more due to the strong pinning of the rear contact line allows us to ruminate on the possibility of achieving smaller volume drops via synchronized physical interventions at the inlet orifice. It is important to note that attempts have been made to use capillary instabilities to pinch off discrete volumes via perpendicular flows in a closed channel T-junction [219]. Such an approach however does not benefit from the use of low energy surfaces that endow minimal sample loss and contamination, prevention of clogging and contact with another liquid, open access, as well as easy integration as offered here. It must be noted that a coaxial two flow approach based on focusing from a capillary had also been reported to harness the pinch off instability effect [220]. This will however require both liquid types to be present in the formed drop.

It has been claimed [221] that small viscous drops will roll down on the inclined plane when the Reynolds number Re<<1. This was confirmed with glycerol drops travelling down inclined superhydrophobic surfaces [222]. The essential physics is due to the viscosity of glycerol being relatively high (1000 times higher than that of water) which causes the initial motion in the edge to draw the inner fluid to roll down due to a strong internal shear force. Using particle velocimetry with water drops (10<Re<100), it has been found that the travel down the inclined superhydrophobic surface had both rolling and slipping modes [223]. In this case, rolling only occurs at the edges of the droplet. For the other regions in the drop, slipping dominates as the slip velocity is higher than the rolling velocity due to the relative lower
viscosity of water. This has secondary ramifications in our intended application. The detached drop, if just moving under a rolling mode, will have lower propensity to pick up any contaminant particles on the surfaces. However, it will also possibly be more likely to deviate from its intended direction of travel from a collision mechanism. Under a rolling and sliding mode, alternatively, contaminant particles (which are assumed to be very small) can be drawn in from the rolling edge to be subsumed into the drop by a flow (which also manifests sliding) and be drawn away with it. In either mode, it will be important to have the surface free of particle contaminants.

3.2.5 Conclusion

In summary, a simple approach is described to obtain discrete drops from continuous flow onto inclined superhydrophobic surfaces to facilitate device fabrication with features of low loss, low contamination, and open access for biochemical analysis. The finding that the rear pinning contact line was strongly influential in retention portends the development of interventions that can improve volume control. Despite strong pinning of the rear contact line, the low surface energy did not permit cornered drops to form and thus prevented daughter droplets, which cause sample loss, from developing. It was found that a high flowrate regime prior to jetting caused the liquid body to develop a grown-out section which was able to flip up first and then down while being airborne. During the time that this component was airborne, the drop was able to increase its volume without the action of the three-phase mechanics dictating the volume.

3.3 Formation of liquid body on an SH semi-spherical well

3.3.1 Background

While superhydrophobic (SH) surfaces are found in nature in the form of lotus and rice leaves and butterfly wings [224, 225], they can now be fabricated through a variety of methods [226, 227]. As they typically possess low surface energies, SH substrates are useful in applications that require self-cleaning. In microfluidics, devices that apply discrete independently controllable sample volumes are now increasingly preferred as they permit the microfluidic function to assume a set of basic repeated operations, whereby one unit of fluid can be moved over one unit of distance, thus facilitating the use of hierarchical and modular approaches that then make up flexible and scalable system architectures with high fault
tolerance capabilities. There is now substantial effort aimed at harnessing SH surfaces as part of the discrete microfluidic system for biochemical applications [228, 229, 230, 231, 232]. At the forepart of matters lies the need to create drops on superhydrophobic surfaces. It has recently been shown that a continuous flow of liquid delivered from a hole on a flat and inclined SH surface offers a means to create them rapidly [233]. On a perfectly horizontal surface, large sessile drops can be developed in this manner whereby the contact angle $\theta$ is typically in the equilibrium state. However, any small extent of surface tilt will cause the drop to possess advancing $\theta_a$ and receding $\theta_r$ angles. With increasing volume, the drop’s ability to accommodate contact angle hysteresis will eventually be exceeded by the gravitational force [234], resulting in its detachment and movement on the surface.

Semi-spherical wells or cavities offer the means for the confinement of various entities, ranging from plasma [235], to radiation [236, 237] to fluids [238]. The introduction of a semi-spherical well on the SH surface presents a possible capacity to support additional hysteresis of the drop through the edges (of the well). The capability of edges to provide additional constrain on liquids this has been harnessed in various applications [239, 240, 241]. The premise in the current context is that the contact line movement on the substrate can be restricted, thereby allowing the input flow to alter the shape of the sessile drop as it develops out of the well.

3.3.2 Experimental methodology

In the experiments, the SH substrates were flat copper plates that have been polished to remove all visible scratches using silicon carbide electro-coated water-proof abrasive paper (KMCA, WET/DRY S85 P600). A press was used to depress a 6 mm diameter steel bearing onto one plate surface to create a semi-spherical well of 3 mm diameter through indentation. After this, a small hole of 1mm diameter was drilled through the bottom of the well. A similar hole was drilled into the other plate without an indented well. The copper plates were first cleaned using absolute ethanol, allowed to air dry, and then immersed in a 24.75 mM aqueous solution of silver nitrate ($\text{AgNO}_3$) for 45 seconds to form micro and nano structures. After this, they were rinsed with copious amounts of distilled water followed by absolute ethanol before being allowed to air dry. Once dried, they were immersed in a 1 mM solution of the surface modifier $\text{CF}_3(\text{CF}_2)_7\text{CH}_2\text{CH}_2\text{SH}$ in absolute ethanol for 15 minutes. After removal, they were again rinsed with copious amounts of distilled water, followed by absolute ethanol, and then air dried.
The SH plates were located on a rotary stage and tilted to as close to 1° to the horizontal using a spirit level as aid. Distilled water was delivered via a flexible tube and Luer-lock adaptor located at the bottom of the plate through a programmable peristaltic pump (NE9000 New Era). Videos of the drops forming on the SH surface were recorded using a high-speed camera (Fastec Troubleshooter TS1000ME) at 500 frames per second. Each reading was made only when the surface (the well) was ensured to be dry.

### 3.3.3 Results and Discussion

Firstly, the situation of the liquid body formed on the surface through the semi-spherical well is studied. Distinct regimes were identified. At flowrates below 16 mL/min, the liquid body formed typical semi-spherical drops (see A in Figure 3-12) which grew before detaching and rolling off from the small incline. At flowrates of 16 mL/min and above, a similar semi-spherical drop shape can be seen at the base, but now with a liquid body appended above it (see B in Figure 3-13). This seems to suggest that a submerged jet mechanism [242, 243] was in operation throughout the drop forming process, except that at these higher flowrates the jet appeared interestingly to be able to “pierce” through the top of the semi-spherical drop without disrupting its form and growth very much. This is underlined by the plot of h (semi-spherical drop height) at the instant before drop displacement from the surface against flowrate which remained somewhat invariant. The values of h’ (extended liquid body height) were however expectedly higher with flowrate increase. In the case of w (width of drop in contact with the surface) the values were relatively constant up to 15 mL/min but fluctuated significantly thereafter with higher flowrates.
Figure 3-12: The delivery of fluid to a SH surface with an indented well 3 mm in diameter inclined at 1°
Following the trace of parameters with time prior to the first drop movement (Figure 3-13) will allow this to be better understood. For the case at 3 mL/min (Figure 3-13A), the advancing $\theta_a$ contact angles were almost consistently larger than the receding contact angles $\theta_r$ up to the point where the hysteresis could not contain the gravitational force due to increased volume filling. The values of $w$ appeared to be increasing rather uniformly with time, indicating a rather smooth progress of the contact line outwards from the well. In the case at 30 mL/min (Figure 3-13B), however, the values of $w$ could reduce at times even as it followed an overall increasing trend. At higher flowrates then, the jetting out of liquid to form the extended liquid body on the semi-spherical drop form imbued it with a degree of instability. This is evident in the higher fluctuations in contact angle distributions found, which naturally lead in turn to the unsteady growth in $w$. At this juncture, one must note that a somewhat more violent creation of this liquid form had been reported during the rebounding phase of a drop impact on hydrophobic surfaces [244, 245] or the perturbation of drops from surfaces using electrowetting mechanisms [246, 247] where a central jet could be similarly induced. It should be noted that similar jets (often called Worthington jets) can also be created when drops impact deep pools of liquid [248]. The manner of jetting in both cases can often lead to daughter drops forming...
due to the occurrence of pinching. In the current case, the delivery of the fluid through the forming sessile drop in the form of a submerged jet will cause it to experience reduced momentum at the exit such that pinching, and daughter droplet formation can be avoided.

For the second drop (and subsequent drops thereafter) it is found that the typical semi-spherical drop formation (without appendage above) could only be attained at flowrates below 14 mL/min. In this regime, the drop height that can be achieved before detachment was relatively constant as expected (see Figure 3-14). For 14 mL/min and above, a different regime was assumed. For instance, the sequence of images in time at 22 mL/min in Figure 3-14 showed a somewhat fusing of the two liquid bodies such that a resultant “egg” shape could develop. The location of more material at the top (than in the first drop) to move the centre of gravity higher meant that the liquid body was more unstable, leading to repeated frustrations in the process to maintain the egg form. This is borne out by the plot of maximum height against flowrate in Figure 3-14 which showed a random rather than increasing distribution with increasing flowrate. At flowrates above 26 mL/min the extent of the instability was so high that there was difficulty in ascertaining a definite maximum height value. This added instability with increasing flowrate (26 mL/min versus 15 mL/min) is collaborated by the graphs of the development of contact angles with time up to the point when the drop was displaced in Figure 3-15 which showed a clearly more heightened degree of fluctuation at 26 mL/min.
Figure 3-14: Plot of maximum drop height against flowrate SH surface with an indented well 3 mm in diameter inclined at 1°
Figure 3-15 Data of the advancing $\theta_a$ and receding $\theta_r$ contact angles, and width $w$ of the second drop formed on the surface with well at various times leading to its movement on the surface under flowrates of (A) 15 mL/min and (B) 26 mL/min

The difference between the first and subsequent drops formed is attributed to the manner of wetting in both cases. In the case of the first drop formed, the well was dry at the outset (Figure 3-16A). The small Bond number of $B_o = g\rho R^2 / \gamma \sim O (10^{-2})$ operational, where $g$ is gravitational acceleration, $\rho$ is liquid density, $R$ is the hole radius, $\gamma$ is surface tension of water, indicated that the action of gravity was relatively insignificant. On the other hand, the Weber number where $We = \rho u^2 R / \gamma \sim O (1)$ where $u$ is the speed, indicated that the inertial forces in action were comparable in magnitude to surface tension. As liquid was delivered into the dry well, it was likely that the contact line was able to move on the SH surface under low friction, such that when it reached the edge of the well, the built-up inertial forces could help it breach the added hysteresis there more readily. With the contact line located on the flat surface outside the well, the drop could then assume a typical sessile drop growth scheme. In the case of the subsequent drops, liquid remained in the well after any prior drop detachment (Figure 3-16B). Consequently, the contact line would not have the opportunity to gather as much inertial force as in the dry well to breach the edge hysteresis so easily.
This then allowed the liquid body, due to the continuous vertical delivery from the bottom, to harness the added hysteresis from the well edge to develop upwards. Hence, even after the contact line was able to breach the edge later, the already upward developed liquid body and the continuing delivery from underneath, could help it to sustain the elongated form that was relatively heavier at the top. The use of the added pinning mechanism at the well’s edge could also account for why the subsequent drops could begin to sustain the extended upward form at a lower flowrate (14 mL/min). The role of contact angle hysteresis here to aid shape formation corresponded with previous findings uncovered in the context of rebounding drops from surfaces [233]. Despite the apparent more fused physical appearance of the “egg” shape, it is maintained that two components (as in the more obvious first drop case) were still inherent. This is based on previous observations made on drop formation on a flat SH surface (without a well) under a higher degree of incline in which the liquid body was able to assume two visible separate components which at times could even perform a “wheelie”. When the SH surface was placed at a high incline angle, the direction of action of gravity caused asymmetric disruptions to the drop formation even as these components were visible. The use of a smaller incline and particularly with the heightened hysteresis offered by the edges of a well here, allowed the asymmetric action of gravity on the drop to be reduce and thus more clearly revealed the competition between the tendency to form a sessile drop and to create a jet.
The results obtained with the slightly inclined SH surface without an indented well provides good contrasts. That there was no difference between the first and subsequent drops developed was a clear basic difference. Regimes similar to the case with the first drop in the well were also uncovered, albeit with some differences (see Figure 3-17). While the drop and drop with appendage formations (Figure 3-17A and Figure 3-17B respectively) were observed, the transition occurred at a much lower flowrate of 7 mL/min. In addition, at flowrates above 13 mL/min there was high propensity for the delivered liquid to jet rather than to assume any drop form (Fig. Figure 3-17C). The plots of the values of h, h’, and w at the instant just before the drop displaced on the surface showed trends quite similar to the case of the well. However, the magnitudes were significantly reduced. These results highlight the reduced capacity to develop larger drops for delivery on the surface when no well was present. It also inferred that the well could serve as some sort of jetting diffuser to limit the flowrate needed to create the appendage above the drop. Another noteworthy observation was the lower extents of fluctuation in the contact angle and width dimensions in the progress towards drop detachment in the regime of growth above the drop when the well was absent (Figure 3-18). This was likely
due to the smaller growth heights developed which then translated to better stability of the liquid body.

![Figure 3-18: Data of the advancing $\theta_a$ and receding $\theta_r$ contact angles, and width $w$ of drop formed on the flat surface at various times leading to its movement on the surface under flowrates of (A) 3 mL/min and (B) 13 mL/min](image)

From a biochemical analysis application perspective, it is clear that the use of flowrates below 14 mL/min would ensure that drops with more consistent volumes could be delivered. While these drops are relatively large in size, they can still be transported over the SH surface, arguably with better efficiency than a series of smaller drops. At the point of usage, they can be partitioned into smaller drops using a steeper SH incline [233] or a SH “knife” [249]. It should be noted that microfluidics is essentially about operating with small liquid volumes rather than small device sizes. The findings related to how a liquid body is able to sustain extraneous growth under higher flowrates here, can serve as a platform from which investigations to better reconcile the tendency of liquids to assume drops or develop jets to be conducted.

### 3.3.4 Conclusion

The formation demeanour of first the subsequent drops on a superhydrophobic substrate (inclined at 1° to the horizontal) that had a semi-spherical well of 3 mm diameter, with liquid
at various flowrates delivered through a hole of 1mm diameter at the bottom of the well has been found to be different. With the first drop, the well was initially dry. Hence, the inertial force from liquid filling allowed the well’s edge hysteresis to be more readily breached. At flowrates of 16 mL/min and above, a jet could be created that appeared to be able to “pierce” through the top of the semi-spherical drop. Its form and growth did not appear to be strongly disrupted. With the subsequent drops, the well was liquid filled. By harnessing the extra hysteresis at well’s edge, an egg shape could be supported at flowrates of 14 mL/min and above. For the substrate without a well, no egg shape formation could be created. The transition in shape from drop to drop with growth above occurred at a lower flowrate of 7 mL/min and was limited to below 13 mL/min due to the propensity for jetting. Furthermore, the dimensions of the drops prior to detachment were all significantly lower. The findings here help to guide the selection of flowrate for consistent discrete volume delivery in biochemical analysis. It also serves as a platform to conduct investigations to better reconcile the tendency of liquids to assume drops or develop jets.

3.4 Uphill airflow transport of drops on SH incline

3.4.1 Background

The capacity to transport liquid continuously uphill on a SH surface in a closed channel or pipe is rather unremarkable when one considers that a simple pump is able to do this. Yet, the ability to transfer individual droplets uphill has proven to be of great interest to the scientific community. This is increasingly significant due to the proliferation of open and discrete microfluidic systems [250]. The basis to transport droplets uphill was theoretically mooted almost 40 years ago and then realized experimentally just over 20 years ago [251]. The modification of the wetting characteristics of surfaces in various ways provides an obvious approach to accomplish uphill droplet transport [251, 252, 253]: Nevertheless, the lateral vibration of surfaces [254, 255], heating surfaces to attain the Leidenfrost effect [256, 257], and illumination of laser beams on the substrate [258] or droplet [259] are among other methods that have also been advanced.

When droplets are placed on flat superhydrophobic (SH) surfaces, they assume an almost spherical shape due to the dominant Cassie wetting state. These surfaces can now be fabricated using various ways [260]. They generally offer ultra-low friction which can present containment problems in applications unless suitable constraints are used [261, 262]. Yet, the de-facto merit of using such surfaces lies with the low levels of sample loss due to the highly
non-wetting characteristic offered, which makes it attractive for biochemical applications [261, 262, 263]. It has been recently shown that when liquid is delivered continuously through an orifice to a SH incline, the increasing weight of the drop will cause the rear contact angle to reduce to the extent where the drop eventually pinches off with the contact line to traverse over the orifice. On making contact with the solid, however, a predominant Cassie wetting state is restored which will quickly allow the drop to detach and travel down the surface. This presents a viable means to rapidly generate droplets as opposed to other methods which can arguably provide more precise volumetric control [262]. In this work, the use of airflow to engender the uphill transport of droplets forming from water delivered continuously through an orifice onto the inclined SH surface, was reported.

### 3.4.2 Experimental methodology

A T-shaped setup was fabricated (see Figure 3-19) to conduct the experiments. As air was delivered along a straight passage from one opening to another diametrically opposite opening, it created a low-pressure region at the intersection with the orthogonal passage, allowing air to be drawn in through the third opening. In the experiments, the airflow delivered was unchanged, such that the air velocity at the point of drop formation, measured using a pitot tube and digital manometer (Digitron, 2002), was kept at 24.9 m/s. The SH substrate was a copper plate with a small hole of 1mm diameter drilled through, polished earlier to remove scratches using silicon carbide electro-coated water-proof abrasive paper (KMCA, WET/DRY S85 P600). Prior to use, it was first cleaned using absolute ethanol, allowed to air dry, and then immersed in a 24.75 mM aqueous solution of silver nitrate (AgNO₃) for 1 minute to form the micro and nano structures. After this, it was rinsed with copious amounts of distilled water followed by absolute ethanol before being allowed to air dry. Once dried, it was immersed in a 1 mM solution of the surface modifier CF₃(CF₂)₇CH₂CH₂SH in absolute ethanol (ethanol with low water content) for 5 minutes. After removal, it was again rinsed with copious amounts of distilled water, followed by absolute ethanol, and then air dried. Distilled water was delivered via a flexible tube and adaptor located at the bottom of the plate through a programmable peristaltic pump (NE9000 New Era). The liquid flowrate was kept constant at 0.1 mL/s to avoid the two-body separation regime observed previously. Videos of the drops forming on the SH surface were recorded using a high-speed camera (Fastec) at 250 frames per second.
3.4.3 Results and Discussion

Firstly, the case of drop formation and transport in the absence of airflow is considered (see Figure 3-20 (a)-(c)). Due to continual liquid filling through the orifice, a liquid bridge first develops, from which it gradually constricts before rupturing. This makes it different from the case of tilting the surface for a drop without infilling where the advancing and receding contact angle hysteresis dictates its movement from the surface. Based on this, it is possible to assume that a constant adhesion force $F_{adh}$ exists for the liquid drop, and its ability to be just detached from the surface is dictated by the balance of forces from gravity at equilibrium in which

$$V = \frac{F_{adh}}{\rho_w g \sin \theta} = k_1 \frac{1}{\sin \theta}$$

Eq. 3.3.3.1

where $V$ = volume, $\rho_w$ = the density of water, $g$ = gravitational acceleration, $\theta$ = angle of inclination. The experimental trend of $V$ against $\theta$ is evident in Figure 3-21 (scatter data), wherein a best fit process (solid line) yielded $k_1 = 0.018 \times 10^{-3}$ m$^3$. Based on this, $F_{adh} = 1.76 \times 10^{-4}$ N. This value is much larger than the adhesion forces of water drops on the same type SH surface previously found $\sim O(10^{-9})$ N [209] but is reasonable due to a different mechanism of attachment here (i.e. through a liquid bridge).
Figure 3-20: Sequence of images showing the detachment and downhill travel of a drop as it forms on the superhydrophobic incline without airflow (a-c), as opposed to a detach and uphill travel with an airflow in the direction indicated by the arrow (d-f).

Figure 3-21: Plots of drop volume developed with various incline angles of the SH surface with and without the airflow.

If the case of drop formation and transport with airflow is considered (see Figure 3-20 (d)-(f)), a somewhat similar case of a liquid bridge developing first, from which it gradually constricts before rupturing, can be seen. In being able to cause the droplet to detach and move uphill, the drag force developed has to overcome both the gravitational force and adhesion force of the drop to the surface. The drag force scales according to the cross-sectional area $A$. 

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of the drop. By conveniently equating to a sphere, it is possible to deduce that \( A = 4.836V^{2/3} \).

If it is assumed that the adhesion force to be unchanged in magnitude from the case without airflow but acting in the opposite direction, the force balance equation can be stated as

\[
4.836 \left( \frac{1}{2} \rho_a u^2 C_d \right) V^\frac{2}{3} - \rho_w g V \sin \theta - F_{\text{adh}} = 0 \quad \text{Eq. 3.3.3.2}
\]

where \( \rho_a \) = the density of air, \( u \) = airflow speed, and \( C_d \) = drag coefficient. Eq. (2) can be rewritten, by grouping into constants, as

\[
k_2 V^\frac{2}{3} - k_3 V \sin \theta - k_4 = 0 \quad \text{Eq. 3.3.3.3}
\]

On inspecting Eq. (3), one finds that it is a multivariable calculus problem, in which an implicit function theorem will allow relations to be converted to functions of several real variables. Essentially, there may not be a single function whose graph is the entire relation, but there may exist a solution based on restrictions placed on the domain of the relation. If \( V > 0 \) and \( \theta > 0 \), a theoretical fitting solution using Eq. (3) with \( V \) scaled to mL from \( m^3 \), is obtained with \( k_2 = 0.23, k_3 = 1 \), and \( k_4 = 0.018 \). While there is an absence of close conformance (which is believed to be due to changes in \( C_d \) during the evolution of the droplet as a soft matter rather than a solid), there is a consistent point at \( \theta = 18^\circ \) in which any increase in \( \theta \) will not produce a solution in the domain. This is observed in the experiments. Based on these fitting values, one is able to determine that \( C_d = 0.012 \). Using the airflow speed, air density, air viscosity, and the characteristic length (diameter of droplet) at detachment applied during the experiments, the Reynolds number is found to be 1020. Based on this value, the \( C_d \) for a sphere is calculated to be 0.023 using known relationships \[264\]. That the experimental value is approximately half of the calculated value can be rationalized from the fact that the shape of the droplet (see Figure 3-20 (c)-(d)) is not fully spherical. It’s more aerodynamic shape then accounts for the reduction in the drag coefficient. An interesting observation here is that smaller volume drops can be created and collected uphill when airflow is introduced. This has usefulness in microfluidic applications.

Since the droplet detachment mechanism (both uphill and downhill) is based on the pinching off effect, the similarities and differences here with the case of pendant drops are examined. With pendant drops, the liquid bridge elongates into a thin diameter \( \sim O(10^{-4}) \) m before it necks off quickly \( \sim O(10^{-3}) \) s to rupture when low viscosity Newtonian fluids like water are used \[265\]. The experimental measurements with airflow here yielded a consistent time to rupture of \( 3.2 \times 10^{-2} \) s from a neck thickness of \( 1 \times 10^{-3} \) m, notwithstanding the range of inclination angles used (see Figure 3-22). This invariance confirms that the pinching off effect is capillary
driven as in the case of the pendant drop. However, the approximately one order of magnitude higher in terms of the neck thickness and time to rupture implies contribution of the adhesion force of the drop to the surface which stabilizes and thus prolongs the necking to rupture process.

Figure 3-22: Plots of the minimum thickness of liquid bridge’s neck region at different times prior to rupture for inclination angles of 4° and 16° to the horizontal when airflow was applied.

From an application perspective, the airflow approach here is useful because it allows rapid uphill transport of a very rapid stream of droplets. Since water was delivered at a flowrate of 0.1 mL/s and the average size of the droplets was 0.03 mL, this translated to the delivery of 33 droplets per second. Due to the low forces involved \( \sim O(10^{-4}) \) N, the approach should also produce lower sample losses through aerosolization, which is known to have significant negative implications if the sample were to contain pathogens such as bacteria [266].

### 3.4.4 Conclusion

In summary, the feasibility of uphill droplet transport using airflow on a superhydrophobic surface incline is shown. The trend of volume on detachment in relation to inclination angle could be described using analytical equations for the range of physical parameters used in the experiment. A pinching off behavior with necking prior to detachment was observed, in which a constant time to rupture of \( 3.2 \times 10^{-2} \) s from a neck thickness of \( 1 \times 10^{-3} \) m was found
regardless of the inclination angle.

3.5 Liquid body resonance while contacting an SH surface

3.5.1 Background

The stick-slip behavior of a free liquid body, or often referred to as a sessile drop, on solid surfaces remains an active area of research due to the desire to control wetting processes and the difficulties in using classical hydrodynamics to account for the fluid mechanics at the three-phase contact line [267]. In the case of mercury moving over glass, picosecond electrical discharges and a flash of light has been shown to accompany the slip following each stick event at the meniscus [268]. While such manifestations are not evident in an overwhelming majority of sessile drops moving on solid surfaces, recent experimental studies on the dynamics of a sessile drop on an oscillated substrate have shown that pinning at the contact line can lead to exciting effects such as the breakup of the drop [269], climbing motion over inclined substrates [254], and motion over physical [270] and thermal gradients [271]. Similarly, unexpected behaviors have also been uncovered from the application of impact forces [272]. This has led to interest on how to model the dynamical perturbations better [273], in particular on how to best incorporate the contribution of contact angle hysteresis in the formulations [274]. This is particularly evident when pendant drops are vibrated using a forcing function along the axis of symmetry of the drop, or commonly described to as the longitudinal mode, where the capacity to assume large contact angle variations are exhibited [275]. While not mentioned previously, this is likely the consequence of sharp edges on the solid surface, or a multitude of projections leading to increase in roughness, which is known to provide additional pinning [276, 277]. Without the aid of these edges, an overhanging sessile drop can easily detach from the solid surface especially when the excitation corresponds with one of the natural frequency modes [278]. It should be noted that while experiments have generally focused on setting drops to oscillate in the longitudinal mode, efforts have also been expended to allow them to vibrate in lateral [279] and rotational [280] modes.

When two solid surfaces sandwich a liquid body, it gives rise to formation of a liquid bridge. An advantage of liquid bridges lies with their relative immunity from detachment. While the quasi-static extension of the liquid bridge [281] has received arguably the highest degree of attention, there have been efforts expended the study dynamical aspects as well [282, 283].
When a liquid bridge, developed between equal diameter disks, was subjected to horizontal vibrations at frequencies ranging from 0.1 to 20 Hz, heightened response amplitudes at a certain frequency, indicative of resonance behavior were found [284], similar to the case of sessile drops [278]. A scheme comprising a liquid bridge, sandwiched between a hydrophilic and a moving superhydrophobic surface, was previously devised to measure the liquid adhesion force of the latter [209]. Superhydrophobic (SH) surfaces offer exciting ways to accomplish novel biochemical applications due to their strong non-wetting characteristics. In such applications, the integration of improved sensing schemes will be highly beneficial, albeit methods of fabrication and usage should be simple for wider usage. Despite the adhesion forces being in the nano-Newton region, stick-slip characteristics have been observed [209]. Here, investigation is conducted to assess whether these characteristics are able to sustain resonant behavior in the liquid body itself.

3.5.2 Experimental methodology

The main experimental setup is shown in Figure 3-23. It comprises a superhydrophobic drum rotated using a variable speed direct current motor (10 to 70 rpm). The drum was fabricated by fitting a 24 mm outer diameter copper tube over a cylinder. A modified method based on electroless galvanization [285] was used to render the copper surface superhydrophobic. It was first cleaned using absolute ethanol (>99.8%, Sigma-Aldrich), allowed to air dry, and then immersed in a 24.75 mM aqueous solution of silver nitrate (AgNO3), for 45 seconds to form micro and nano structures. After this, it was rinsed with copious amounts of distilled water followed by absolute ethanol before being allowed to air dry. Once dried, it was immersed in a 1 mM solution of the surface modifier F3(CF2)7CH2CH2SH (Sigma-Aldrich) in absolute ethanol for 15 minutes. After removal, it was again rinsed with at least 1 litre of distilled water, followed by immersion into absolute ethanol for another 45 seconds, and then air dried. The video sequences were recorded using a black and white high-speed camera (Fastec Troubleshooter) at a frame-rate of 500 frames per second. Backlighting was provided by a high brightness LED source in which a ground glass was used to diffuse the light. A manual pipette (Eppendorf) fitted with a plastic tip (0.68 mm) was used to apply milli-Q water drops on the superhydrophobic drum surface, forming a liquid bridge. The tip was held in place to allow the liquid body not to be displaced when the drum was rotated. The distance between the tip and surface, as well as centeredness of the tip to the apex of the drum was adjusted using a three axis optomechanical stage. In experiments apart from the use of water, glycerol-water mixtures ranging from 0 – 100% glycerol by volume, were prepared by adding
glycerol (Sigma Aldrich, G5516) to Milli-Q water. These solutions were prepared in a polypropylene centrifuge tubes and mixed to homogeneity using a vortex mixer (Proscitech EVM80). The mixtures were allowed to settle for 1 hour before use.

Figure 3-23: Depiction of primary experimental setup

In order to investigate the contribution of drive train noise in the motor, incremental rotary optical encoder (Sick, DFS60A-S4PM65536) was coupled to the setup (see Figure 3-24). The coupling process was done carefully to ensure minimal axial misalignment between the motor and encoder. A secondary experiment was then conducted to obtain angular displacement versus time traces as the motor was rotated at different speeds. In these experiments, the resolution was set at 200 pulses per revolution and recording was taken over 10 seconds from the start of revolution of the motor.

Figure 3-24: Depiction of secondary experimental setup
3.5.3 Results and Discussion

Figure 3-25 provides images of drops at various volumes (V) and distances located between the pipette tip and SH surface (h) when the drum was rotated. The condition in (a) and (b) can be generalized as condition I, where the liquid body exists almost like a sphere between the tip and surface. In the case of (c) (where V = 10µL, h = 2.45mm), generalized as condition II, the drop is strongly deformed following liquid dispensation such that the contact line breached the additional hysteresis offered by the edge of the tip. As the SH surface rotated to the left, the drop was brought in that direction such that the right side of the contact line could reattach to the edge of the tip there. The resultant effect was an offset in the position of the drop to one side relative to the tip. The degree of movement of the drop was taken from its location on the SH surface. Yet in another generalized condition III, the liquid body displaces and moves towards the side of the tip. From experiments conducted with a 10 µL liquid body, conditions of I, II, and III were established to occur at $2.73 \leq h < 2.45 \text{ mm}$, $2.45 \leq h < 2.15 \text{ mm}$, and $h \geq 2.15 \text{ mm}$ respectively. In order to provide better physical insight into this, it is possible to apply a dimensionless parameter $\Lambda = \frac{h}{V^{1/3}}$ for depiction. With this, the conditions of I, II, and III occur at $1.267 \leq \Lambda < 1.137$, $1.137 \leq \Lambda < 1.00$ and $\Lambda \geq 1.00$ respectively. The type II drop essentially describes a liquid body that has buckled [286]. Interestingly the type III drop is another state that the liquid body can assume beyond the critical buckling state, which due to motion provided by the rotating drum, is able to allow it to escape compression altogether.

When conducting image analysis, points A and B were determined on each image frame (Figure 3-25 (a)), from which the center, contact length (distance between A and B), and the advancing and receding contact angles ($\phi_a$ and $\phi_r$) could be established.

Figure 3-25: Typical images of drops recorded with respective volumes (V) and tip to substrate surface separations (h) of (a) 5 µL, 2.33 mm, (b) 10 µL, 2.73 mm, and (c) 10 µL, 2.45 mm, as the substrate was rotated
The movement of the liquid body’s centre on the substrate surface (between A and B), taken from the center of the stationary tip (between C and D) provided indication of its perturbation. After approximately 10 seconds in almost all cases, a relatively clean sinusoidal signal was consistently obtained (see Figure 3-26(a) of typical time and power spectrum distribution with a type I drop) which indicated the liquid body going into resonance. That the position of the liquid body was altering close to the zero spatial position (Figure 3-26 (b)) indicates the axis-symmetric nature of the liquid body notwithstanding the rotation of the surface. Figure 3-27(a) provides distributions of advancing and receding contact angles corresponding to the dataset in Figure 3-26 (a). It can be seen that rather low hysteresis is accommodated. The stochastic fluctuations impute the presence of stick-slip processes occurring throughout which although previously measured to be in the nano-Newton range [209], provide sufficient perturbation force to drive the liquid body towards resonance. Despite these small forces, it is important to note that it is not the instantaneous but rather the cumulative contribution of the perturbations that will drive the liquid body towards resonance. This is not surprising as the capacity of small random perturbations to significantly affect dynamical systems had been reported previously [287]. There is of course a possibility that the random perturbations may be caused by the drive train noise of the motor used. This is addressed later when discussing the results from the secondary experiment.

![Figure 3-26](image)

**Figure 3-26**: A typical distribution of (a) position of the centre of a liquid body with a type I drop \((V = 5 \, \mu L \text{ and } h = 2.33 \, \text{mm})\) in contact with the rotating (at 10 RPM) SH surface, the power spectrum of the distribution (b), another typical distribution of (c) a type II drop \((V = 10 \, \mu L \text{ and } h = 2.45 \, \text{mm})\) in contact with the SH surface at 10 RPM the power spectrum of the distribution (d),
Figure 3-27 Plots of the advancing and receding contact angles as well as contact length of a liquid body with (a) a type I and (b) a type II drop ($V = 10 \, \mu L$ and $h = 2.45 \, mm$)

Figure 3-28: Distributions of the average natural frequency for type I, II and III drops

The distribution of contact lengths of liquid with the surface was roughly constant at 0.8 mm, in which the fluctuations can again be attributed to stick-slip processes. Similar trends were observed with another type I drop ($V = 10 \, \mu L$, $h = 2.73 \, mm$). In tracing the frequency corresponding to the peak in the power spectrum over a range of rotating speeds of the drum.
for both type I drops (Figure 3-28), it can be seen that the values are relatively consistent (15 Hz for the former and 11 Hz for the latter) with relatively low spreads encountered. It is be noted that the frequency (vertical axis) and speed (horizontal axis) in Figure 3-38 represent the natural frequency of vibrating droplet and the rotation speed of the drum. This result confirms that the liquid bodies were driven to resonance rather than by any other effect (e.g. eccentricity of the rotating surface, heightened pinning due to imperfections at specific locations on the surface). In a surface-tension-controlled vibration mode, the natural angular frequency $\omega$ is obtained by balancing the pressure due to inertial ($\sim \rho V^2/3 \omega^2$) and capillary force ($\sim \sigma/\sqrt[3]{V}$), where $\rho = \text{density}$, $\sigma = \text{surface tension}$, $V = \text{drop volume}$, such that $\omega \sim (\sigma/\rho V)^{1/2}$. The plot shown in Figure 3-29 (a) showed the expected inverse frequency dependence on volumes of water from 5 to 10 µL, while the use of $V^{-1/2}$ as abscissa in Figure 3-29 (b) indicated a good degree of scaling to this mode within the volume range used. This allowed us, as a first step, to eliminate the possibility of the oscillations being of a rotational mode which is governed by balancing the centripetal force ($\sim \rho V^4/3 \omega^2$) with the gravitational force ($\sim \sigma V g$), where $g$ is the gravitational acceleration, such that $\omega \sim (g/\sqrt[3]{V})^{1/2}$. Video recordings at different angles made allowed us to confirm that the rotational vibration mode was not operational. In an applied situation, the liquid body will first be dispensed onto the tip, after which the drum will be raised towards it to form the liquid bridge. From that, the drum will be rotated to bring the liquid body into resonance. From a series of tests conducted, the maximum volume of water that can be dispensed on the tip was 12 µL, beyond which it will simply detach, thus limiting the use of larger volumes to interrogate the range of scaling. The use of glycerol-water mixture liquids has also been attempted in order to extend this range in terms of density (densities of glycerol and water are 1.26 and 1 g/cm$^3$ respectively). However, the results have not been consistent, due mainly to the inability of the surface to exhibit superoleophobicity as well. The results with contact angle measurements presented in Figure 3-30 attest to this.
Figure 3-29: Distributions of (a) the average natural frequency determined using varied volumes ($V = 5, 6, 7, 8, 9, 10 \mu L$) based on 5 measurements each at a rotating speed of 10 rpm with the SH drum. The abscissa of (b) is plotted using $V^{-1/2}$ in order to interrogate a surface-tension-controlled vibration mode.

Figure 3-30: Equilibrium contact angles measured using a 10 µL volume glycerol-water mixture sessile drop on the superhydrophobic surface with varying percentages (by volume) of glycerol.

The results in using a type II drop ($V = 10 \mu L, h = 2.45 \text{ mm}$) were rather unexpected. While the liquid body went into resonance after 10 seconds in almost all cases as previously, the positional distribution (Figure 3-26 (c)) was centered negatively due to the liquid body being displaced to the left by the movement of drum relative to the tip (Figure 3-25 (c)).
Although a sinusoidal signal indicative of resonance was observed, it was amplitude modulated over a lower sinusoidal signal that served as carrier. By comparing with the results at different speeds, it was found that the lower sinusoidal perturbation corresponded closely with the angular speed of the rotating drum. On inspecting the power spectrum (Figure 3-26 (d)), a spread out rather than a sharp peak (at other speeds) was observed, rendering high uncertainty in the determination of the oscillating liquid body’s natural frequency (Figure 3-28). On returning to the positional-time plot (Figure 3-26 (c)), it could be seen that there were changes in the resonance frequency with time as well as higher noise. The former is likely related to contact length variations of the liquid body on the surface (Figure 3-27 (b)) which also corresponded to the angular speed of the drum. The dependencies on the angular speed of the drum indicate the presence of a small degree of eccentricity in the drum which caused the liquid body to be able to increase and decrease its contact with the solid surface with time. In a nutshell, the drum eccentricity resulted in the ability of the resonant related sinusoidal perturbations to be amplitude modulated onto the sinusoidal distribution that was associated with it. As a secondary effect, the drum eccentricity caused variation in the contact length on the surface with time, which would allow the resonant related sinusoidal distributions to undergo frequency drifting.

Figure 3-31: Plot of the surface oblate spheroid / sphere ratio for normalized values of c/r, where the volumes of the oblate spheroid and sphere are equalized

The reason that did this not occur in the cases of the type I drops (V = 5 µL, h = 2.33 mm, and V = 10 µL, h = 2.73 mm) is that in the first instance, the surface energy of a drop is given by (\(=\sigma S\)) where S is the surface area. Since the type II liquid body is approximately an
oblate spheroid (Figure 3-25 (c)), the ratio of its surface area over a sphere is close to its surface energy ratio over a sphere. The surface area and volume of an oblate spheroid (see inset of Figure 3-31) can be respectively found using

\[
S_{\text{oblate}} = 2\pi a^2 \left(1 + \frac{1 - e^2}{e} \tanh^{-1} e\right) \quad \text{(Eq. 3.5.3.1)}
\]

\[
V_{\text{oblate}} = \frac{4}{3} \pi a^2 c \quad \text{(Eq. 3.5.3.2)}
\]

In this, \(e^2 = 1 - c^2/a^2\), with \(c\) = polar radius, \(a\) = equatorial radius. By equating the volume of the oblate spheroid to a sphere with radius \(r\), it is possible to determine \(S_{\text{oblate}} / S_{\text{sphere}}\) using a normalized parameter \(c/r\) which indicates the degree of deformation in the oblate spheroid. This distribution (Figure 3-30) indicates that the oblate spheroid has an intrinsically higher amount of surface energy than the sphere. The larger the extent of deviation from the spherical shape (i.e. the smaller \(c/r\) is), the higher this surface energy will be. The work done \(\delta w\) associated with the movement of the three phase contact line can be related to the respective liquid-vapor, solid-liquid, and solid-vapor surface tensions \(\gamma, \gamma_{SL}, \gamma_{SV}\); liquid-vapor, solid-liquid, and solid-vapor area changes \(dA_{LV}, dA_{SL}, dA_{SV}\); Laplace pressure change \(\Delta P\), and volume change \(dV\) via

\[
\delta w = \gamma dA_{LV} + \gamma_{SL} dA_{SL} + \gamma_{SV} dA_{SV} + \Delta P dV \quad \text{(Eq. 3.5.3.3)}
\]

If the \(1^{\text{st}}\) and \(4^{\text{th}}\) terms on the right of Eq. (2) are taken to be zero (i.e. through \(dV = dA_{LV} = 0\)), and apply a simple assumption of incompressibility of both fluids, then \(dA_{SV} = -dA_{SL}\) and simplifies the \(2^{\text{nd}}\) and \(3^{\text{rd}}\) terms on the right to \(dA_{SL} (\gamma_{SL} - \gamma_{SV})\). Due to the manner in which the drop resides relative to the tip, larger vertical forces will be used at the tip to push the drop against the superhydrophobic surface. Consequently, when the drop oscillates, it may not tap into the totality of the energy indicated. Despite this, the oblate spheroid should intrinsically have higher surface energy and is less stable, hence it is able to create higher fluctuations in \(\delta w\) during the stick-slip process, causing greater corresponding variations in \(dA_{SL}\) than in a more energetically stable sphere-like liquid body. This in turn caused greater variation in the contact length of the liquid body as it attempts to stay on the substrate surface.
Figure 3-32: Image of (a) 5 µL liquid body with the tip located 2 mm to the right of the apex of the drum and at 2.42 mm to the surface. In using a 10-rpm rotation rate, it is possible at times to observe (b) an unstable behavior in which resonance is interspersed in time

The amplitude modulation effect may also be attributed to the location of A and B relative to the apex of the drum. To uncover if this was the case, the tip was moved to be slightly eccentric to the right (2 mm) of the apex of the drum, such that both A and B were on the right of the apex. In using a 5 µL drop separated at a sufficient distance (2.42 mm) from the drum surface (see Figure 3-32 (a)), we tracked its position with time after the drum was rotated for 10 seconds. The manner of perturbation of the liquid body in this way is not stable. In many instances the liquid body would simply detach from the tip altogether. In some cases, a quasi-stable like condition was observed, in which a typical position versus time trace is given in Figure 3-32 (b). They corresponded to the situation where the liquid body, because it was inherently asymmetrical at the outset, had imbalanced stick-slip forces acting at points A and B. Since there was lack of a strong enough vertical force pushing the liquid body downward from the tip onto the surface to counteract this imbalance, this resulted in the interspersed rather than constant resonant behavior with time. A parallel analogy lies with a similar need for a stylus reading from a rotating vinyl record to be sufficient weighed down, or else it would easily skip between the grooved tracks. There was a skew of the center of vibration towards the right (relative to the tip), which is to be expected due to an overall tilt of the drum surface downward from the horizontal on the right hand side. Due to the inherent asymmetrical position of the liquid body relative to the drum surface, it would be reasonable to assume that some modulation due to eccentricity ought to be observed during rotation. That this was not evident indicated the need for either a strong enough downward force from the tip, or for a shape that was energetically less stable for it to manifest. Experiments were also conducted in which the
offset position of the tip was moved to the left of the apex of the drum. In this case, the drop would consistently be detached from the tip.

In returning to the original condition, some added physical interpretations can also be gleaned by recognizing the type II drop as a liquid body that is compressed to the extent of buckling [286]. In solid mechanics, it is well known that buckled structures are pliable enough to be able to exhibit “negative stiffness” wherein the reaction force acts in the same direction as the displacement [288]. Clearly, such structures must rely on constraints to keep them in place. For the type II drop, this constraint is offered by the pinning at the tip and augmented by compression force at the region of liquid between the tip and drum (rather than on the entire drop), which then allows the liquid body to respond to two perturbations that are orthogonal to each other; one from the stick-slip events that drive it to resonance, and the other from the eccentricity of the drum which helps to create the carrier modulation.

![Figure 3-33: Plots of angular displacement against time measured using an optical rotary encoder coupled to the motor as it was rotated from rest to around 10 seconds](image)

If drop were to be placed on one side of the tip, it should remain fairly spherical without a force to press it onto the surface. Through further investigation, it was found that the drop would tend either to move up this side of the tip or be displaced from the tip altogether. Hence, no repeatable data can be achieved using this approach. This behaviour of the liquid body also showed that its stability on the surface involved harnessing the extra pinning provided by the edges of the tip as well as sufficient compressive force between the tip and drum.
The possible contribution of drive train noise from the motor is then examined. Using the readings from an optical rotary encoder (from the setup in Figure 3-24), it was possible to obtain traces of angular displacement against time as the motor is rotated from rest. The plots made of these at various rotational speeds of the motor show highly linear trends (Figure 3-33), where linear regression computations revealed values of $0.999 < R^2 < 1$ in all cases. These highly linear trends indicate that acceleration (which is the rate of change of velocity) was minimal. In order to examine the contribution of drive train noise from the motor, it will be necessary to calculate the variations in angular displacement $\theta_{\text{var}}(t)$ in time $t$ taken from the distributions in Figure 3-32 using

$$\theta_{\text{var}}(t) = \theta(t) - \theta_{\text{tim}}(t)$$  \hspace{1cm} (Eq. 3.5.3.4)

where $\theta(t)$ and $\theta_{\text{tim}}(t)$ are the respective measured and estimated (from linear regression) angular displacements with time. The plots of these variations (see Figure 3-34) indicate that the drive train noise from the motor was periodic. In addition, there was no gradual progression through a series of frequencies before reaching this frequency. The respective frequencies in the angular displacement variation are dependent on the speed of the motor, as shown in Figure 3-34. From this it was possible to deduce that a forcing function from the drive train was not responsible for driving the system to resonance [284]. In the main experiments, resonance can be obtained notwithstanding the motor speed used.

![Figure 3-34: Plots of angular displacement variations from the linear trend against time at three different rotation speeds (23, 45, and 68 rpm) of the motor](image)
Some insights, however, may be drawn from the marked difference in the ability of the liquid body to maintain resonance when it was located at the apex of the drum (see Figure 3-26 (a)) as opposed to when it was placed at an offset position to the right of it (see Figure 3-32 (b)). It highlights the importance of the stick-slip processes, wherein the forces acting at points A and B need also to be somewhat balanced for the resonant behaviour to be stably sustained with time.

At this juncture, it must be noted that the ability to achieve resonance has been reported for drops rolling down a surface with asperities lithographically produced using fibrils having well-defined lengths, cross-sections and spacing [289]. A drop that travels down an incline does not experience constant velocity but rather acceleration. Hence, there will be significant inertial force ascribed to the liquid’s body motion. Consequently, while the stick slip events contributed to the drop attaining self-excitation, the inertial influences can play a major role and will tend to work against the attainment of steady resonance unless the natural vibration modes interact in a synergetic way that a critical speed is achieved. Another prior work has also found elements of self-excitation when attempting to establish the adhesion forces of superhydrophobic surfaces [209]. However, there was a restoring force in action due to the deflection of the cantilever and as such inertial forces may have also contributed strongly to the ability of the liquid body to self-excite apart from the stick slip events. The approach here has limited inertial effects, and while it does not offer total immunity from noise, it allows a clearer manifestation of the liquid body resonance. From an application perspective, drops moving down an incline tend also to offer fewer opportunities of being harnessed for processes.

A promising possible application for the results here in microfluidic based photonic sensing is considered. Whispering-gallery waves can be made to manifest through the passage of light on various shapes such as spheres [290]. The effect with spherical droplets has been harnessed for lasing [291] and sensing [292] applications. Since the light waves are almost perfectly guided round by optical total internal reflection, Q factors in excess of $O (10^{10})$ are possible. In terms of sensing, the passage of light around the resonator droplet along the air-liquid interface offers interesting vistas, such as in the investigation of the adsorption of proteins at the liquid-air interface [293]. A fluid–fluid interface has advantages in such studies in that area can be precisely measured, allowing parameters such as area per molecule to be monitored, and impurities can be readily detected and removed, unlike the situation with solid interfaces. Provided that there is no energy barrier to adsorption, all surface-active molecules that are near the interface can be used for the process. The rate of adsorption is given by the
rate of diffusion of molecules from the bulk solution to the sublayer. Vibrations have been known to give rise to density gradients in liquids [294], from which convection can then cause changes to the diffusion rates of molecules present in the liquid sublayer. This affords a capacity to alter the adsorption kinetics in a controlled fashion whilst permitting accurate measurements to be made. Quite naturally, optical sensing can only be done when the oscillations have died down, albeit diffusion should remain in a heightened state for longer. In returning to the plot in Figure 3-29, the slope has been found to be $0.97 \times 10^{-3} m^{3/2} s^{-1}$ (32.5 $\mu L^{1/2}s^{-1}$) which is of the same order of magnitude as $(\sigma/\rho)^{1/2} = 10^{-3} m^{3/2} s^{-1}$ for water ($\sigma = 0.001 Nm^{-1}$, $\rho = 1000 kgm^{-3}$). Hence, if some means can be devised to create superoleophobic drum-like surfaces as used here, there is potential to apply the sensing approach to liquids such as glycerol-based mixtures that are widely used in biochemical applications [295].

It is important to note that when a drop is allowed to hang freely, its lack of physical insulation makes it prone to producing a noise floor signal that is mainly attributed to ambient disturbances during sensing [290]. In addition, a step-like excitation such as the arrival of an aerosol particle can cause unwanted sudden discrete signal shifts. If the droplet makes contact with a SH surface, it is plausible that these deleterious effects can be minimized via improved insulation. It is important to note that when the liquid contacts the SH surface, it does so in a predominant Cassie rather than Wenzel wetting mode. Hence, the liquid does not invade into the air-filled micro and nano cavities [296] where sample loss can occur. While it is possible to restore a predominant Wenzel to Cassie wetting mode on a superhydrophobic surface, it will typically exact some form of energy input [297, 298]. The use of a rotating (instead of vibrating) function is also more cost-effective, easier, and more silent in operation. Due to the heightened perturbations in liquid bodies when it adopts an approximate oblate spheroid form, it will be tempting to assume that enhanced diffusion can be achieved. Nevertheless, oblate spheroid droplets will likely not function well as spheroid droplets as whispering-gallery-mode resonators.

### 3.5.4 Conclusion

In summary, it was found that liquid bodies sandwiched with tip to surface spacing in the range of $2.73 \leq h < 2.45$ mm for a volume of 10 $\mu$L between a stationary tip and rotating SH surface can exhibit resonance primarily through excitation using stick-slip events that were independent of the speed of the drum. The scaling in the frequency values in relation to volume...
obtained indicated that the mechanics was found to be due to a surface-tension-controlled vibration mode. This was corroborated using video observations at different orientations to eliminate the rotational controlled vibration mode. When the spacing imposed was in the range of $2.45 \leq h < 2.15$ mm imposed for a volume of $10$ µL, the contact length of the liquid body was found to vary with rotation of the SH drum as a consequence of the stick-slip events being able to generate higher energy fluctuations to vary the liquid-solid contact areas since the almost spheroid shape of the liquid body possessed intrinsically higher surface energies. This resulted in the natural frequency perturbations to be both frequency and amplitude modulated over a lower frequency carrier. For spacing with $h \geq 2.15$ mm, the liquid body tended to move towards the side of the tip. Experiments conducted with an optical rotary encoder indicated that drive train noise from the motor would not bring the liquid body into resonance through periodic forcing.

3.6 Linear stepper actuation driving drop resonance and modifying hysteresis

3.6.1 Background

Sessile and pendant liquid drops can be set into resonant vibration, and this has been known for some time [299, 300]. When driven in the longitudinal direction, various mode shapes for sessile drops have been observed [89, 301], leading to applications such as mixing and particle focusing [302]. Similar mode shapes have also been observed when the drop is driven in the lateral sense [279]. The need to provide a driving excitation to match the sessile drop’s resonant frequency necessitates the availability of precision equipment to do so. Yet, if stochastic excitation is introduced such that it falls within the range in which the resonant frequency resides in, it is possible for the system to be set into resonance. This is exemplified in recent exercises of energy harvesting using solid structures [303, 304]. In returning to the case with sessile drops, Chaudhury and Goohpattader demonstrated that drops rolling down a surface with asperities lithographically produced using fibrils with well-defined lengths, cross-sections and spacing could elicit resonant behaviour [289]. In previous section, it was shown that a more practical approach was achieved by having the liquid body on a stationary tip coming in contact with a rotating SH drum, such that resonance is maintained primarily from stochastic stick-slip events between the liquid and SH surface.
While drops can easily slide down surfaces due to the action of gravity, schemes such as substrate wetting transformation and air perturbation have been shown to achieve motion up inclines notwithstanding gravity [305, 251]. The ability to control the speed of transport of the drop, however, is not possible using these schemes. Yet, the capacity to attain controlled transport of a drop while it continues to exhibit resonant rocking behavior offers important advantages in biochemical analysis. This advantage promises to be heightened if the drop is able to do this, not only when it is moved along the horizontal plane, but also when translated up and down inclines, where gravitational effects are significant.

In this work, the scheme of transport of drops as they are attached on moving surfaces with high contact angles is advanced. This is achieved by creating a circular wetting region that is bounded by superhydrophobicity on the substrate so that the drops can be easily set into resonance. Concepts of Newtonian displacement, velocity, and acceleration are well understood in dynamical mechanics. When depicting a solid body that is set in motion under constant velocity, it is often assumed that acceleration is non-existent. In reality, however, it is highly unlikely for an actuator to eliminate acceleration. The manner of its appearance is not strictly random although it retains the capacity to drive objects at low frequency bandwidths and with net acceleration. Close attention in our studies will be paid to the different shape characteristics that the drops manifest when the substrate surface is made to move horizontally, uphill, and downhill.

### 3.6.2 Experimental methodology

The SH substrate was a copper plate polished to remove scratches using silicon carbide electro-coated water-proof abrasive paper (KMCA, WET/DRY S85 P600). Prior to use, it was first cleaned using absolute ethanol, allowed to air dry, and then immersed in a 24.75 mM aqueous solution of silver nitrate (AgNO₃) for 1 minute to form the micro and nano structures. After this, it was rinsed with copious amounts of distilled water followed by absolute ethanol before being allowed to air dry. Once dried, it was immersed in a 1 mM solution of the surface modifier CF₃(CF₂)₇CH₂CH₂SH in absolute ethanol (ethanol with low water content) for 5 minutes. An impression of 1 mm diameter created using a pipette tip on the surface. This was meant to create a circular region that is hydrophilic bounded by superhydrophobicity.

The contact angle of a sessile water drop on the superhydroophobic region of the substrate was measured using the Kruss DSA100S system. The mean value from 10 separate readings was ascertained to be 158° (σ = 0.80°). The surface microstructure of the substrate with
superhydrophobic and non-superhydrophobic regions was examined under vacuum using a scanning electron microscope (FEI, NovaNanoSEM 430).

In one set of experiments, 2 µL of water was dispensed on the substrate (where the circular impression was made). The substrate was placed on an adjustable incline and side images of the drop were made at various angles. The advancing and receding contact angles were then measured.

In another set of experiments, a linear stepper motorized actuator (Zaber) was used to translate the substrate with drop on it. The actuator and substrate were then placed on an adjustable incline. The speed of the actuator was programmed to operate at 4 mm/sec throughout. Videos of the drops forming on the SH surface were recorded using a microscope lens fitted to a high-speed camera (Fastec) at 500 frames per second with the substrate moving inclined upwards and downwards at angles up to 6°.

To characterize the linear stepper motorized actuator (Zaber), an inertial measurement unit (IMU) was attached to it and readings from it recorded as the actuator was made to travel at 4 mm/sec. To detect for any directional differences, measurements were made with the actuator moving forwards and backwards.

In order to compare results with surfaces that do not have any SH boundaries, alternative experiments were conducted using a cylindrical untreated copper rod with 1.3 mm diameter. This was then attached to the actuator with the speed again programmed to operate at 4 mm/sec. A similar volume of water (2 µL) was dispensed on one end of the rod. Videos of the drops were again recorded using a microscope lens fitted to a high-speed camera (Fastec) at 500 frames per second with the rod moving downwards at angles at 3°.

3.6.3 Results and Discussion

The image of the SH substrate with a circular impression made on it is shown in Figure 3-35. The SEM micrograph of the region that is SH clearly depicts hierarchical microscale and nanoscale structures that permit a predominant Cassie wetting characteristic. Within the circular impression, however, there is clear absence of these structures which then should permit a predominant Wenzel wetting state to develop. At the interface between these two regions, there is a distinct separation of the two structure types, indicating the means for a liquid drop that rests in the circular impression region to possess high apparent contact angle at the three-phase contact line.
Figure 3-35: Image of the superhydrophobic surface with a circular impression (top left), in which SEM micrographs recorded at different regions.

Figure 3-36 provides a typical side image of the drop superhydrophobic surface with a circular impression (inset). When tilted at various inclination angles (θ) to the horizontal, it showed typical advancing and receding values which is often referred to as contact angle hysteresis. It is clear that the drop is able to remain on the surface at higher inclination angles than on typical SH surfaces despite the contact angle being very high (> 130⁰). At low inclination angles (up to 6⁰) used in this investigation, the differences between the advancing and receding angles are relatively small.
The acceleration readings obtained from the IMU attached to the linear stepper motorized actuator (Figure 3-37(a)) affirm the thesis that acceleration components exist notwithstanding the signals sent to translate it at constant velocity. The distribution is clearly stochastic and sufficiently wideband. While its average amplitude constitutes a small fraction (0.3%) of the gravitational acceleration, it promises an ability to drive any drop residing on it to resonance. This is evident in the traces of points L and R (see inset image of Figure 3-36) against time (Figure 3-37) with the substrate (and actuator) both orientated horizontally. At this juncture, it is to be noted that that these displacements are taken relative to corresponding fixed points L’ and R’ on the substrate determined when the drop was at rest. To provide more sensitive depictions of the drop perturbations than contact angles, points L and R were monitored. Essentially, this result shows that it is not necessary to use an actuator driving laterally at a matching frequency in order to propel the drop to rock at resonance as is normally done [279]. An added advantage lies with tandem controlled transport of the drops without any direct physical contact.
Figure 3-37: The trace of (a) acceleration against time obtained using the IMU located on the linear actuator which shows fluctuating values that have a positive offset. With the substrate horizontal, the displacements (b) of L (relative to L’) and R (relative to R’), from the inset of Figure 3-36.
The actuation done at small inclines, both downwards and upwards, yielded interesting results. In the case of the former (Figure 3-38), the drop continued to undergo resonant rocking, even as it exhibited an increase in the degree of contact angle hysteresis (which is defined by the difference between the advancing and receding contact angles) with progress of the actuation, over the case when the drop was not actuated (Figure 3-36). These contact angle
trends can be deduced by the increase of L and R in the positive sense (down the incline) as well as the fact that the three phase contact points remaining unchanged. After a period of 3s, there is a tapering off in the increase, which indicates a limiting condition being attained.

Figure 3-39: Trace of the displacements in mm (vertical axis) of points L (relative to L’) and R (relative to R’) on the drop, based on the inset of Figure 3-36, with time (in ms) for the substrate translating upwards at inclination angles of 3° and 6° to the horizontal.

With the substrate translated up the incline (Figure 3-39) there is an initial rapid swing (within 100 ms) of both points L and R on the drop down the incline, followed by a relatively gradual restoration towards the initial positions, in the midst of resonant rocking. The values achieved after the initial step, are similar to that ultimately attained in the case of the substrate
moving down (Figure 3-38). This indicates the limiting contact angle hysteresis condition reached almost instantly. With each resonant rocking event, this condition is reversed in small steps such that the original contact angle hysteresis condition is restored with time.

In order to elucidate these observations, it is important to consider the workings of contact angle hysteresis more carefully, which is underpinned by the system seeking out a local minimum in the Gibbs free energy [306]. It is important to note that these angles are not necessarily stable, although the energy barriers to reach the free energy minimum are usually so large that they almost always manifest as such. Typically, static hysteresis allows a drop to be deposited in a state where it maintains a constant difference between the two angles, without relaxation or motion. Even when the drop moves slowly on a rough surface, static hysteresis will continue to dominate. However, when the drop translates at higher velocities or if the surface has low energy, dynamic hysteresis asserts itself by the increase and decrease of the advancing and receding contact angles respectively over that developed in the static hysteresis state.

Since the drops in this work do not move relative to the substrate, it will be tempting to assume that static hysteresis should solely be in operation. However, perturbations that give rise to drops rocking resonantly comprise three regions in the liquid body that is organized in accordance to their distances (nearest to furthest) from the substrate; the Stokes layer, sublinear velocity gradient, and free-surface regions [279]. At the sublinear velocity gradient region, the fluid responds readily to inertial forces and is thus responsible for the manifestation of drop rocking. Since this region connects with the Stokes layer region immediately below it as a continuum, any significant change in the former will naturally affect the latter. That the capillary length of water $= \sqrt{\gamma/\rho g} = 2.7 \text{ mm}$, where $\gamma$ = surface tension, $\rho$ = liquid density, and $g$ = gravitational acceleration, is larger than the dimension of the drop, implies only that its shape at static equilibrium is unaffected by gravity. It does not infer that inertial effects, acting in conjunction with gravity cannot alter its shape when dynamical perturbations are present.

Another contributing factor comes from the superhydrophobic region that bounds the drop on the substrate surface. If the liquid phase is able to breach significantly into it, the three phase contact points offer little pinning to inhibit movement of the contact line. Otherwise, it’s highly wetting resistant nature allows the drop to assume a much larger contact angle than it normally could on typical copper surfaces (~80°). Essentially, as the drop is dispensed on the
substrate when it is orientated horizontally, the contact angle is already advancing. The depiction of advancing and receding contact angles when the substrate is tilted is only a matter of nomenclature. It is to be reiterated from earlier that since the superhydrophobic regions contain hierarchical microscale and nanoscale structures, both micro and nano Cassie wetting states exist in which air (commonly referred to also as plastrons) are present [307]. Under the right conditions, the conversion of micro Cassie to Wenzel states is able to proceed relatively more readily, but not the transformation of nano Cassie to Wenzel states. Similarly, under the right conditions, the micro Cassie states can be restored through the unconverted nano Cassie states.

![Diagram](image)

Figure 3-40: Depiction of the underlying mechanics with the drop as the substrate is transported (a) horizontally, as well as (b) down and (c) up an inclined angle.

In the case of the substrate being translated horizontally to the left (Figure 3-40 (a)), the random acceleration components in the actuator ensure not only the ability for the drop to rock resonantly but also for an initial movement and thus flow within the drop to the right. This is akin to the situation of a passenger holding a full cup of coffee and having it spilled on himself when the car accelerates suddenly. Since the drop is located horizontally, it is in its lowest state energetically as the whole of the gravitational acceleration is used to keep it on the substrate. Coupled with the fact that the acceleration components are comparatively weaker (Figure 3-36 (a)), there is no impetus for the drop to undergo any overall shape change apart from the rocking resonant action in order to dissipate the mechanical energy supplied.

When the substrate is inclined at an angle to the horizontal, the energy state of the system is raised to some extent, since a fraction of the gravitational acceleration tends to move it down the incline. This is, however, resisted by the force balance at the three-phase contacts, which in turn results in the static hysteresis appearing. As the substrate is translated
downwards, there is again an initial movement and thus flow within the drop acting towards the right (Figure 3-40 (b)). Since the direction of gravity counters this, there is no immediate effect on the drop shape. With the progress of actuation, however, the acceleration components which act downwards, abetted by the action of gravity that reduces stability, allows the drop to seek lower energy states by increasing its contact angle hysteresis, even as the resonant rocking is occurring at the same time to dissipate the mechanical energy supplied. This is aided by the SH surface characteristics at the three-phase contact line, in which the micro-Cassie states there are progressively converted to Wenzel states. This permits the drop to “lean” more down the incline, leading to the increasing trend in values of L and R with time found in Figure 3-38. The hysteresis increasing effect however does not proceed indefinitely but stabilizes after a while (~ 3s). This can be accounted for by almost all the micro Cassie states now being converted to Wenzel states, in which the conversion of the nano Cassie to Wenzel states would incur energy that the system is unable to supply. Overall, the manner in which the hysteresis is changed can be described as a cyclical alternation between the dynamic and static modes.

When the substrate is translated upwards alternatively, the initial movement and thus flow within the drop acts towards the left (Figure 3-40 (c)). Since gravity now assists this effect, the drop accommodates this by moving towards the limiting contact angle hysteresis condition quickly (Figure 3-39) by converting all the initial micro-Cassie states into Wenzel states. It is noteworthy that during this process, there is no rocking action of the drop, which illustrates how the drop is able to adapt by using a faster energy adjustment mechanism. After this stage, the actuator acceleration components are now acting progressively up the inclination angle, and aided by the instability from gravity, allows the hysteresis to be shifted back gradually towards the original static condition with time. This process then entails the Wenzel states in the SH regions at the interface reversing progressively back to micro-Cassie states. This is possible since the nano Cassie states were not converted prior.

It should be noted that at higher inclination angles and with higher volumes of liquid, the advancing contact line will breach. When this happens, the behaviours mentioned will not be observed. Clearly, it is the right balance between the inertial and surface tension forces acting of the liquid body that allows for the mechanisms described to manifest.

It would be ideal to be able to provide direct observations that depict the micro Cassie to Wenzel wetting state transformations directly. This is however difficult in this work due to the stochastic hierarchical structures on the surface. Yet, because these structures are somewhat
fragile, they allow the non-SH circular region to be created by simple contact pressure. This then allows sharp region separations, as evidenced in the SEM micrographs of Figure 3-35. The transformations however can be inferred indirectly. The ability of the drop to exhibit high contact angles (even at equilibrium), is due to the presence of the bounding SH region. One other way in which such a behaviour is possible is by adopting sharp edges [308, 309]. Figure 3-41 presents results conducted using untreated copper rods (1.3 mm in diameter), with the same volume of water (2 µL) placed on them and translated downwards at 3° inclination to the horizontal. They reveal absence of any hysteresis modification trends. The lack of any liquid interaction with SH regions in this case indicates that the reversible microscale Cassie to Wenzel transitions are responsible for the hysteresis modification behaviour in the main results.

![Graph showing displacements](image)

Figure 3-41: Comparative experiments conducted by placing a drop on a cylindrical rod (see inset picture). The traces are of displacements in mm (vertical axis) of points L (relative to L’) and R (relative to R’) on the drop, with time (in ms) for the substrate translating downwards at an inclination angle of 3° to the horizontal

3.6.4 Conclusion

In summary, this work has shown that the random acceleration components from linear stepper actuator are able to drive a drop that has high contact angles on a substrate to rock resonantly. When translating the substrate upwards and downwards on inclines limited to 6° to
the horizontal, the 2 µL drop also exhibited hysteresis changing characteristics that are based on the reversible conversion between micro Cassie and Wenzel wetting states.

The characteristics of liquids, notably drops, on surfaces that are fully or partially superhydrophobic have been investigated. Some unexpected characteristics have been uncovered. These findings provided the platform for further work that needs to be conducted before they can have the stability needed to be useable in biochemical applications associated with UAV transportation.
Chapter 4 UAV Systems Characterisation

4.1 Background

The weather can affect flight performance of UAVs and thus affect flight characteristics like endurance, aircraft control and flight path. It remains desirable to physically test flight models and control strategies using actual UAVs in simulated weather conditions. A novel method to study the effect of gusts on small UAVs is presented in the following sections.

In a wind-tunnel, the speed of airflow can be varied but typically only in a gradual fashion. Yet, the stability performance of small UAVs, particularly when they are hovering, is affected more by gusts of wind. A gust is a sudden, brief increase in the speed of wind followed by a lull. One method to simulate gusts in a wind tunnel is to use arrays of oscillating vanes upstream of the wind tunnel test section [310]. This generates vertical (or lateral) velocity components according to the amplitude of the vane motion and the frequency of their operation. Alternative methods include having fixed aerofoils with oscillating vanes [311] and rotating cylinders with slots [312]. These however appear more suited to simulating aircrafts operating at high flight speeds. Another approach involves using banks of axial fans to test the response of small UAVs operating at relatively low airflow [313].

In this section, the design and operation of a gust simulator is described, that operates within a wind tunnel, where airflow conditions can be closely controlled. An important feature that it possesses is that no modifications to the wind tunnel itself are needed. The airflow performance of this simulator is evaluated using numerical models and the gust characteristics determined experimentally. The simulator is then used to test the operation of a mini blimp UAV based on data derived from an inertia measurement unit that is housed in it.

4.2 Gust Simulator Description

The gust simulator was designed and built to work within the test section of a wind tunnel. The cross section of the gust simulator was 1m x 1m. This was as an ideal size since most mini and small UAVs can fit within this simulator. The design of the gust simulator was modular. It can be scaled up or down in size based on the testing requirements for a UAV. A schematic depiction of the gust simulator can be seen in Figure 4-1(A). Hence, its dimensions...
were made smaller, although the extent of the reduction is marginal. The operation is based on the principle that when the vanes at the front of simulator are closed Figure 4-1(B), the space within it undergoes a lull flight condition. However, as soon as they are opened Figure 4-1(C), a gust condition is introduced, in which the duration can be controlled until the vanes are closed again. All sides of the chamber are closed except for its rear (downstream to the airflow) to ensure that no stagnation in the airspace occurs when the gust condition is at play.

The most convenient construction is for the vanes to be arranged orthogonal and in a straight row to oppose the flow when in the closed position. This, however, creates separation flows over the bluff body that results in high drag (drag coefficient ~ 2 if taken as a 2D case) as well a large stagnation flow region upstream of the vanes. The latter can present issues when the vanes are opened suddenly to simulate a gust. The simulator developed here (Figure 4-2) overcomes this problem by having the vanes organized in the form of a circular arc. This reduces the drag considerably (drag coefficient ~ 1.2 if taken as a 2D case) and reduces the stagnation region upstream of the vanes when they are closed.
The built simulator has dimensions of $L = 86$ cm, $H = 45$ cm, and $W = 53$ cm, with wood being the main material of construction. The favourable strength/weight and ease of fabrication characteristics of wood are seldom appreciated, despite it being shown to be a viable material to construct whole wind-tunnel units [314]. The opening and closing of the vanes is attained by actuating four servomotors (HITEC, HS-485HB) through the coordination of a micro-controller (Arduino Uno). Two of the closed sides and its top comprise transparent plexiglass plates to enable viewing, and LED lighting is incorporated to provide clearer images during recordings. The simulator is relatively light at 10.9 kg which facilitates the placement into and removal from the wind tunnel test section.

4.3 Numerical Modelling of Gust Simulator

The operation of the gust simulator is depicted using numerical modelling. Due to the high level of symmetry, a two-dimensional flow model was applied. The incompressible Navier–Stokes equations were used to describe the flow field. In two dimensions, the dimensionless Navier–Stokes equations take the following form:

$$\frac{\partial u}{\partial x} + \frac{\partial v}{\partial y} = 0 \quad \text{(Eq. 4.1.2.1)}$$
\[
\frac{\partial u}{\partial t} + u \frac{\partial u}{\partial x} + v \frac{\partial v}{\partial y} = -\frac{\partial p}{\partial x} + \frac{1}{Re} \left( \frac{\partial^2 u}{\partial x^2} + \frac{\partial^2 u}{\partial y^2} \right) \quad \text{(Eq. 4.1.2.2)}
\]

\[
\frac{\partial v}{\partial t} + u \frac{\partial v}{\partial x} + v \frac{\partial v}{\partial y} = -\frac{\partial p}{\partial x} + \frac{1}{Re} \left( \frac{\partial^2 v}{\partial x^2} + \frac{\partial^2 v}{\partial y^2} \right) \quad \text{(Eq. 4.1.2.3)}
\]

where \( u \) and \( v \) are velocity components along the \( x \) and \( y \) directions, \( p \) is the pressure, and \( Re \) is the Reynolds’ number. It is assumed that these equations are subject to no-slip and no-penetration for regions that are solid.

The salient parts of the simulator were reproduced digitally on a computer-aided design software (Solidworks, Dassault Systems). Based on this, a 2D section fluid flow analysis was conducted using a finite element software package (COMSOL). The structure of the finite element mesh used is as shown in Figure 4-3. The region of the flow fields with the largest temporal and spatial gradients are those around and directly in front of the vanes. The mesh is thus made more refined around these regions. To reduce the computational load, the mesh is less refined elsewhere where smaller temporal and spatial gradients are expected. In doing so, the largest element was 2 mm while the smallest was 0.02 mm. In using the mesh improvement facilities available in COMSOL, the curvature factor was kept at 0.2 to limit how big any mesh element can be used along a curved boundary. The maximum element growth rate was also maintained at 1.05 to limit the size difference of any two adjacent mesh elements. Additional work was carried out to increase and decrease the number of elements and the step sizes of the temporal integration. These were found not to influence the accuracy of the computed results. To further reduce the computational load, half the gust simulator was modelled to take advantage of symmetry.
4.4 Experimental methodology

In the first part of the experiment, the air flow speed characteristics of 7 speed settings on the gust simulator were tested in a closed-return wind tunnel. At each setting the wind speed was monitored 5 times to verify consistency. The test section of the wind tunnel used had a cross-section of 1 m x 1 m and a length of 5 m, in which the gust simulator was placed into and anchored down. At each speed setting, the air flow in the wind tunnel was allowed to run for at least 3 minutes in order to obtain consistent values at the test section. The air flow speed was monitored using a digital wind sensor (Modern Devices, LSM9DS0) driven by an Arduino Uno controller. The sensor works by heating an element to a constant temperature and then measuring the electrical power required to maintain the heated element at that temperature. This measured electrical input is then directly proportional to the square of the wind speed. The electronic outputs of the sensor were recorded in a laptop computer (Microsoft Surface Pro) for collection and processing.

In the second part of the experiment, a balloon UAV was introduced into the gust simulator. This was devised by attaching a balloon filled with helium to a diameter of 37 mm to a mini quadcopter (JRC Minidrone) which has a mass of 35 g as indicated in Figure 4-4. The quadcopter is powered by a 100 mAh lithium battery wherein flight can be controlled using a smartphone application (FreeFlight, Android or iOS). An inertial measurement unit (IMU) (LSM9DS0, ST Microelectronics) was attached to the lowest portion of the UAV, from which information of the yaw, pitch, and roll can be conveniently calculated. It is a system-in-package featuring a 3D digital linear acceleration sensor, a 3D digital angular rate sensor, and a 3D digital magnetic sensor. It has a linear acceleration full scale of ±2g/±4g/±6g/±8g/±16g, a
magnetic field full scale of ±2/±4/±8/±12 Gauss and an angular rate of ±245/±500/±2000 dps. The LSM9DS0 has an I2C serial bus interface that supports standard and fast mode data transmission (100 kHz and 400 kHz) and an SPI serial standard interface. It can be configured to generate interrupt signals on dedicated pins, and the thresholds as well as timing of interrupt generators are programmable. The IMU is driven by an Arduino Uno controller, and its electronic outputs were recorded in a laptop (Microsoft Surface Pro) for collection and processing. Additional mass was added to ensure that the downward weight was balanced by the upward buoyant force of the entire device.

Figure 4-4: The blimp UAV devised by attaching a helium balloon to a mini quadcopter
4.5 Results and Discussion

The mesh model used is depicted in Figure 4-3. It was kept at a high density overall (Figure 4-3(A)) in order to achieve high accuracy. It can also be seen that graded meshes were used to accommodate expected large temporal and spatial gradients around and directly in front of the vanes Figure 4-3(B). The numerical predictions of air flow patterns inside and outside the gust simulator during its operation are presented in Figure 4-5 when it is switched from an “off” to “on” state. At $t = 0$ s, it can be seen that flow moves around the gust simulator in the wind tunnel without any stagnation areas in front of the vanes. This thus limits the creation of separation and excessive drag forces on the simulator. The flow is also laminar throughout in which no vortices are produced. Vortex induced vibration is a known problem in wind swept structures [315]. There were regions of speed increases but limited to only 2 times the wind tunnel speed applied. At 0.3 s after switching, the streamlines inside the simulator were almost aligned to those in the wind tunnel. As expected, however, the speed magnitudes were slightly lower. At 1 s after switching, the alignment and speeds of the streamlines in the simulator and wind tunnel equalized due to full flow development. The simulator design was therefore able to switch relatively quickly from the “off” to “on” condition flow, which should then depict the condition of gust appropriately.
Figure 4-5: The air flow simulation results obtained at times of (A) 0 s, (B) 0.3 s, and (B) 1 s after the vanes were switched from close to open

Figure 4-6 presents the experimental air speed measurements with time attained in the gust simulator using two different example fan settings Figure 4-6 (A and B) on the wind tunnel. It is evident that when the vanes are switched “off” each time to prevent any air flowing through, the speed dropped to zero. The ability to achieve this allows for a quiescent environment to be created prior to and after a gust episode. When the vanes were switched to the “on” position a number of times to allow air through, similar speed profiles were attained. This suggested consistency, albeit it is necessary to quantitatively evaluate this further.
Figure 4-6: Measurements of air speed with time on the gust simulator using two different flow settings (A and B)

Based on the 5 readings made, box plots of the speed distributions (when the gust simulator is set to the “on” condition) were generated and presented in Figure 4-7. It can be seen that the mean speed attained each time was somewhat identical. The data spread was however higher in the case when the air speed was lowered. This can be attributed to typical non-uniformities in the spatial velocity profile when the overall velocities were lower. Such a phenomenon had been reported in the context of instrumentation calibration at low flow speeds previously [316]. It is however instructive to consider this in relation to the range of air speeds introduced. An apt metric for description is the coefficient (cv) of variation which defines the ratio of standard deviation (σ) over the mean (µ) of the flow speeds during the time when the gust simulator is kept in the “on” condition, whereby

$$cv = \frac{\sigma}{\mu}$$  \hspace{1cm} (Eq. 4.1.4.1)
Figure 4-7: Box plots of the speed distributions when the gust simulator is set to the “on” condition

Figure 4-8 presents a plot of this metric at various speeds. The overall values are low, indicating relatively uniform velocity profiles throughout. Nevertheless, there appeared to be a threshold of above 5 km/h in which the velocity profiles had appreciably lower spreads.
The performance of a mini blimp UAV that was located in the gust simulator is then considered. In particular, it will be important to obtain the response of the mini blimp UAV in terms of its yaw, pitch, and roll when air gusts are introduced. These parameters are derived based on measurements from an IMU placed below the buoyant balloon (see Figure 4-9). There are two phases to be considered, the first when the simulator was switched from an “off” to “on” condition, and secondly when it is switched from an “on” to “off” condition. For the former, it can be seen that at 1 km/h (Figure 4-10), the yaw, pitch, and roll measurements did not alter much from values at the original “off” state. This indicated that, for this phase at least, the blimp UAV could remain positionally stable under this gust speed. When a gust speed of 2.1 km/h was introduced instead, the yaw and pitch changed up to a magnitude of 10 degrees while the roll remained unaffected (Figure 4-11). The stability to roll is likely imbued by the manner in which flow was incoming towards and then symmetrically deviated around the device (as seen from direction of air flow). This then provided greater immunity towards any twisting about the axis of roll. Since the IMU was located below the balloon rather than at its center, the momentum developed in response to the incoming flow of air made the UAV susceptible to the torques that resulted in pitch changes. In addition, the streamlines could not be perfectly tangential to the input axis of the simulator in practice. As there were no implements such as rudders incorporated on the UAV to mitigate this, this then resulted in the yaw changes. These depictions are corroborated by the pitch and yaw changes exhibiting the same sign at all speeds between 1 km/h and 2.1 km/h (see Figure 4-9 and Figure 4-10).
Figure 4-9: The response of the mini blimp UAV in terms of yaw, pitch, and roll to the air flow based on measurements from an IMU located at O.

Figure 4-10: Distributions of yaw, pitch, and roll obtained from the IMU attached to the mini blimp UAV with time when the mean air speed is 1 km/h.
The interesting result, however, occurred at the phase when the gust simulator was switched from the “on” to “off” condition. Quite clearly, there were significant short-term changes to the yaw, pitch, and roll when this happened (see Figure 4-10 and Figure 4-11). A depiction with velocity equipotential lines, which are orthogonal to streamlines, allows for this to be better explained. When the simulator moves from an “off” to “on” condition, equipotential lines can be visualized to be supplied continually from the source towards the object. When interacting with the blimp UAV, this “arrival” of equipotential lines caused the somewhat controlled changes in the yaw, pitch, and roll as explained previously. In the case of the simulator moving from the “on” to “off” however, the supply of air was removed suddenly, creating a pressure loss at the rear even as the equipotential lines upfront were still present in the vicinity of the blimp UAV. This transient perturbation in the flow fields caused distortions in the velocity equipotential lines (which eventually would diminish to zero since flow has ceased), making them responsible for the sudden strong changes in the yaw, pitch, and roll. The extent of this perturbation should be proportional to the air flow speed (which was terminated abruptly), which is duly attested to in Figure 4-8 and Figure 4-9. That the oscillations died off after 15 seconds was a matter of concern. If, for instance, there were to be a series of gusts occurring over time, the blimp could be kept in an unstable state over a prolonged period. It is noteworthy that when perturbed with a stronger gust magnitude, the
blimp UAV also tended not to return to its original angular position even after the oscillations have ceased (see Figure 4-9). This lack of restoration could be a problematic should the blimp be used in conjunction with cameras for surveillance (since it would essentially be panned to a dissimilar angular location after each episode).

As it was not the onset of a gust that causes significant positional instability to the blimp UAV but rather when the gust ended suddenly, there are possible mitigation measures that are can be incorporated. If the duration of the gust is sufficiently long, or if wind sensors mounted on the device are able to provide measurements that are rapid enough, it may be possible to deploy control schemes to counteract the yaw, pitch, or roll changes during the “on” to “off” phase. Figure 4-8 and Figure 4-9 also indicated that the transient changes to yaw were more significant than to pitch and roll. It is conceivable that strategies such as the introduction of well-sized and well-placed rudders may be able to provide greater stability against this effect. The simulator thus provides an effective experimentation pathway to arrive at optimal architectures for this. In aerospace applications, the increase in load associated with any structural add-ons is an important factor that cannot be overlooked. It is also noteworthy that trajectory planning is generally needed to allow UAVs to navigate efficiently to different locations [317]. The simulator here will allow for these algorithms to be developed in tandem with the proper ability to adjust for the effect of gusts in the process. Finally, it should be noted that the simple simulator design portends the ability to be scaled up to fit into wind tunnels that have larger test areas. Furthermore, it is a cost-effective design. The materials and instrumentation involved needed to construct the simulator in this work amounted to less than US$200. The ability to run the simulator remotely via a smartphone device is also possible if an advanced but inexpensive controller kit recently developed is used (www.pfidocontrols.com).

4.6 Conclusion

A novel gust simulator design that be easily placed into and removed from a wind tunnel has been demonstrated here. The fabricated version was able to experimentally generate an effective quiescent condition when the “off” condition was applied, while consistent and constant air speeds could be created when the “on” condition was applied. This behaviour matched the results obtained from finite element models done prior to interrogate the flow fields. In testing a small blimp UAV, it was found that the “on” to “off” phase of a gust episode created more changes to the yaw, pitch, and roll than during the “off” to “on” phase. The extent
of yaw changes was also higher than pitch and roll. These results highlight the usefulness of the simulator in guiding the development of small UAVs that are more stabilized towards episodes of gusts in practice. It is recommended that similar experiment is conducted using multi-rotor UAVs and fixed-wing UAVs to get more insights on how different designs of a UAV respond to gusts.
Chapter 5 UAV Systems for Biochemical Applications

5.1 Background

In this chapter, innovative methods using UAV Systems for in-flight biochemical analysis are presented. There is understandable interest towards increasing use of point-of-care Testing (POCT) for biomarker measurements with associated advantages of reduced delays in diagnosis and treatment, minimal sample handling, ease-of-use with lower staff training burdens to maintain formal accreditation, as well as opportunity for broad applications both in the clinical and nonclinical setting. POCT offers to bypass many steps where pre-and post-analytical errors most commonly arise in centralized laboratory-based testing, which include appropriate specimen collection, handling, liquid transport and storage; instrument set up; testing; and result reporting. Although significant advances in POCT technologies in terms of improved test reliability and increasing spectrum of available POCT menus, there remains issues of relative cost burdens, analytical and clinical performances compared to laboratory-based testing that continue to require sustained efforts to address. Clearly, the role of laboratory testing remains a core pillar for disease management where rapid diagnosis, timely and appropriate intervention are central to quality healthcare.

The transport of liquid samples to the laboratory is strongly governed by the twin factors of timeliness (to ensure their viability) and cost [318, 319], where the use of autonomous schemes have clear advantages. In hospitals, pneumatic tubes have long been implemented in this vein, to the extent that they are now indispensable to the function of most healthcare support systems [320, 321]. More recently, the use of drones, or unmanned Aerial vehicles (UAV), has been explored for outdoor biological Specimen delivery [322]. While some instances of malfunctions have been highlighted as issues of concern [323], these glitches will likely be overcome through the use of more robust electronics technologies, thus enabling their potential use not only in remote venues but also in traffic congested urban environments, where route planning strategies can only offer limited improvements in timeliness of sample delivery. The timeliness aspect can be further augmented if some parts of the pre-processing of samples are done en-route during transport on a UAV.
It should be noted that some of the knowledge attained in Chapter 3 regarding liquid behaviour helped to guide the work in this chapter. Due to limitations of stability and isolation that have yet to be solved, the use of drops (which allow for very small volumes) could not yet be applied as originally planned.

5.2 Drone inflight mixing of biochemical samples

5.2.1 Background

In the first aspect of this chapter, the ability of this manoeuvre to helpfully mix samples inflight is investigated. Appropriate mixing of samples is arguably the most common sample handling step which is key to obtaining quality specimens for laboratory testing. In the case of blood collection into receptacles, proper mixing for homogenous dispersion of silica particles, separator gels, clot activators or anti-coagulants is vital for optimal downstream separation of serum or plasma. Controlling the level of agitation applied during mixing is also important for minimizing haemolysis of red blood cells which could render the sample unsuitable for testing. Automated methods for mixing are generally based on sustained and controlled agitation for specific periods of time.

Novel schemes have been devised to mix volumes of liquid samples in lab-on-chip devices [324], capillaries [325], and liquid bridges [326], and drops [327] in the micro-litre range in the laboratory. Samples that are transported on a UAV, to the laboratory are mostly in the millilitre volume range in order to provide margins for repeat testing procedures [328] which occasionally can be due to process [329], assay [330] or substrate [331] inconsistencies.

The limited payload of a typical quadcopter drone will render it unfeasible to attach devices such as vortex mixers onto it for sample transport and mixing in tandem. In addition, the forces generated by a vortex mixer in operation can disrupt the planned flight pattern of a quadcopter. A solution however may be offered by the flight manoeuvring capability of the quadcopter itself. The aerodynamic lift in quadcopters, as opposed to fixed-wing aircraft, is generated by four vertically oriented propellers. This allows movements to be generated laterally, longitudinally, and rotationally with ease. In the flight control of many quadcopters, there is an in-built capability for them to perform flipping (looping) manoeuvres. This is generally offered as a means of showcasing their aerobatic abilities, where the control schemes needed to attain the kinematics has been well studied [332].
5.2.2 Experimental methodology

In order to conduct experiments to explore the possibility of in-flight mixing of biochemical solutions, a multi-rotor (quadcopter) drone was used. The drone was sourced from Parrot Inc. and the model was Parrot Mini. The drone weighed 63.5 g with diagonal size of 150 mm without the propellers. It is powered by a 550 mAh lithium battery and its flight is controlled through Bluetooth using a smartphone application namely FreeFlight. The liquid media used to illustrate the mixing capability were glycerol and deionized water. They were stained with red and blue food dye respectively. Glycerol is an essential reagent for cryopreservation, which is believed to be attributed to glycerol molecules acting to modify the hydrogen bonding ability of water molecules [333]. The highly disparate viscosities of glycerol (1.414 Ns/m²) and water (0.001 Ns/m²) however do not engender their easy mixing. For testing, 0.5 mL of glycerol and 0.5 mL was added sequentially to each 2 mL glass vial (Verex) with plastic cap. A fixture to hold an array of vials was designed and fabricated using a 3D printer (Uprint SE Plus).

Figure 5-1 shows the quadcopter that was modified to house 4 glass vials for transport. With a larger quadcopter, it will be possible to adapt this to haul a larger number of vials.
5.2.3 Results and Discussion

The vial that contains water (top) and glycerol (bottom) prior to the maneuver clearly shows segregation that is confirmed by quantitative color analysis (Figure 5-2A). After the quadcopter (with vials) was made to perform the flipping maneuver 14 times (see example in video provided), a good extent of mixing was attained (see Figure 5-2B). To ensure that the maneuver was responsible for the mixing, similar samples were transported without flipping. The results were identical to those found in Figure 5-2A.

It is important to note that the manner of mixing here follows more of a tumbling mode which is normally applied to the preparation of blended granular formulations [334]. This is unlike the reliance on aggressive shaking utilized in vortex mixers. This gentler mixing action may ameliorate problems of cell damage [335]. In our experiments, we used a quadcopter manufactured by a reputable consumer drone manufacturer (Parrot Inc). The drone was very efficient and had an endurance of around 10 minutes on a very small battery. Also, the cost of the whole setup including the drone, vials, 3D printed vial holder was under $200. The lack of any need to incorporate devices to the quadcopter is also cost effective and energy conserving. The latter offers to increase the range of travel distance that is crucial for outdoor sample transport.
5.2.4 Conclusion

In summary, the ability to transport and mix samples using the simple flip maneuver offered in quadcopters is demonstrated. This promises to advance the timeliness aspect in the transport of biochemical samples for analysis.

It is foreseeable that maneuvers aside from flipping can be performed to optimize mixing. Hardware modifications to the quadcopter, such as tilted rotors, may offer better outcomes in this respect. It should be noted that fully autonomous operation should be pursued in order to ensure mixing consistency and to lower the possibility of flight crashes. It is recommended that similar experiments are conducted using a larger quadcopter must also be explored to assess the possibility of hauling a larger number of vials.

5.3 Drone inflight centrifugation of biochemical samples

5.3.1 Background

In this second section of this chapter, a solution to conduct in-flight centrifugation using UAVs is presented. Centrifugation is another factor that is needed to control pre-analytical errors arising from sample handling and processing. In the case of whole blood samples, it is vital to separate serum and plasma from contact with the cell components within a given time frame as some test analyte levels are significantly influenced by the time between collection and centrifugation [336]. Serum that are not centrifuged within 60 minutes from collection may be contaminated by cellular components that are released by cell lysis [337] while some plasma-based assays require prompt centrifugation to avoid spurious elevated analyte levels arising from platelet activation and degranulation [338]. The payload constraint of a typical quadcopter drone renders it unfeasible to attach a centrifuge onto it for sample transport and centrifugation in tandem. This limitation can be circumvented by adapting the propeller blades of quadcopters to perform the centrifugation of samples collected in capillary tubes. The ability of capillary tubes to permit the self-filling of liquid [339] makes them attractive for the collection of samples such as blood [340] and milk [341]. During flight, these propellers can spin up to 15,000 revolutions per minute (RPM) which has been experimentally demonstrated to be sufficient to separate plasma from blood cell components held in glass capillary tubes [342]. The work described here addresses the main challenges of having (a) the drone flight characteristics unchanged with adaptation of the propellers, (b) no dislodgment of components
notwithstanding the forces developed from high speed spinning, and (c) easy installation and removal of the sample-containing capillary tubes.

5.3.2 Experimental methodology

In order to conduct experiments to explore the possibility of in-flight mixing of bio-chemical solutions, a larger multi-rotor (quadcopter) drone was used. The drone was sourced from DJI and the model was Phantom 4 Pro (Figure 5-3A). The UAV had a mass of 1388 g and diagonal size of 350 mm without the propellers. It is powered by a 5870 mAh lithium battery. Its flight can be controlled using a dedicated controller provided by the manufacturer with smartphone and tablet interfacing. The drone consisted of four removable propellers which were adapted to contain a fixture that will hold standard 75 µL glass capillary tubes (Hirschmann). While conducting the experiments, it was important to devise them in a way where the drone flight characteristics unchanged with adaptation of the propellers to ensure the flight aerodynamics were not affected. Another important was that there was no dislodgment of components notwithstanding the forces developed from high speed spinning. For ease of use and to ensure the repeatability of experiments, it was also important that the installation and removal of the sample-containing capillary tubes should be done in a simple and easy way. In order to achieve these characteristics, a fixture was devised that comprised a hollow stainless-steel tube affixed using strong adhesive (Araldite Ultra Clear) rated at 12–15 MPa to the top surface of the propeller. When the capillary tube is slid into the steel tube, it can be held in place by a bent rod fashioned to operate like a latch which is located by a hinge that is also affixed by adhesive to the propeller. An initial series of tests were conducted to ensure that the fixture could be retained notwithstanding standard manoeuvres conducted with the quadcopter. From these tests, it, it was noticed that, when the propellers rotated at high speed, there was a tendency for them to flex, leading to a possibility of failure by curved delamination.
It was found that the application of adhesives at three spots (Figure 5-3 B & C), rather than continuously, along the length of the stainless-steel tube was effective in preventing this. Once integrity was assured, tests were conducted with non-homogenized milk and blood samples. The blood sample, obtained from a healthy 34-year-old male volunteer with consent, was directly drawn into the capillary tube after a finger prick with a lancet. The capillary tubes with samples were inserted into sealing clay (Hirschmann) to ensure no losses during centrifugation.

In the test with milk samples, the capillary tube was spun for a total of 35 minutes. The tube was removed after specific time intervals to query the separation of milk. Milk fat is typically secreted from mammary epithelial cells as fat globules which are primarily composed of a globule of triglyceride surrounded by a lipid bilayer membrane similar to the apical membrane of the epithelial cells. This membrane helps to stabilize the fat globules in an emulsion within the aqueous environment of milk. Lipid has a lower buoyant density than water, so when raw original feed milk is centrifuged, the fat component is displaced to develop a cream layer. In the test with blood samples, the capillary tubes were spun for a total of 10 minutes using the adapted propellers. The tube was removed after specific time intervals to query the separation of blood sample. The resulting components of separation are red blood cells, buffy coat which is a thin layer of white blood cells mixed with platelets, and a clear solution of blood plasma.

5.3.3 Results and Discussion

The separation of milk and blood was studied after the centrifugation using the drone. When the milk was centrifuged, the cream portion becomes visible after 5 minutes of centrifugation.
but the ability to measure it quantitatively required using the line profile plot (based on brightness level) feature in a software (ImageJ). It was established that 30 minutes of centrifugation with the quadcopter was necessary to ensure good separation (see Figure 5-4).

![Figure 5-4: A sample of non-homogeneous milk centrifuged in a capillary tube and line plots](image)

When the blood was centrifuged using the drone, component separations were distinctively noticed after 5 minutes (see Figure 5-5). However, longer centrifugation is needed to pellet the cells and obtain good yields of plasma. A convenient indicator will be to determine the Packed Cell Volume (PCV), which is essentially ratio of the volume occupied by the red blood cells to the volume of the whole blood by percentage. As indicated in Figure 5-5, this value stabilizes after 8 minutes of centrifugation. This relatively short processing time offers the ability to use smaller capacity batteries, which are lighter and take a shorter time to recharge, for en-route centrifugation while delivering in traffic congested urban environments.
It is acknowledged that the use of glass capillary tubes for blood collection poses a potential safety threat as possible breakage of these fragile tubes can cause injury or infection from blood-borne pathogens. The likelihood of tube breakage occurs when some force is applied to one end of the tube for insertion into the sealing clay or possibly during centrifugation. However, capillary tube breakage was not observed during centrifugation with the quadcopter even after many countless hours of testing. Nonetheless, a safe alternative to reduce potential injury risks is to use plastic capillary tubes where previous studies have demonstrated comparable performance characteristics with glass capillary tubes [343]. Other possible compatible products include glass capillary tubes wrapped in puncture-resistant film.

There is potential to apply the milk sample centrifugation approach here to advance human lactation investigations [344]. Sub-optimal breastfeeding, especially non-exclusive breastfeeding in the first six months of life, has been found to be responsible for 1.4 million deaths and 10% of disease burden in children younger than 5 years [345]. These concerns are accentuated in areas where access to healthcare services is more limited [346]. On a similar vein, paediatric blood tests, conducted exclusively using capillary tube collection, remain a vital tool in the early detection of human immunodeficiency virus (HIV) [347, 348]. While perinatal HIV has been virtually eliminated in high-income countries, the situation in remote sub-Saharan African and Asian communities remains highly challenging [349].
5.3.4 Conclusion

The adaptation of the propellers of a quadcopter to house capillary tubes detailed in this work was shown not to affect flight characteristics, operate notwithstanding the forces that develop from high speed spinning, and allowed the capillary tube to be easily introduced and removed. Distinct separation of non-homogenized milk into its creamy component required 30 minutes of centrifugation while only 8 minutes was needed to separate human blood into red blood cells, buffy coat, and plasma. This approach offers to advance optimal medical care through improved timeliness in diagnosis procedures requiring centrifugation and complements the recent uncovered ability to conduct en-route mixing. There is also scoping to extend the centrifugation approach to environmental-based studies [350].
Chapter 6 Overall Conclusions

The investigations done in this thesis sought to develop novel methods to use UAVs for tailored bio-chemical solutions. The first part of the thesis was dedicated to review the liquid characteristics and current UAV technology and applications. The survey of liquid characteristics included studying the properties of biofluids, liquid receptacles, wettability of surfaces, impact of environmental factors on the transport of blood and stability of liquid during transport. For UAV technology and applications, a survey was conducted, of different types of UAVs, UAV flight control, impact of weather conditions on UAV flights and current applications of UAV.

On the basis of the review conducted (Section 2.1), the behaviour of liquids on superhydrophobic surfaces was investigated in Chapter 3. In the first section of this Chapter (Section 3.2), the properties of liquid droplet transforming on a superhydrophobic incline was studied. A simple approach was found to obtain discrete drops from continuous flow onto inclined superhydrophobic surfaces to facilitate device fabrication with features of low loss, low contamination, and open access for biochemical analysis. In the next section (Section 3.3), it was investigated how liquid bodies are formed on a semi-spherical superhydrophobic well. A novel method to guide the selection of flowrate for consistent discrete volume delivery in biochemical analysis was discovered. In Section 3.4, the possibility of transporting drops on an uphill superhydrophobic surface was investigated. Through the use of airflow, it was shown that uphill transport of a droplet on a superhydrophobic surface was possible.

Following this, the stability of liquid droplet on a superhydrophobic surface was studied in Section 3.5. In this section, it was investigated how a liquid body was effected by resonance when it contacted with a superhydrophobic surface. It was found that the liquid bodies sandwiched with a tip to surface spacing in the range of $2.73 \leq h < 2.45 \text{ mm}$ to a rotating surface for a volume of $10 \mu\text{L}$ exhibited resonance. In the final section of this chapter (Section 3.6), the effect of linear stepper actuation and how resonance modifies hysteresis of a liquid droplet was studied. The work in this section demonstrated that the random acceleration components from a linear stepper actuator can drive a drop that has high contact angles on a substrate to rock resonantly. In summary, Chapter 3 demonstrated some unanticipated characteristics of liquid droplets. Further research will need to be conducted on
the stability of liquid droplets before they can be considered useable in biochemical applications using UAV platforms.

In the following Chapter (Chapter 4), it was aimed to develop methods study the impact of weather conditions on UAV flights. In this vein, a gust simulator was developed to study the effects of gusts on small UAVs. The portability of the gust simulator to be easily used in any wind tunnel was also demonstrated. In testing a small blimp UAV, it was found that the “on” to “off” phase of a gust episode created more changes to the yaw, pitch, and roll than during the “off” to “on” phase. It was also found that the changes in yaw were considerably higher than that of the pitch and roll of the blimp UAV. This system will be useful in developing small UAVs that are stable during episodes of gusts. Further research will need to be conducted by studying the stability of multi-rotor UAVs and fixed-wing UAVs in a similar setup to study its applicability in other UAV platforms.

Lastly, in the final chapter (Chapter 5), novel UAV systems for biochemical applications were developed. In the first section (Section 5.1), an advanced UAV system was developed that was capable of mixing of liquid samples during its flight with making major hardware changes to the UAV system. The ability to transport and mix samples was achieved using a simple flip manoeuvre on multi-rotor UAV system. In the subsequent section (Section 5.2), a novel method using UAVs to centrifuge blood samples in-flight was developed. This was achieved through modification of the propellers to complete the process of centrifugation. The ability to perform in-flight mixing and centrifugation offers to advance optimal medical care through improved timeliness in diagnosis procedures requiring centrifugation and en-route mixing.

It is envisaged that the development of improved in-flight control measures will be a natural extension to the work done here. The salient issues in being able to carry this out are discussed in Section 2 of Chapter 2.
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