



Expulsion Effect of Superhydrophic Materials

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Abstract

This paper presents the hypothesis and experimental results of expulsion of spheres that are made up of polytetrafluoroethylene (PTFE), also known as Teflon, and Teflon spheres layered with superhydrophobic substances, from water under microgravity conditions. The microgravity was simulated in a drop tower. The microgravity tests were conducted in the 2.2 Second Drop Tower at the NASA Glenn Research Center in Cleveland, Ohio. The experimental test objects chosen were: a) the unmodified PTFE sphere to serve as the base case (30 mm diameter); b) a PTFE sphere of same diameter layered with butyl rubber (25 mm of PTFE and 5 mm of butyl rubber); and c) a PTFE sphere of same diameter layered with paraffin (25 mm of PTFE and 5 mm of Paraffin). We hypothesized that the PTFE sphere with a butyl rubber coating would rise the highest distance in the drop chamber due to its super-hydrophobicity as determined by the contact angle of the objects. We tested the effect of microgravity on fully submerged and partially submerged objects in water. The expulsion data from our experimental runs in both fully and partially submerged protocols is presented, as well as our analysis and recommendations. Our test resulted in measurable reduction of the hydrophobicity effect while exposed to microgravity. We propose new research regarding a compounding effect of hydrophobicity based on surface properties and roughness of substances.

Introduction

The microgravity environment during space flight and prolonged stay by humans in space as experienced by longterm living at the International Space Station imposes numerous adverse effects on cellular interactions within the cell systems of plants and humans.^{1,2} Lack of gravity can also affect mechanical units where solid-liquid interactions are the fundamental design function of the system.³ However, little is known about the effects of combined microgravity and solid-liquid interactions. Conducting experiments to study this phenomenon in space is expensive and time limited. Simulating microgravity on Earth is a cheaper alternative to conducting research in space.

Microgravity can only be achieved on or near Earth by putting an object in a state of free fall. We can conduct microgravity experiments on Earth using drop towers and aircraft flying parabolic maneuvers, and in space using unmanned rockets, and the International Space Station.^{4,5} Using drop towers for creating short windows of microgravity as an object falls freely serves as one of the cheaper experimental tools to conduct microgravity research on Earth.⁶ We designed and conducted our experiments at the Drop Tower to measure the effects of surface tension properties of super hydrophobic materials on expulsion of test objects from water when density, weight and other properties are eliminated by creating a microgravity condition. These experiments were conducted at the NASA's 2.2 Second Drop Tower is one of two drop towers located at the Glenn Research Center site in Brook Park, Ohio. We used the 2.2 second drop tower to conduct our experiments.

Material and Methods

The selection of materials to test under microgravity conditions that meet the superhydrophobic test were based on identification of readily available substances exhibiting maximum contact angle as calculated by Young equation. The hydrophobic materials tend to have high "contact angles" with water droplets. A contact angle is the angle between the plane and a droplet of water resting on the plane. The plane of the surface and contact angles made by the droplets are shown in Fig. 1. As contact angle increases, there is less surface area for the droplet to touch the surface with. This is what creates hydrophobic substances, where any water it is exposed to cannot stick to the substance very well.



Fig. 1. Schematic diagram of a liquid drop showing the contact angle calculations in the Young equation.

In the expulsion experiment, our goal is to maximize the water contact angle of the surface of the test object in order for the water to be "repelled", which when under microgravity conditions, would cause the object to rise out and away from the water. To maximize contact angles, we focused on certain chemical properties of substances (primarily their structures) that influence hydrophobic properties. Table 1 lists substances and their contact angles in an increasing order (i.e., from lowhydrophobicity to high-hydrophobicity): When determining the shape of the object we established a few necessary characteristics the object must have to be successful: a) it has a

Table 1.	List of substances and their Contact angles (in
	increasing order of hydrophobicity).

■#3 ▲ ►	Polymer Name ⁽²⁾ ▲▼	CAS # ⁽³⁾	γs ⁽⁴⁾ ▲ ▼	Contact Angle (5)
38	Polyvinylidene fluoride (PVDF)	24937- 79-9	31.6	89
15	Poly <i>n</i> -butyl methacrylate (PnBMA)	25608- 33-7	29.8	91
32	Polytrifluoroethylene	24980- 67-4	26.5	92
10	Nylon 10,10	-	32	94
14	Polybutadiene	9003- 17-2	29.3	96
20	Polyethylene (PE)	9002- 88-4	31.6	96
18	Polychlorotrifluoroethylene (PCTFE)	9002- 83-9	30.8	99.3
28	Polypropylene (PP)	(a)	30.5	102.1
19	Polydimethylsiloxane (PDMS)	9016- 00-6	20.1	107.2
16	Poly t-butyl methacrylate (PtBMA)	25189- 00-9	18.1	108.1
3	Fluorinated ethylene propylene (FEP)	25067- 11-2	19.1	108.5
4	Hexatriacontane	630- 06-8	20.6	108.5
13	Paraffin	8002- 74-2	24.8	108.9
31	Polytetrafluoroethylene (PTFE)	9002- 84-0	19.4	109.2
23	Poly(hexafluoropropylene)		16.9	112
24	Polyisobutylene (PIB, butyl rubber)	9003- 27-4	27	112.1

uniform shape to rise out of the water; and b) it should have a surface that is hydrophobic to maximize the rise out of water. Both of these criteria met in using a sphere made of PTEF. We determined that PTEF spheres exhibited high contact angle or superhydrophobic and would have the greatest "repel" from water under microgravity.

We believe the hydrophobic nature of the object would cause repulsion from the water source below. As a result of this we attempted to measure our object's hydrophobicity and tried to achieve super-hydrophobicity -- a property where an object is hydrophobic to the point where it can increase the bond angle of the water acting on it--by using a combination of superhydrophobic materials. Prior to testing, our approach to design of our test objects was predicated off a few key factors which we believed had the most relevance on the objects, once placed in an environment of microgravity. As such, we chose to pay closer attention to the way our objects interacted with the water in the chamber as opposed to other factors that would have negligible impact in microgravity (i.e., object's weight or density). We concluded that measuring our objects for hydrophobicity was a good metric because the degree to which our objects exhibited hydrophobic characteristics would determine how far the object would rise once it was out of the water.

Through our research, we determined that the main way to measure hydrophobicity was through the contact angle of the substance, or the degree to which the substance bends the water acting on it. Our objects all consisted of a Teflon core and outer coatings all with different contact angles. The goal of using varying degrees of hydrophobic substances was to determine the effect that contact angle had and to determine the effect of coatings on the hydrophobic nature of the substance. Our hypothesis is based on our use of contact angles information which concluded that, due to its very high contact angle, the butyl rubber had the highest chance of achieving optimal rise in the chamber. We also thought that this situation could give us an opportunity to test the viability of compounding hydrophobicity. As this ball was made of a PTFE core, and a butyl rubber exterior we wanted to test the effect of how these two hydrophobic substances would compound.⁷ Thus, we hypothesize that the PTFE sphere with a butyl rubber coating would experience highest expulsion due to its high contact angle and the ball's compounded superhydrophobic substance. We decided to create test objects made from Teflon (see PTFE on the chart), paraffin and butyl rubber which all have very high contact angles. We believed that these objects would rise out of the water quite high as their contact angles with water was very high, maximizing hydrophobicity.

Drop Tower for Simulated Microgravity Experiment

The NASA Glenn 2.2 Second Drop Tower is one of two drop towers located at the NASA site in Brook Park, Ohio. The drop tower's 2.2 second microgravity test time is created by allowing the experiment package to free fall 79 feet (24 m).





Fig. 2. Dropping in Microgravity Environment (DIME) Facility at NASA Glenn Research Center.

physical phenomena and to develop new technology for space missions. The Drop Tower uses an experiment drag shield system to minimize the aerodynamic drag on the free-falling

Table 2. Specifications of NASA Glenn Research Center2.2 Second Drop Tower.

OPERATIONAL PARAMETERS				
Microgravity Duration	2.2 seconds			
Free Fall Distance	79 feet, 1 inch (24 m)			
Gravitational	0.001 g			
Acceleration				
Mean Deceleration	15 g			
Peak Deceleration	30 g			
EXPERIMENTAL DROP PACKAGES				
Gross Drop Package Weight	1075 lbs. (487 kg) (drag shield and equipment)			
Experimental Payload Weight	up to 350 lbs. (159 kg)			
Experimental Payload	38 inches wide, 33 inches			
Diameter	high, 16 inches deep			
	(96cm x 84cm x 40cm)			

experiment. Experiments are assembled in a rectangular aluminum frame which is enclosed in an aerodynamically designed drag shield (which weighs 725 pounds, 330 kg). This package is hoisted to the top of the tower (the eighth floor), where it is connected to monitoring equipment (e.g., high-speed video cameras and on-board computers) before being dropped. A low gravity environment is created as the package freefalls from the eighth floor to the first floor, 79 feet 1 inch (24 m). The experiment was isolated from aerodynamic drag because it is not attached to the drag shield. The experiment itself falls seven and one-half inches (19 cm) within the drag shield while the entire package is falling. The drop ends when the drag shield and experiment are stopped by an airbag, located at the bottom of the tower.



Fig. 3. Test objects with their dimensions and coatings (control; wax paraffin; and butyl rubber).

Test Objects and Experiment Design

The Teflon balls were used as the base for all three test objects. The first ball was designated as the control test object and the other two Teflon balls were quoted with a thin film of paraffin and butyl rubber. The objects are defined in Fig. 3. These three objects then positioned in a rectangular container with dimensions of 63×210 inches. The first experiment (Experiment 1) was conducted with objects fully submerged in water as shown in Fig. 4. The second experiment (Experiment 2) was conducted with objects only half (or partially) submerged in water as shown in Fig. 5. The test objects were positioned at the geographic center of the test cell.

butyl rubber sprayed test subject (right). As of this point, there do not seem to be any errors that were unexpected that could change our results. While this may seem insignificant, analyzing this image and other pre-experiment images is important to understand what could have gone wrong such that our objects did not rise out of the water. This analysis helps rule out any errors with our coatings. The following Fig. 6 through Fig. 9 represent the position of the test objects before and after the drop. For example, Fig. 6 presents the test objects positioned in the test cell before the drop and Fig. 7 presents the same test objects after the drop and in microgravity.



Fig. 4. Experiment 1 positioning of the objects (fully submerged).

Results

The experiment was conducted under two different conditions. We designed experiments to study the effect of fully submerged objects and partially submerged objects in water. In microgravity, we believe the only property that will affect the degree of expulsion of the test objects is the surface tension properties at the solid-liquid interface. In Experiment 1, we fully submerged the three objects (i.e., PTFE; Paraffin coated PTFE; and butyl rubber coated PTFE objects) in water as presented in the schematic below:

Experiment 1 (Fully Submerged Test Objects)

Prior to the objects undergoing testing in the drop chamber, they looked and behaved exactly as predicted. The image shows the positions, surface and orientation of each test object in the water containers prior to drop. Each test object is sitting on the bottom of the tank as per entry requirements (meaning their densities are greater than that of water). They also all appear to have coatings or outside layers firmly attached, mainly the paraffin-wrapped test subject (middle) and the

Discussion

Data Analysis (Experiment 1)

The first set of data that we examined was very surprising to us. This was due to the fact that there was little to no movement of the balls throughout the duration of the drop. As explained above in the rationale, we suspected that the superhydrophobic nature of the test objects would lead to a large rise. When starting to analyze the data we first began to analyze why the hydrophobicity of the materials did not make an effect on the result.

We eventually concluded that there were many other factors that contributed to masking the effect of hydrophobicity. Based upon appearance many factors appeared to prevent this hydrophobic effect such as the weight of the object. However, upon future examination we thought that weight had no effect because weight is based on gravity (eliminated in microgravity). This leads to the conclusion that the coatings themselves were not actually super-hydrophobic, but as



Fig. 5. Experiment 2 positioning of the test objects (partially submerged).



Fig. 6. *Experiment 1* - Test objects positioned in the test cell BEFORE the drop into microgravity (from left to right: PTFE; Paraffin coated PTFE; and butyl rubber coated PTFE objects).

described earlier in the Materials and Methods section, each material/surface coating was hydrophobic and had relatively high-water droplet contact angles.

Data Analysis (Experiment 2)

After viewing the footage of our 2nd drop we observed very minimal changes between the first and second tests. As a result, we analyzed our rationale behind the change for the 2nd drop. Between both tests we dramatically lowered the amount of water in the chamber to make use of the hydrophobic nature of our objects. The reasoning behind our lowering of the water level was since in the first test, the balls started out entirely covered in water, we suspected that the reason the balls did not rise was because they did not leave the water.



Fig. 7. *Experiment 1* – Test objects positioned in the test cell AFTER the drop into microgravity (from left to right: PTFE; Paraffin coated PYFE; and butyl rubber coated PTFE objects).

Once the balls leave the water, the hydrophobic effects of the objects will be exacerbated because there is one force vector from the water onto the ball which would push it away, however while underwater there is force from all sides of the ball which maintains its position underwater. Thus, we assumed that taking part of the ball out of the water would make sure that it not only has an easier time getting out of the water, but also that it has less resistance to its rise. However, we never got to test this hypothesis (regarding the differential between leaving the water and staying in the water) because our balls never left the water.

We thus suspect that there was a different factor that we did not account for when we decided to make this change. Despite not being able to test effectively and completely the viability of the switch, upon comparing the video from test 1 and 2, we can see that in test 2 the balls did rise higher, even if it was slight. This might be an avenue for future testing and research on this subject.





We observed from the data obtained from both experiments that the degree to which the test objects were submerged in water had no apparent effect on the expulsion of the objects within or above the water line. This suggests that the superhydrophobic surface properties of the test objects under microgravity have minimal effects on expulsion. For both tests, we observed little change in the expulsion of the test objects from the liquid. Specifically, our hypothesis was that complete submergence of the test subjects lead to no difference in expulsion and was disproved by the results of the second trial. It means that there is a different, unrelated variable/issue which we have yet to explore. Future avenues for research are presented within the recommendation section.



Fig. 10. Microphotograph of crenellations on a lotus leaf's already hydrophobic surface.

Recommendations

With respect for the second part of our hypothesis (the compounding effect of hydrophobicity), we were forced to reject this hypothesis given that the degree to which each object rose was equal indicating that it was only the outer substance that would have made a difference. This implies that the PTFE core did not add in any way to the hydrophobic nature of the substance and only served to increase the density of the object. As a result, we turned to other properties that may have a greater effect on the hydrophobic properties of a



Fig. 9. *Experimen2* – Test objects position in the test cell AFTER the drop into microgravity, the droplets on the container are from previous tests, (from left to right PTFE; Paraffin coated PTFE; and butyl rubber coated PTFE objects).

substance. One that we had not looked at when creating the test subjects is the "roughness" of a substance. Roughness refers to the smoothness of a substance, where making a surface rougher would provide liquid droplets less space to rest on the surface.

By imagining the position of a droplet in such a scenario, it becomes clear that this would increase the contact angle for the substance. The Wenzel equation predicts that if the surface of a hydrophobic substance is etched to make it rough, the object's hydrophobic properties would be amplified (the same amplification occurs for hydrophilic properties). After a bit more research, it became clear to us that current cutting-edge advancements in creating superhydrophobic substances are not researching the chemical properties of materials themselves (which is where our research had primarily gone to for this experiment) but creating micro-etchings via lasers on surfaces. For our experiment specifically, we could use a laser printer to try and etch marks on the Teflon core. Obviously, this would not have as great of an effect on hydrophobic properties as current researchers, but it would hopefully increase it enough such that the balls would rise out of the water in the drop chamber.

While not done in our experiment, we believe that this form of compounding (not in layers as described in our proposal, but in etchings on hydrophobic materials) could have profound implications for the test objects. The addition of "spikes, or crenellations, or even thin stripes, lead to such a state because of their edges which allow the pinning of the contact line" for the droplet against the surface.⁸ The image in Fig. 10 illustrates how even in nature, rough surfaces can increase hydrophobic properties. While lotus leaves are already waxy (helps give hydrophobicity to the surface), the image shows microscopic bumps on the surface of the leaf which provide a smaller contact line for liquid droplets, thus increasing hydrophobicity.

Another error could be that our design worked against itself by being too hydrophobic. This is because, when the ball was attempting to break the surface of the water, the water above it was constantly exerting force down on the object. The result is a net neutral direction for the ball under microgravity conditions. However, as shown in Data Analysis 2, even when the balls were not completely submerged they still failed to leave the liquid. This new analysis provides insight into future experiments, drawing on existing data to propose a new way to increase expulsion based on hydrophobicity.

Conclusion

From this experiment we rejected our original hypothesis; that the PTFE sphere with a butyl rubber coating would rise the highest in the chamber due to its extreme contact angle and the ball's compounded superhydrophobic substances. While rejecting the hypothesis in a vacuum, we do believe that we were able to implicitly prove our thesis regarding the measurable effect of hydrophobicity on the rise of an object in microgravity when exposed to water. Through the pictures provided we were able to prove that the paraffin ball was most definitely non-wetting and/or hydrophobic, as was the butyl rubber ball (but to a lesser degree). Through the fact that a non-wetting object will create an indent in the water surface where the ball is exposed to the surface, we can clearly see that these objects are non-wetting.

Thus, we were able to show a measurable impact of microgravity on reducing the effects of hydrophobicity because gravity modifies the effects of surface tension to mitigate the impact of hydrophobicity on the rise of the test objects. With respect for the second part of our hypothesis (the compounding effect of hydrophobicity), we were forced to reject this hypothesis given that the degree to which each object rose was equal indicating that it was only the outer substance that would have made a difference, this implies that the PTFE core did not add in any way to the hydrophobic nature of the substance and only served to increase the density of the object. However, Data Analysis 1 shows how such a compounding effect could be possible not with multiple materials that are hydrophobic, but with a rough hydrophobic material as described by Wenzel equation.

Acknowledgments

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Conflicts of Interest

No conflict of interest.

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