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3D Printed Bioinspired Hierarchical Surface Structure With Tunable Wettability

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Nature has examples of impressive surfaces and interfaces with diverse wettability stemming from superhydrophilicity to superhydrophobicity. The multiscale surface structures found in biological systems generally have high geometric complexity, which makes it challenging to replicate their characteristics, especially using traditional fabrication techniques. It is even more challenging to fabricate such complex microstructures with tunable wettability. In this paper, we propose a method to tune the wettability of a microscale surface by changing the geometrical parameters of embedded microstructures in the surface. By taking inspiration from an insect (springtails), we designed micropillar arrays with different roughness by adjusting geometric parameters such as reentrant angle, pitch distance, and the number of spikes and pillars. This study shows that, by changing geometrical parameters in microscale, the apparent contact angle, and hence the surface wettability can be calibrated. The microscale pillars were fabricated using a precise microdirect light processing (μDLP) three-dimensional (3D) printer. Different printing parameters were studied to optimize the geometric parameters to fabricate 3D hierarchical structures with high accuracy and resolution. The largest apparent contact angle in our experiments is up to 160 deg, with pillars of 0.17 mm height and 0.5 mm diameter, 55 deg reentrant angle, and a spacing of 0.36 mm between pillars. The lowest contact angle is ∼35 deg by reducing the pillar size and spacing. By controlling the size of different features of the pillar, pillar number, and layout of the mushroom-shaped micropillars, the wettability of the surface is possible to be tuned from a highly nonwetting liquid/material combination to highly wetting material. Such wettability tuning capability expands the design space for many biomedical and thermofluidic applications. [DOI: 10.1115/1.4064051]

1 Introduction

In nature, various surfaces demonstrate different functionalities in terms of wettability. The multifunctional characteristic of surfaces in terms of both hydrophilicity and hydrophobicity has become lucrative for intensive research to adopt its unique mechanism for a wide array of applications [1]. For instance, hydrophilicity can be observed in the spines of cactus', which can absorb mist from the air and store it [2], Namib Desert beetles' (Stenocara sp.) hydrophilic bumps help to collect water from fog [3], Spider silk from Uloborus Walckenaerius possesses the capability to collect water in a definite direction [4]. Namib grass (Stipagrostis Sabulicola) also shows similar hydrophilic behavior of harvesting water droplets with the help of the texture of its surface [5]. On the other hand, hierarchical arrangement of textured surface from Springtails (Collembola), lotus leaf, rose petal, Salvinia Paradox causes repellence of aqueous medium, self-cleaning behavior, separation of oil from water, and drag reduction while demonstrating hydrophobic behavior [6–10]. Multiscale (nano to meso) hierarchical structures' existence on all of these natural surfaces determines the wettability of surfaces [11]. Orientation of these hierarchical structures and the threshold of inherent wetting determine the liquid motion's behavior along with the wetting state resulting the hydrophobic or hydrophilic behavior [12–14]. Different nano/microhierarchical structures can be found in nature in various shapes such as micropillar structure, mushroom structure, reentrant structure in lotus leaf, springtail, and gecko's feet [15,16]. Modification of the surfaces has been popular to achieve nature mimicking properties in terms of wettability of the surface and used widely to manufacture variety of biological, biomedical, microfluidics devices [6–10].

Hierarchical structures with reentrant microstructures are capable of producing local energy barriers resulting in tunable wettability [17]. Custom design of reentrant surface structures leading to tunable wettability with different functionalities is inspired by fascinating examples found in nature. Reentrant angles found in microstructure of springtail's skin surface help it to repel water [18]. In a pitcher plant, sharp-edge reentrant structures act as a dam to navigate fluids on surface [19,20]. Therefore, inspired by nature, designing reentrant structures for tunable wettability can provide an effective route to create surfaces with hydrophilic/hydrophobic surfaces, directional fluid navigation, strong adhesion [21]. The manipulation of reentrant angle can result in gradient solid–liquid–air interfaces in surfaces, therefore, leading to tunable wettability and other characteristics. And such surfaces can have various applications in different domains and industries such as soft robotics, microfluidic devices, drug delivery, and biomedical engineering. Researchers have conducted multiple studies on reentrant structures and predicted the wettability of different

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surfaces with them. Tuteja et al. studied the surface of a microstructure containing reentrant angle and introduced four parameters for designing a hydrophobic surface with reentrant structures [22]. From then, multiple studies used this knowledge and successfully developed different ranges of hydrophobic surfaces with traditional manufacturing processes [23–25].

Different manufacturing methods to fabricate surfaces and interfaces with tunable wettability and hierarchical structures include dip coating, lithography, spray/spin coating, electrochemical deposition, vapor deposition evaporation, self-assembly, and anodizing [26–28]. However, manufacturing surfaces with tunable wettability with hierarchical structures are often constrained with symmetry of structure and complexity of experiment, which is one of the reasons that most of the existing works with traditional manufacturing processes only focus on single scale, geometry, and material [29–33]. To circumvent these limitations, additive manufacturing (AM) can be useful where complicated three-dimensional (3D) parts are manufactured in a layer-by-layer method with high resolution. Various AM processes have been used so far for fabricating surfaces with multiscale hierarchical structures, such as stereolithography (SL) [33], two photon polymerization [34], and direct laser projection (DLP) [35]. There are some limitations to fabricate such surfaces with multiscale hierarchical microstructures such as the limitation of usable material and depth, more processing time in case of two photon polymerization. From that perspective, DLP process has multiple advantages like high resolution, good precision, and less manufacturing time. In micro-DLP (μDLP) a source projector is used for curing liquid photopolymer resin, in a layer-by-layer manner either in top-down or bottom-up method. After curing one layer completely, it starts curing the next successive layer till the completion of the desired part. High precision enables fabricating microstructures with higher aspect ratio, which is beneficial for surfaces with hierarchical structural array. Most of the photopolymer resins used in μDLP are hydrophilic, which can be manipulated to achieve desired wettability [36,37]. Chen et al. took inspiration from *Salvinia Molesta* and developed an additively manufactured (μDLP) surface with eggbeater shaped microstructure and cylindrical stalks resulting in good hydrophobicity. Also, they reported the effect of change in number of arms that caused change in adhesive force [38,39]. Hu et al. successfully designed and fabricated spring set in mesoscale with microstereolithography, taking inspiration from springtail cuticles, which shows hydrophobicity [40].

Current studies mostly focus on manufacturing the hydrophobic surfaces taking inspiration from the nature [41], but a few studies have been conducted for manufacturing hydrophilic ones. Among the notable studies that have been conducted for producing hydrophilic surfaces, Chen et al. used Selective Laser Melting Process to produce bio-inspired ceramic scaffold with better cell viability for its hydrophilic characteristics [42]. However, most works lack the design parameters' systematic optimization to modify the wettability of a single μDLP manufactured surface in a way that it will give desired wettability from hydrophilic to hydrophobic range with the change of few design parameters. There has not been any study as per our knowledge in literature, which shows a single design and modification of its different characteristic features to give desired tunable wettability from hydrophilic to hydrophobic range. This study aims to introduce the knowledge base for getting desired wettability as per the demand or its application. By only changing geometrical features, one can easily get customized surface with tunable wettability (Table 1).

This research demonstrates the design and μDLP fabrication of a surface with bio-inspired microstructures, which facilitates tunable wettability as a functionality. This paper explores several design parameters of the novel design, such as the reentrant geometry (inspired from springtail's cuticles) that affects the wettability of the surface. The surface structures inspired by the Springtail's cuticles along with our novel μDLP fabrication method allow us to develop 3D printed parts ranging from hydrophilic to hydrophobic. Furthermore, the μDLP method has an inherent innovation, which allows us to print hierarchical surface structures with local mechanical characteristics.

Table 1 Geometrical design parameters

Radius of the overall structure with spike	R	0.085 mm
Radius of the overall structure without spike	r	0.025 mm
Height of the structure	H	0.26 mm
Height of the upper structure	h	0.15 mm
Pitch distance	p	0.23 mm
Reentrant angle	β	45 deg
Angle of revolution around the dome structure	α	45 deg
Radius of upper end of spike	a	0.01 mm
Radius of lower end of spike	b	0.08 mm
Slant height of spike	s	0.18 mm

2 Design and Methods

2.1 Bio-Inspired Design for Tunable Wettability. The wettability of any surface can change due to the change in surface tension between liquid and the surface. Low surface tension causes high surface energy, which leads to hydrophilicity. On the other hand, low energy with high surface tension leads to hydrophobic surfaces. In nature, there are many examples of different hierarchical designs that lead to change in surface wettability. In hydrophobic surfaces, a structural barrier against liquid invasion is created by manipulating reentrant geometry of the micropores. The negative curvature created because of the reentrant geometrical structure between the air liquid interface creates negative pressure and higher surface tension. The reentrant angle at some point makes the liquid–solid surface flat which makes the pressure gradient negligible for liquid penetration. On the other hand, the reentrant angle can be tuned to make the surface pressure gradient high to make a surface more hydrophilic. We have taken inspiration from such a hierarchical surface seen in the soil-dwelling springtail, which breathes through the skin and maintains dry skin in wet soil habitats to avoid suffocation. The worm has an outer skin, which has a mushroom-like topography (reentrant structures with negative geometric angle), that can prevent liquid invasion even in smaller contact angles. In our bio-inspired surface structure, the reentrant geometry has been tuned to fabricate surfaces with tunable wettability. The dome design has been formulated by taking inspiration from the springtails' secondary granules (Fig. 1(*a*)). The secondary granules are structured with multiple primary granules on the skin pattern of springtails. This feature has been adopted in our proposed design. Reentrant angles in hierarchical structures can take many shapes like

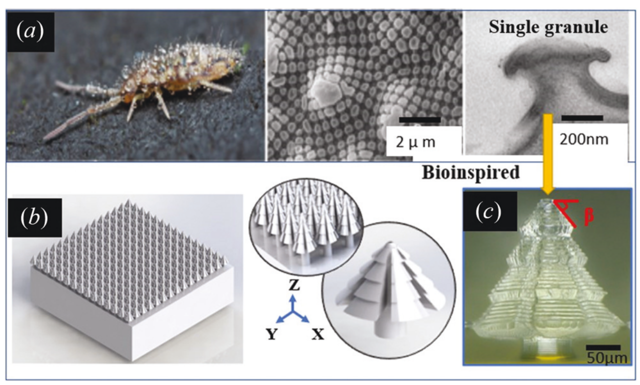

Fig. 1 (*a*) Image of a springtail insect with hydrophobic skin, images from scanning electron microscope (SEM) and transmission electron microscope (TEM) show texture on skin, and double reentrant nanostructures. Image, SEM and TEM images courtesy of Brian Valentine and are reproduced with permission [43]. (*b*) Bio-inspired computer aided design (CAD) model created with Solidworks. (*c*) 3D printed microstructure from the designed CAD model with reentrant angle *β*.

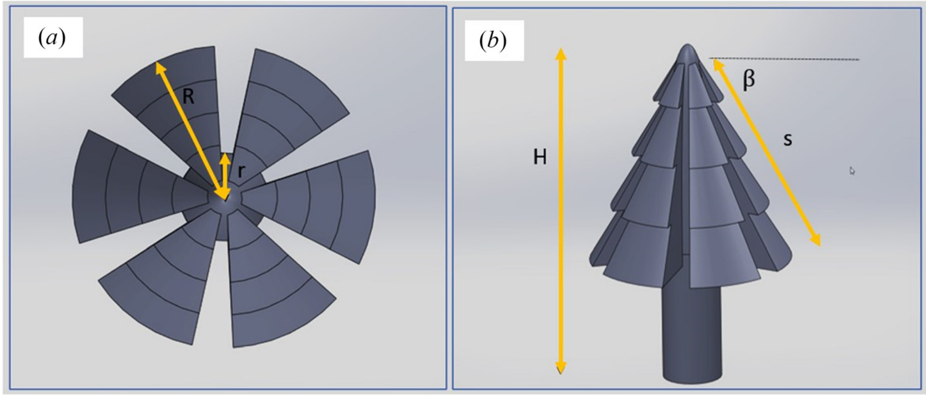

Fig. 2 Geometrical parameters of the unit structure for the designed surface of tunable wettability: (*a*) top view and (*b*) isometric view

dome, pinecone, and mushroom. Our surface design has hierarchical arrays and dome hierarchy in individual structures on its *Y*-axis (Fig. 2). The designed structure has a pinecone shape, with dome hierarchy on its *Y*-axis. Each of the domes has negative reentrant angles that are customizable for tunable features [44,45].

2.2 Overview of the Printing Process. The flowchart in Fig. 3 demonstrates the process of design and fabrication of multiscale surfaces with different reentrant angles, hence tunable wettability. The printing process starts with an initial computer aided design (CAD) design inspired from the Springtail using Solidworks (manufactured by Dassault Systems SOLIDWORKS Corp.), Version 2020, Massachusetts, USA. The artificial pinecone like arrays in different arrangements has been designed and fabricated using a μ-DLP 3D printing to achieve tunable wettability. The first part is the selection of design parameters to achieve the tunable wetting property. In the proposed design, a set of conical arrays with stair stepping effect have been designed to be printed with different

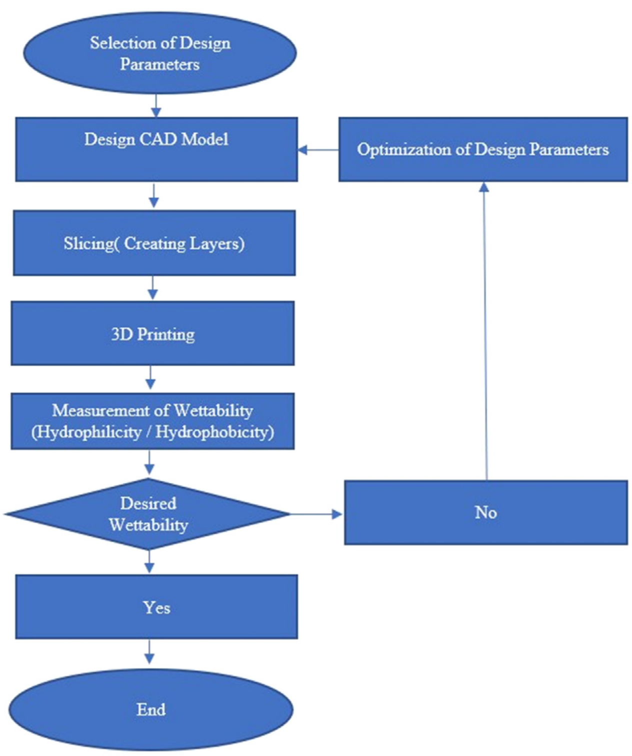

Fig. 3 Flowchart of designing surfaces with tunable wettability

reentrant angles (similar to springtail skin) ranging from 15 deg, 25 deg, 35 deg, 45 deg–60 deg. A second level of stair stepping has been created due to the layer accumulation during the 3D printing process. Such stepping effect is beneficial since it helps to tune the reentrant angles. The neighboring cone is also an important design parameter since they can increase or decrease the surface tension, changing the wettability characterization. The digital design also contains other structural information and the related governing parameters, which are later optimized according to the targeted wettability measurements. These data have been used for producing the appropriate printing and curing parameters. After loading the sliced images into the software module, the *Z* elevator moves down to initiate the μDLP and completes the fabrication process step by step. Upon the part fabrication, the desired wettability is characterized with the microscopic images.

2.3 Experimental Setup and Materials. The μDLP printer has the print volume of $15 \times 15 \times 150$ mm and minimum Z layer thickness of 5 μm. It also has tunable UV power source of 385 or 365 nm. For printing, 3DSR (viscosity 330 cP, Density 1.1 g/cm^3, Tensile Strength 29.2 MPa), an ultrahigh resolution photopolymer resin, has been used to fabricate the desired surface, which can print as low as a 5 μm feature. Canon EOS Rebel T3i DSLR (18–55 mm macrolens) along with ImageJ software has been used to capture the images for measuring the contact angles for the wettability test of the printed surfaces.

2.4 Characterization of Geometrical Constraints for Proposed Design. The manufacturing constraints for this specific printing setup have been investigated thoroughly. In our printing device, printing any dome structure requires 0.1 mm cylindrical shape to be placed beneath the dome. The design of tunable surface was designed according to that.

The dimensional parameters that have been considered in this study for manufacturing hydrophilic surface are listed as below:

2.5 Manufacturing Process. The first step to manufacture the desirable surface with tunable wettability is to design a CAD file that has been inspired from Springtail. The CAD file has been taken into 3D Builder (Microsoft, USA) to check the design's printability according to voxel size in micrometer scale. Then, the CAD file is taken to the slicing software, Kudo3D. After slicing, the design has been used for printing. The microscopic images of the printed parts are shown in Fig. 4.

3 Results and Discussion

3.1 Characterization of Contact Angles. After printing the bio-inspired (springtail) microstructure array on the 3D printed surface with photopolymer resin, contact angles were measured with in-house custom-built contact angle measurement. The tests were

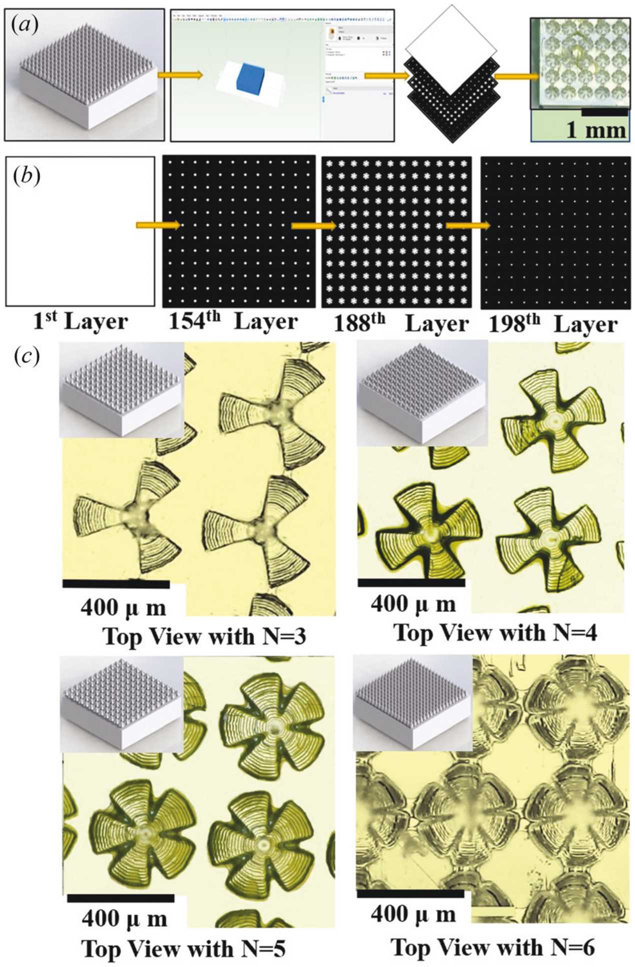

Fig. 4 Manufacturing of springtail inspired microstructure. (*a*) Design of Springtail inspired surface which was used for slicing the CAD file, and microscopic image of the printed surface with springtail inspire microstructure. (*b*) Images after slicing the CAD file of the surface with springtail inspired microstructure (spike number, $N = 6$). (*c*) Microscopic images of the 3D printed surfaces with various number of spikes from $N = 3$ to $N = 6$.

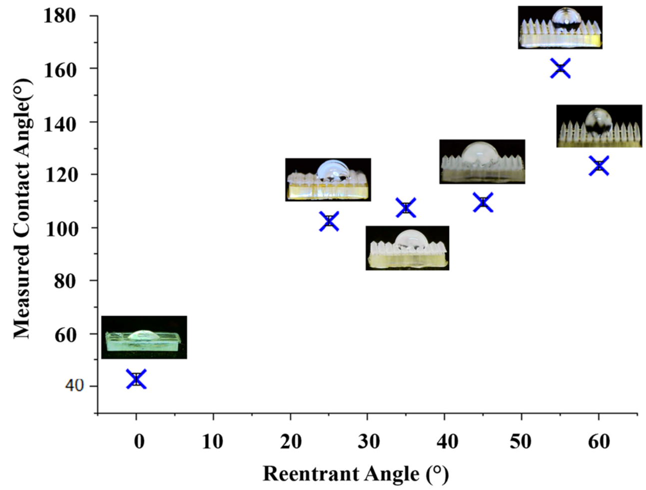

Fig. 6 Measured contact angle versus reentrant angle for surface with springtail inspired microstructure

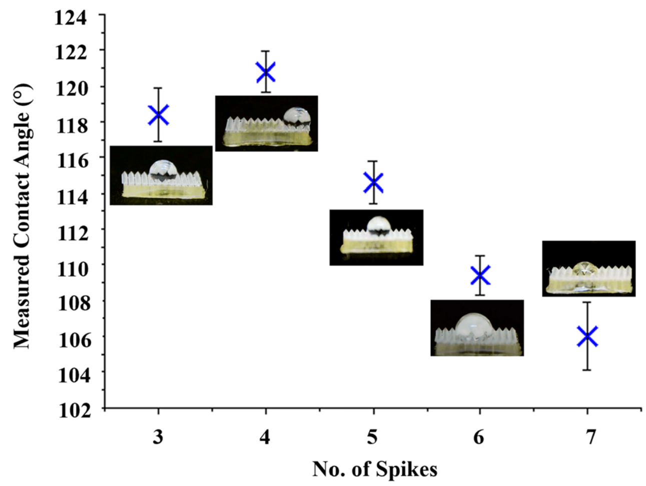

Fig. 7 Measured contact angle versus number of spikes for surface with springtail inspired microstructure

conducted with water and dispensed on the manufactured surface with the help of a micropipette. For each test, droplet volume was 3 μL (Fig. 5).

Contact angles were measured with varying three major parameters of the design that were reentrant angles, pitch distance, and number of spikes. The feedstock photopolymer resin is hydrophilic in nature. A flat surface was printed to characterize the wetting behavior of the polymer (without any microstructure) and corresponding contact angle was measured as 42.6 deg. The effect of reentrant angles is shown below at Fig. 6 where hydrophobicity was observed with increment of reentrant angles, measured contact angles are increased and got the highest value of 160 deg with 55 deg reentrant angle for this springtail inspired microstructure. For this experiment, pitch distance and number of spikes were kept as constant. Once the reentrant angle reaches 60 deg, the measured contact angle decreases due to decrement in interspikes gap. Five replications of experiments were performed, and the mean standard deviation was 1.56 ($p < 0.01$).

The effect of number of spikes on the 3D printed springtail inspired surface was observed and shown in Fig. 7. It was noted that with the increment of spikes the apparent contact angle gets increased till it is 4, once the number of spikes are increased from more than 4, due to the decrease in interspikes gap the contact angles kept lowering.

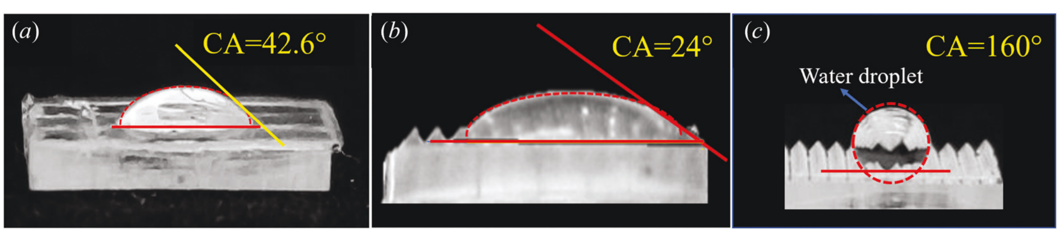

Fig. 5 Measurement of contact angle on fabricated springtail inspired structured surface with different reentrant angle showing: (*a*) surface without any structure, (*b*) hydrophilicity, and (*c*) hydrophobicity

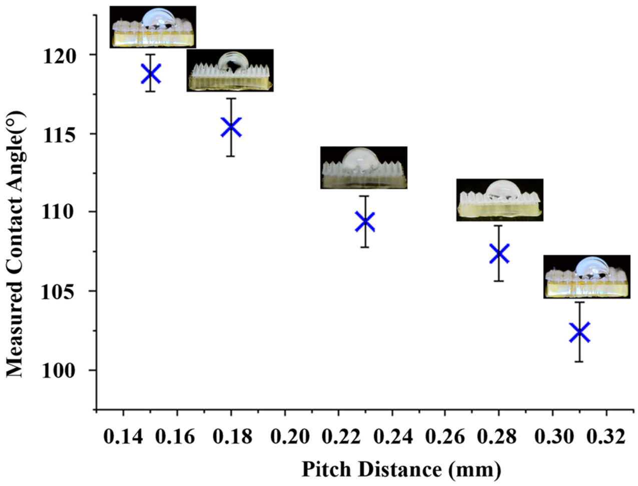

Fig. 8 Measured contact angle versus pitch distance for surface with springtail inspired microstructure

Pitch distance is one of the crucial factors that varies the wettability of a surface. For our case, keeping the number of spikes as constant, pitch distance was varied from 0.15 mm to 0.31 mm, which was constrained with the overlapping of two consecutive structures. It has been found out that for minimum distance, the measured contact angle can be highest and the more the distance between the structures get, the more it loses hydrophobicity and moves toward the hydrophilic surface (Fig. 8).

4 Conclusion

This study demonstrates the design and fabrication of a bio-inspired surface structure with tunable wettability using a μDLP AM process. It introduces one possibility that can be used further to achieve tunable wettability easily by just modifying few parameters. Three different design parameters (reentrant angles, number of spikes, pitch distance) are studied for the surface with springtail inspired microstructure. It has been shown that with the change of all these parameters for the specific bio-inspired design tunable wettability, both hydrophilicity and hydrophobicity, can be achieved. With varying reentrant angle from 15 to 60 deg, and number of spikes from 3 to 6 with varying pitch distance 0.15–0.31 mm, contact angles are measured to reach up to 160 deg. With further experimental work in our future work, we aim to correlate our experimental findings with surface energy, surface tension and how a theoretical model compares with our experimental data.

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Funding Data

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Data Availability Statement

The datasets generated and supporting the findings of this article are obtainable from the corresponding author upon reasonable request.

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