Wetting Characteristics of 3-Dimensional Nanostructured Fractal Surfaces

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KEYWORDS. Fractal, Wetting Characteristics, Two-photon Photolithography, Hierarchical

ABSTRACT: This article reports the fabrication and wetting characteristics of 3-dimensional nanostructured fractal surfaces (3DNFS). Three distinct 3DNFS surfaces, namely cubic, Romanesco broccoli, and sphereflake were fabricated using two-photon direct laser writing. Contact angle measurements were performed on the multiscale fractal surfaces to characterize their wetting properties. Average contact angles ranged from 66.8° for the smooth control

surface to 0° for one of the fractal surfaces. The change in wetting behavior was attributed to modification of the interfacial surface properties due to the inclusion of 3-dimensional hierarchical fractal nanostructures. However, this behavior does not exactly obey existing surface wetting models in the literature. Potential applications for these types of surfaces in physical and biological sciences are also discussed.

1. INTRODUCTION

Modern science relies more and more on understanding physics at the micro/nanoscale to explain phenomena at the macroscale and increase the efficiency of engineering systems. For example, heat transfer[1–6] and fluid transport [7,8] can be better understood. Consequently, surfaces can be functionalized to improve transport by optimizing their interfacial and wetting properties at the micro/nanoscale. The wetting characteristics of a surface is generally a function of surface chemical composition and topographical structure. The inclusion of multiscale structures has been proven to markedly alter the wettability of surfaces [9–16]. Inspiration for these types of surfaces often comes from nature with the intent to mimic its structures to achieve similar physical traits (e.g., superhydrophilicity, superhydrophobicity, etc.) [17–19]. Biomimetic surfaces have been therefore modeled after plant leaves for their self-cleaning attributes, different aquatic animal skins for reduced drag, and various insect eyes for antireflective properties [20]. The superhydrophobic lotus leaf is a well-known example used for engineering surface functionalization [21–23].

Going beyond simple multiscale structures, we also find in nature structures with extraordinarily ordered geometrical patterns such as fractals. The term ‘fractal’ was introduced by Benoit Mandelbrot in 1975 to describe a type of geometry that exhibits self-similarity on every length scale [24]. Fractal structures have several appealing properties: (1) A fractal

structure is inherently hierarchical due to its spanning of multiple length scales and (2) these structures can be easily characterized, as will be shown later in this letter. Additionally, fractal structures appear throughout all of nature such as in geology[25], human anatomy[26], and river networks[27]. Fractal geometries have been found to be deterministic and inherently optimal, and thus can be used to design engineering systems [28,29]. In this article, we explore the wetting characteristics of 3-dimensional nanostructured fractal surfaces (3DNFS) created using direct laser writing for functionalized surface applications.

2. EXPERIMENTAL DETAILS

2.1 Fractal Surface Fabrication

3-dimensional nanostructured fractal surfaces were fabricated using two-photon photolithography (TPP) methods. First, a Computer Aided Design (CAD) program was used to design and export the fractal surfaces as .STL files to a mesh fixing, slicing, and hatching software. This software (DeScribe) translates the files to .GWL files to be then imported to the 3D laser lithography system (Nanoscribe GmbH, Photonic Professional GT). Three distinct 3DNFS were fabricated: a cubic fractal surface, a Romanesco broccoli fractal surface, and a sphereflake fractal surface. In order to quantitatively compare the contact angle measurements for the 3DNFS, a flat control surface was also fabricated. CAD images of the individual fractal structures and corresponding surfaces are shown in **Figure 1**. Characteristic dimensions for each of the fractal structures are listed in

Table 1.

Table 1. Characteristic dimensions for each fractal structure fabricated

Fractal Structure (Dimensions from CAD models)	Largest Dimension [nm]	Smallest Dimension [nm]
Cubic (Side Length)	12,800	800
Romanesco Broccoli (Base Radius)	15,000	422
Sphereflake (Sphere Radius)	7,500	352

Figure 1. CAD models of each fractal structure and associated surface.

An acrylic-based monomer liquid photoresist optimized for TPP applications (IP-DIP, Nanoscribe GmbH) was used along with our 3D laser lithography system to transfer the 3DNFS to a glass substrate. A femtosecond laser (780 nm wavelength, 80 MHz repetition rate, and 100 fs pulse duration) was directed to an objective lens (63x zoom and numerical aperture of 1.4) that is immersed in the photoresist. The photoresist was drop-casted onto square glass substrates with side length of 2.5 mm. Four (4) duplicates of a single 140x140 μm fractal surface were fabricated on one substrate. Fabrication of three (3) distinct fractal surfaces and one (1) control sample resulted in a total of sixteen (16) surfaces.

A laser power of 15 mW was used in the TPP process and was controlled by an acousto-optic modulator. The writing speed used in this experiment (10 mm/s) was controlled by a galvo-mirror scanner. After TPP, the samples were removed from the sample holder and developed in propylene glycol monomethyl ether acetate (PGMEA) for 20 min, followed by a cleaning in isopropyl alcohol (IPA) before drying. After completion, each sample was imaged using an optical microscope to detect the presence of major defects. Successful samples were then imaged via field-emission scanning electron microscopy (SEM).

2.2 Contact Angle Measurements

Contact angle measurements on each of the fabricated 3DNFS were carried out using a micro contact angle meter (Kyowa Interface Science Co., Lt. MCA-3). Four independent contact angle measurements were taken for each fractal pattern. A single droplet of de-ionized water was dispensed from a 5 μm coated glass capillary tip on each 140x140 μm fractal surface and optically imaged at 2x zoom. Measurement and analysis software (FAMAS) integrated with the micro contact angle measurement system was then used to obtain equilibrium contact angle. Temperature and humidity were constant for all tests (75°F and 41%RH, respectively).

Due to restrictions stemming from the relative size of the dispensed droplets, each 140x140 μm surface was limited to only one contact angle measurement. For this reason, the average contact angle of the 4 tests was considered to be the true equilibrium contact angle for each 3DNFS. To compare droplet size to the roughness scale of each surface, the droplet radius was calculated. Volume calculations were undertaken from the pictures obtained during contact angle measurements. At such a small scale, gravity effects on the droplets were considered negligible and it was assumed that each droplet was spherical in shape. From this information, the droplet radius was calculated for each test.

3. RESULTS and DISCUSSION

Scanning electron microscopy images in **Figure 2** show the final 3DNFS created via the TPP fabrication process. These images show successful creation of controllable and complex hierarchical surfaces at the micro- and nanoscales. Seen in this figure, the features of each structure and surface match closely the CAD models shown in **Figure 1**. Discrepancies between the CAD models and fabricated surfaces are attributed to process resolution limitations from a combination of the laser lithography system and the photoresist used in the fabrication process.

Figure 2. SEM images of each fractal structure and corresponding surface.

The fractal dimension is a convenient way to characterize a geometry that exhibits self-similarity [30]. There are many different fractal dimensions or methods that can be used to describe such a geometry. These methods include, but are not limited to, triangulation algorithm, the variogram, and the box counting method [31]. Generally, there exists a difference in the

fractal dimension depending on which method is used to describe the geometry. However, for many classical fractal geometries, all methods result in the same fractal dimensions.

For the current work, fractal dimensions were calculated using the box counting method. The aim of the box counting method is to quantify the fractal scaling of a structure. Realistically, that would entail knowing the scaling beforehand. In other words, one must know the correct size of box that encapsulates the structure that is repeated at each scale. Box counting algorithms are then used to find an ideal way to partition a structure for calculating the fractal dimension. Using the box counting method, the fractal dimension of a structure is defined as.

$$D = \lim_{r \rightarrow 0} \frac{\log(N_r)}{\log\left(\frac{1}{r}\right)} \quad (1)$$

Where N_r is the number of repeated features and r is the scaling factor. For instance, if, at each iteration, the structure being repeated were reduced by a factor of 2, r would equal 2.

In the present study, modified MATLAB codes [32,33] were used to import and voxelize the .STL files and calculate the box counting dimensions. The box counting program gives the number of cubes, n , of size r that it took to completely cover the fractal structure. The log of n was then plotted against the log of r and a linear curve fitting was applied. The slope of the resulting curve fit is the negative of the fractal dimension for each structure.

In order to validate the procedure used to approximate the fractal dimensions of our structures, the process was performed on a simple cube. Exhibiting no fractal behavior, the fractal dimension of a cube should be close to that of its Euclidean dimension. From the procedure, the cube was found to have a box counting dimension of 3 with a 95% confidence interval of (3, 3). The results are in agreement with the expected fractal dimension of a cube. It should be noted that even though the process works for a simple cube, the box counting method is an approximation limited by the number of iterations performed.

The plots that resulted from the box counting method can be seen in **Figure 3** and the calculated fractal dimensions from this method are tabulated in **Table 2**. Each linear curve fitting can be written in the form:

$$y = mx + b \quad (2)$$

Where b is the y-intercept and m is the slope of the line given by

$$m = \frac{\log(n)}{\log(r)} = -D \quad (3)$$

and is equal to the negative of the box counting dimension, D , as shown in **Equation 1**.

Figure 3. Linear fits for determining fractal dimension of (a) simple cube (b) cubic structure (c) romanesco broccoli structure (d) sphereflake structure

Table 2. Fractal dimension for each structure with 95% confidence intervals.

Fractal Structure	Cube (Control)	Cubic	Romanesco Broccoli	Sphereflake
Fractal Dimension	3 ± 0.0	2.657 ± 0.082	2.794 ± 0.054	2.688 ± 0.06

Figure 4. Images of contact angle measurements for (a) flat control surface (b) Cubic fractal surface (c) Romanesco broccoli fractal surface (d) Sphereflake fractal surface

Photos from distinct tests for each surface are shown in **Figure 4**. Average contact angle of the flat control surface was found to be 66.8° , suggesting that the acrylic-based monomer material used to fabricate these structures is intrinsically hydrophilic ($\theta < 90^\circ$) with a moderate surface energy. Inclusion of each 3DNFS lowered the contact angle even further. Contact angle measurements for each of the surfaces are tabulated below in **Table 3**.

Table 3. Experimental data for measured contact angle and drop volume for all fractal surfaces.

Test	Flat (Control)		Cubic		Romanesco Broccoli		Sphereflake	
	Contact Angle	Drop Volume [pL]	Contact Angle	Drop Volume [pL]	Contact Angle	Drop Volume [pL]	Contact Angle	Drop Volume [pL]
1	69.3	88.3	22.4	115.1	36.5	136.4	0.0	N/A
2	66.4	54.1	18.6	94.1	37.1	144.3	0.0	N/A
3	64.8	50.3	17.2	79.4	37.0	141.0	0.0	N/A
4	66.8	82.1	16.6	71.2	36.6	131.6	0.0	N/A
Average	66.8	68.7	18.7	90.0	36.8	138.3	0.0	N/A
Standard Dev.	1.9	19.3	2.6	19.3	0.3	5.5	0.0	N/A

When concerned with the wetting of rough surfaces, there are two standard wetting models used to describe the observed phenomenon: Wenzel's model or the Cassie-Baxter model [34]. In the Wenzel model, the droplet completely wets the surface. This model is given by [35]

$$\cos\theta_W^* = r\cos\theta_e \quad (4)$$

In Eq. (4), θ_W^* is the apparent Wenzel contact angle and r is the roughness ratio of the surface, calculated as the ratio of the total surface area to the projected area of the surface. In the Cassie-Baxter model, the droplet sits on top of the structured surface. The model can be formulated as [36]

$$\cos\theta_{CB}^* = r_f f \cos\theta_e + f - 1 \quad (5)$$

In Eq. (5), θ_{CB}^* is the apparent Cassie-Baxter contact angle, f is the fraction of the projected area that is in contact with the droplet, and r_f is the roughness ratio of the wetted portion. In both Eq. (4) and Eq. (5), θ_e is the intrinsic, or Young, contact angle on a flat surface.

From the optical images seen in **Figure 4** it appears that the droplets completely wet the surfaces. Therefore, it is assumed they are in a Wenzel state. Using Eq. (4), a plot of the

theoretical Wenzel model and the experimental contact angles was constructed to determine the effectiveness of this model to characterize the 3DNFS. **Figure 5** shows that the cubic and sphereflake surfaces do not follow the Wenzel model. However, the experimental contact angle and theoretical model of the Romanesco broccoli surface are within the bounds of measurement error.

Figure 5. Comparison of experimental contact angles to the theoretical Wenzel model.

In an attempt to rationalize the trends seen in the measured contact angles, geometric properties of each surface were obtained from the CAD drawings. Surface area and surface area to volume ratio were calculated for each individual fractal structure. In addition to these values, the roughness of each surface is also tabulated and can be found in

Table 4. A bar graph of these tabulated values can be found in **Figure 6**; the contact angle of each surface is located next to its label along the x-axis.

Table 4. Comparison of geometric properties of the fabricated 3DNFS.

Surface	Average Contact Angle [deg]	Surface Area [μm^2]	Volume [μm^3]	SA:V [μm^{-1}]	Roughness [ ]
Cubic	18.7	5332.48	5022.208	1.06	7.25
Romanesco Broccoli	36.8	2597.192	4628.601	0.56	2.03
Sphereflake	0.0	3311.443	3231.7	1.02	3.89

Figure 6. Geometric properties of each fractal surface used to explain contact angle trends. Surfaces are listed in order of decreasing contact angle. Respective contact angles are bracketed next to each surface along the x-axis.

4. SUMMARY and CONCLUSIONS

As seen in the plots from **Figure 6**, there appears to be no discernable trend based on the geometrical properties of the three structures. One could attempt to correlate these geometric properties to the reduction seen in the contact angle from the Romanesco broccoli surface to the cubic surface, but the sphereflake surface does not follow the same tendency. Additionally, there seems to be no clear correlation between fractal dimension and contact angle between the three distinct surfaces. It is important to note that only four samples of each surface were tested and further experimentation may be needed. Nevertheless, we learned that 3DNFS are as fascinating in their interfacial physics as they are in their appearance. Because of their hierarchical nature and ability to be mathematically characterized, 3DNFS are an excellent platform for understanding interfacial transport in nanostructured surfaces while having great potential in many engineering applications.

For example, 3DNFS that exhibit multiscale roughness may have interesting applications in increasing heat transfer in the two-phase regime such as in condensation and boiling [1–3,6]. Another important area where 3DNFS can play a transformative role is in microfluidics. Similar structures have been used to decrease the drag on a surface, manipulate droplets, control adsorption of biomolecules, and promote cell adhesion [37–40]. Recently, flexible organic light-emitting diodes were fabricated with a nanoimprinting process to increase the efficiency for wearable electronics [41]. Similar structures to those discussed above could be fabricated to act

as a mold for this nanoimprinting procedure and help increase the efficiency of components in the rapidly growing field of electronics integration.

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Notes

The authors declare no competing financial interest.

Author Contributions

The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript.

ACKNOWLEDGMENT

This work has been supported by a NASA EPSCoR Grant # -NNX13AB17A and by funds from the Department of Mechanical and Materials Engineering and the College of Engineering at UNL, awarded to SN. This work was also supported by the National Science Foundation (CMMI 1068510 and 1129613) and funds from the Nebraska Center for Energy Sciences Research (NCESR) awarded to Yongfeng Lu. This work was performed in part in Facilities of the Nebraska Center for Materials and Nanoscience which is supported by the Nebraska Research Initiative.

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