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Fabrication of Mechanically Stable Superhydrophobic Aluminium Surface with Excellent Self-Cleaning and Anti-Fogging Properties

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Abstract

Development of the self-cleaning and anti-fogging superhydrophobic coating for aluminum surfaces which is durable in the aggressive conditions has raised tremendous interest in materials science. In this work, by employing chemical etching technique with mixture of hydrochloric and nitric acid, followed by passivation with lauric acid, superhydrophobic aluminum surface was synthesized. The surface morphology analysis reveals the presence of rough microstructures on coated aluminium surface. Superhydrophobicity with water contact angle of 170 ± 3.9° and sliding angle of 4 ± 0.5° is achieved. Surface bounces off the high speed water jet, indicating excellent water-repellent nature of coating. It is also continuously floated on water surface for several weeks, showing excellent buoyancy nature. Additionally, coating maintains its superhydrophobicity after undergoing 100 cycles of adhesive tape peeling test. Its superhydrophobic nature withstands 90° and 180° bending, and repeated folding and de-folding. Coating exhibits the excellent self-cleaning property. In low temperature condensation test, almost no accumulation of water drops on the surface, showing the excellent anti-fogging property of coating. This approach can be applied to any size and shape of aluminium surface and hence has great industrial applications.

Keywords: Superhydrophobic; Water-repellent; Chemical etching; Self-cleaning; Anti-fogging

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1. Introduction

The wettability of solid surface, characterized by contact angle and sliding angle, is determined by the combined effect of the chemical composition and the surface morphology [1, 2]. In recently, superhydrophobic surfaces (with water contact angle larger than 150° and sliding angle (SA) is smaller than 10°) have shown great interest due to the potential application in self-cleaning materials [3], antifogging surfaces [4], antireflective surfaces [5], microfluid manipulation [6] and so on. Many novel methods for the fabrication of super hydrophobic surfaces have been developed such as anodic oxidation [7, 8], chemical deposition [9], chemical etching [10-15], chemical vapor deposition [16-19], colloidal self-assembly [20-22], electrospinning [23, 24], sol-gel [25, 26] and some others [27, 28].

Aluminium has plenty applications in industries as well as in household activities due to its low weight, excellent heat and electrical conductivities, natural availability, and high mechanical properties [29, 30], but these applications are limited due to corrosion or deterioration of aluminium. Superhydrophobic coating on aluminium surface keeps water and moisture away because of its self-cleaning, anti-fogging, and water-repellent properties. This leads to keep surface dry and can slow down the process of corrosion or deterioration of surface. It is therefore desirable to create mechanical stable superhydrophobic aluminium surface with self-cleaning, anti-corrosive, anti-fogging properties which can make suitable for practical applications [31].

Superhydrophobic coating can be fabricated by using above mentioned synthesis techniques. Some of them are simple and inexpensive and some of them involve harsh conditions or require specialized reagents and equipment, which leads to increase the cost of coating. Among them, chemical etching is a facile method to prepare superhydrophobic coating for aluminium

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substrate as it has dislocations on its surface and selective dislocation etching can be easily done. Along with, chemical etching also increases its anti-corrosive property. Recently several studies on creating superhydrophobic coatings on aluminium surfaces using chemical etching technique have been done. For instance, He et al. [32] created roughness on aluminium surfaces by chemical etching using boiling water and then achieved superhydrophobicity by treating roughed aluminium using PEI and STA dip coating. Ren et al. [33] prepared superhydrophobic aluminium surfaces by creating roughness using chemical etching with hot water and then dipping of roughed aluminium in fluorosilane. Guo et al. [34] achieved superhydrophobic aluminium surfaces by roughening aluminium surface by immersing in sodium hydroxide solution and then treated with fluorinated silane. Saleema et al. [35, 36] obtained superhydrophobic aluminium surface by treating it with mixture of fluoroalkylsilane and sodium hydroxide solution. Xie et al. [37] prepared the roughness on aluminium surface by sodium hydroxide etchant and then prepared superhydrophobic coating by immersion of rough aluminium in lauric acid solution. Fu et al. [38] fabricated superhydrophobic aluminium surfaces by chemical etching using $Cu(NO_3)_2$ and HNO_3 mixed etchant solution followed by silane coating. Wang et al. [39] created roughness on aluminium surface using HNO_3 and H_2O_2 mixed etchant solution and then treated roughed aluminium in mixed solution of stearic acid and N,N-dicyclohexylcarbodiimide to achieve superhydrophobicity. Qian et al. [40] prepared superhydrophobic aluminum surface using Beck's dislocation etchant and fluorination. Superhydrophobic aluminium surface was achieved by chemical etching with sodium hydroxide etchant followed by fluorosilane coating [41]. Superhydrophobic aluminium surfaces were prepared by chemical etching and anodization using hydrochloric acid, sulphuric acid and boracic acid, followed by self-assembly using fluoroalkylsilane [42]. Zhang et al. [43] obtained superhydrophobic aluminium surfaces by immersing in hydrochloric acid and myristic

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acid solution. Li et al. [44] used hydrochloric acid as chemical etching solution to prepare superhydrophobic surface on aluminum alloy.

Despite having excellent properties like self-cleaning, anti-corrosive, anti-icing, etc., superhydrophobic surfaces are not widely industry applicable because of lack of mechanical stability. In current work, a superhydrophobic coating on aluminium surface was prepared by chemical etching technique using mixture of hydrochloric and nitric acid and coating with lauric acid solution. Additionally, wetting stability for mechanical disturbances on superhydrophobic aluminium was studied. Further, self-cleaning and anti-fogging characteristics were also studied.

2. Experimental details

2.1 Materials

Aluminium sheets (7cm X 2cm X 1mm and 1 gm) were used as substrates for the development of superhydrophobic surface. Nitric acid (HNO_3) (Emplura, Merck Specialties, Pvt. Ltd India), hydrochloric acid (HCl) (35%, Emplura, Merck Specialties, Pvt. Ltd India), ethanol (Emsure,Merck KGaA, Germany) and lauric acid (99%, Loba Chemie Laboratory reagent and fine chemicals Pvt. Ltd, India) were used in preparation of superhydrophobic coatings.

2.2 Synthesis of superhydrophobic surfaces

Synthesis of superhydrophobic coating on aluminium surface includes two-steps: first creating rough aluminium surface and then lowering the surface energy of roughed aluminium surface (Figure 1). Aluminium substrate was initially cleaned with acetone and distilled water multiple times, then aluminium substrate was immersed in a five times diluted solution of mixture of

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HNO_3 and HCl (ratio 1:3) in distilled water solution for 30 minutes. Acidic solution roughed the aluminium surface. Subsequently aluminium substrate was rinsed with distilled water and ethanol. After etching, sample was kept in oven at 60 °C for one hour. Afterward, etched aluminium sample was immersed in 20 gm/liter ethanol solution of lauric acid for 24 hours and then it was dried in air for 24 hours. Lauric acid lowered the surface energy of aluminium surface. Experiment was performed under atmospheric conditions.

2.3 Characterization of superhydrophobic surfaces

Contact angle measurements were done by using drop shape analyzer (25, Kruss, Germany) with droplet of distilled water having drop volume of 4-5 µL. The experiments were repeated at five different points on each sample and their average with standard deviation was calculated. Surface morphologies of uncoated and coated samples were examined using scanning electron microscopy (SEM) (Nova Nano SEM FEI). The roughness of uncoated and coated samples was measured by stylus surface profilometer (Veeco Dektak 150). Five scans of 1.5 mm were carried out on different surface position of each sample in order to drive the corresponding roughness.

In floatation on water surface test, coated sample was kept floating on the water surface in a petri dish and floatation time was recorded till sample started sinking. Water jet impact test was carried out by spraying water on uncoated and coated samples from a 25 ml syringe. Water jet was kept about 3 cm above the surface with angle of nearly 45° for 1 minute along with impact speed of 2.6 m/sec. The interaction between the water jet and surface was later observed.

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Mechanical durability of the superhydrophobic aluminum surface was carried out by adhesive tape peeling and surface bending tests. In surface bending tests, coated samples were bent in different directions and angles, and multiple times folded and de-folded. To check superhydrophobicity, water droplets were placed at different positions on bending or kink areas. For adhesive tape peeling test, an electrical insulation tape of adhesive strength of 100 N/m was glued and unglued multiple times on coating. Peeling tests were continued until coating lost its superhydrophobicity.

The self-cleaning test was performed by sprinkling small amount of chalk powder on the uncoated and coated aluminium surfaces. Water droplets were slowly dropped on the chalk powder sprinkled surfaces and flow of droplets were observed. In anti-fogging test, uncoated and coated aluminium samples were kept in the deep freezer (-18 °C) for five hours and then they were kept in humid atmosphere of 80% relative humidity.

3. Results and discussion

3.1. Surface morphology and wetting properties

Prior to the synthesis of coating, the aluminium substrate was cleaned and contact angle were measured. It is found that untreated aluminium surface has a water contact angle of about 70° and this means that it is hydrophilic in nature. Superhydrophobic coating on aluminium substrate was synthesized by two-step process. In this, roughness on aluminium surface was generated by etching it with HNO_3+HCl acidic solution and then lowering the surface energy of roughed aluminium substrate was performed by immersing into ethanol solution of lauric acid.

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Water contact angle and surface morphology of modified aluminium surface were characterized using contact angle measurement technique and scanning electron microscopy, respectively.

After etching aluminium substrate by HNO_3+HCl acidic solution, the etched aluminium surface was immersed in ethanol solution of lauric acid which results in the formation of sponge like layer on the surface. This happens because the carboxyl in the positive end of the lauric acid reacts with the hydroxyl or the aluminium atom through dehydrating process (reaction 1).

$$Al^{3+} + 3CH_3(CH_2)_{10}COO^- \rightarrow Al(CH_3(CH_2)_{10}COO)_3 \qquad (1)$$

Bonding the long non-positive end of the alkyl to the etched aluminium surface creates a low surface energy surface. This helps increase in water contact angle. Water static and dynamic contact angle are found to be 170±3.9° and 4±0.5°, respectively.

Figure 2 shows the SEM images of untreated and treated aluminium samples. When aluminium was etched in acidic solution, surface morphologies change and micro-pits form. Roughness of untreated and coated surfaces were measured and average surface roughness of superhydrophobic aluminium is found to be 8.76±1.50 μm while average surface roughness of untreated aluminium is observed as 0.58±0.22 μm. Air trapped in these micro-pits form air-pockets for water droplet and water contact angle increases. After immersing in lauric acid solution, aluminium laurate ($Al(CH_3(CH_2)_{10}COO)_3$) makes on a hydrophobic tails on the rough aluminium surface, which promotes water repellency on original hydrophilic aluminium surfaces according to Cassie-Bexter theory [2].

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3.2 Wetting stability of the coatings under perturbation conditions

To check the mechanical durability of superhydrophobic aluminium; water jet impact, adhesive tape peeling, and surface bending tests were carried out for. Figure 3 shows the water jet impact test for both uncoated and coated aluminum surfaces. It reveals that untreated aluminum surface does not prevent the water from spreading on its surface and water spreads immediately without bouncing off. This is due to smoothness of untreated aluminum sample with its hydrophilic nature. Whereas superhydrophobic aluminium surface prevents water from spreading and it bounces off the water jet in the opposite direction as shown in Figure 3. This is because of superhydrophobic nature of coating. Presence of air pockets and lower surface energy on the surface do not allow the impacting water jet to enter into the rough structure of the surface and it leads the bouncing off from the surface [45]. Generally, impacting water stream can irreversibly ruin the water repellent properties of the superhydrophobic surface [46-48]. The water jet was targeted at the same position for several minutes and the water jet was still continuously bouncing off the superhydrophobic surface, indicating excellent water-repellent and mechanical strength of coating.

Figure 4 shows the floating of untreated and coated samples on the water surface. Untreated aluminium sample is not able to float and sinks down immediately in the water. On the other hand, coated sample does not sink and starts floating on the water surface. Superhydrophobic aluminium repels the water and weight of displaced water becomes more than the body of sample. Therefore it remains floated for several weeks. This indicates the excellent water repellent nature of coating.

In order to check adhesive strength of coating, an electrical insulation tape was glued and unglued multiple times on surface. Figure 5 shows the different stages of the peeling test. It is

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observed that coating remains unaffected as the water droplets fall off the surface till 100 cycles of peeling. After 100 cycles, coating loses its superhydrophobicity and achieves sticky superhydrophobicity. In this situation, the static contact angle decreases and dynamic angle rapidly increases and water drop cannot roll down and remains sticky on the surface [49]. Water droplets do not even fall off the surface when surface is tilted at 90°. It is due to destroying the coating surface by multiple gluing and de-gluing of tape on the surface. Recently, Wang et al. [50] have reported a mechanically stable superhydrophobic steel surface which endured its surface microstructure against the 70 times adhesive tape peeling tests.

By introducing mechanical disturbances such as surface bending and folding, wettability of a superhydrophobic surface affects [51]. Therefore, the effect of bending as well as folding on superhydrophobicity of present coated surface is studied. In this regards, superhydrophobic sample was bent in different directions and angles. It was also repeated folded and de-fold. Bending and folding do not exhibit any effect on the superhydrophobicity. Figure 6 shows how the water droplets form bead like shape at 90° and 180° bending and water droplets also slide off by small air blow or tilting of the surface. Further, coated sample was 10 times folded and de-folded, and it is observed that water droplets still maintain their shape and roll off easily. It is concluded that superhydrophobic nature of coating remains unaltered on these mechanical disturbances.

3.3 Self-cleaning and anti-fogging properties of the coatings

On superhydrophobic surfaces, liquid drops exhibiting a spherical shape and low adhesion, roll off from the surface. During rolling off, the liquid drops carry away dust particles present on the surface, it is known as self-cleaning phenomena [52]. In this paper, self-cleaning characteristic of the superhydrophobic aluminium surface was also studied. The chalk powder particles as

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characteristic dust particles were spread on both uncoated and coated samples. Water droplets with the help of a needle were injected on both surfaces as shown in Figure 7. In case of uncoated aluminium sample, water immediately spread on the surface and dust particles retain on the surface, i.e. it does not show any self-cleaning property. On the other hand for superhydrophobic aluminium sample, water forms spherical drops on the coated surface and these droplets start rolling off the surface and carry away dust particles. This implies the strong self-cleaning ability of the coating.

In general, superhydrophobic surface prevents the condensation of water in the form of small droplets on its surface and it is known as anti-fogging property. Recently Zhang et al [53] have fabricated a superhydrophobic aluminium surface with controlled condensation effect. To test the anti-fogging property of present superhydrophobic coating, both uncoated and coated samples were kept in a refrigerator for five hours and then placed in open environment. Figure 8 shows the condensate water on the surfaces. It is observed that moisture present in the air immediately condenses on uncoated aluminium surface and accumulated water droplets are seen on the surface within few minutes. Whereas small and few spherical water droplets on superhydrophobic aluminium surfaces are formed, indicating its anti-fogging property.

4. Conclusions

In this paper, superhydrophobic coating on aluminium surface was synthesized by two-step process: creating roughness on aluminium surface by immersing in $HCl + HNO_3$ solution and then generating superhydrophobic coating on roughened aluminium surface by immersing in lauric acid solution. Surface morphology, contact angle, self-cleaning, anti-fogging, and water-repellent characteristics were investigated at various conditions. Furthermore, mechanical stability of this coating was also studied.

The surface morphology analysis reveals the presence of rough microstructures on etched aluminium surface. A static contact angle of 170 ± 3.9° with 4 ± 0.5° sliding angle is obtained of aluminium surface after chemical modification by lauric acid. The coated sample remains floating on the water surface for several weeks, showing excellent water-repellent nature. Superhydrophobic surface bounces off the water jet of high speed stream and no change in superhydrophobicity is found, indicating excellent mechanical strength of coating. Coating withstands 100 cycles of adhesive tape peeling test and after that it loses superhydrophobicity and achieves sticky superhydrophobicity. Additionally, mechanical disturbances due to bending and repeated folding and de-folding have not much effect on the superhydrophobicity. Coating shows the excellent self-cleaning property. Almost no accumulation of moisture from air on the cooled superhydrophobic surface asserts the excellent anti-fogging property of coating. The aforesaid mechanical stable superhydrophobic aluminium surfaces have potential industrial applications.

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Figure captions

Figure 1. Schematic diagram of the experiment

Figure 2. SEM images of (a) as-received untreated and (b) superhydrophobic aluminium surfaces. Insert shows a water drop image on corresponding aluminium surface

Figure 3. Optical images of water jet impact on uncoated and superhydrophobic aluminium surfaces

Figure 4. Optical images of superhydrophobic aluminium sample floating on the water surface

Figure 5. Optical images of different stages of adhesive tape peeling test.

Figure 6. (a, b) Optical image of water droplet on the bendable (about 90°) superhydrophobic aluminium surface in different directions. (c) Optical images of water droplets on the superhydrophobic aluminium surface after bending at 180° and released back to the original position.

Figure 7. Optical images of self-cleaning behavior of as-received uncoated and superhydrophobic aluminium surfaces.

Figure 8. Optical images of condense droplets due to low temperature on uncoated and superhydrophobic aluminium surfaces

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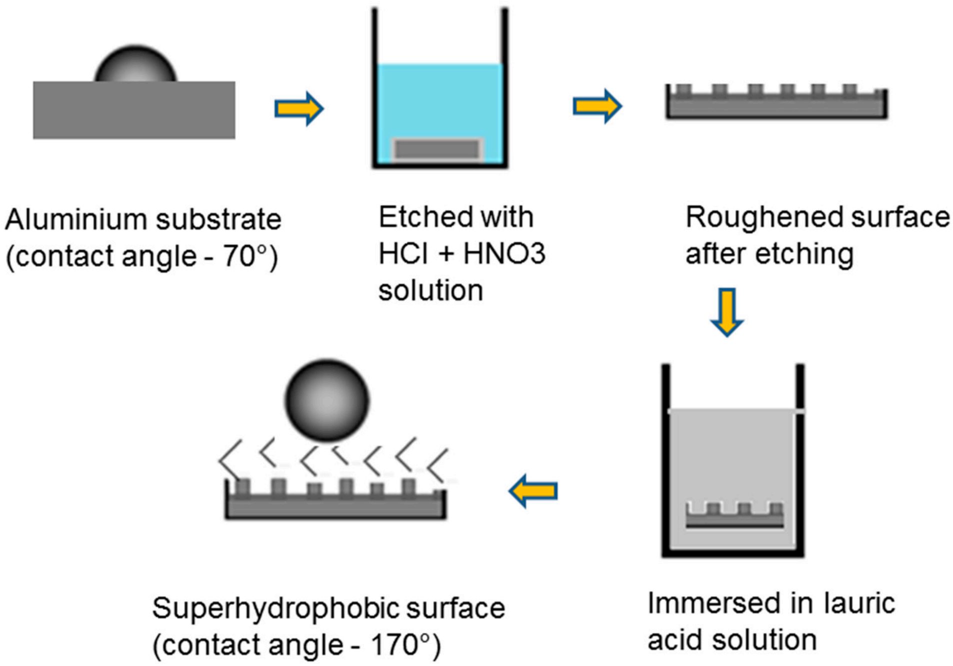

Figure 1.

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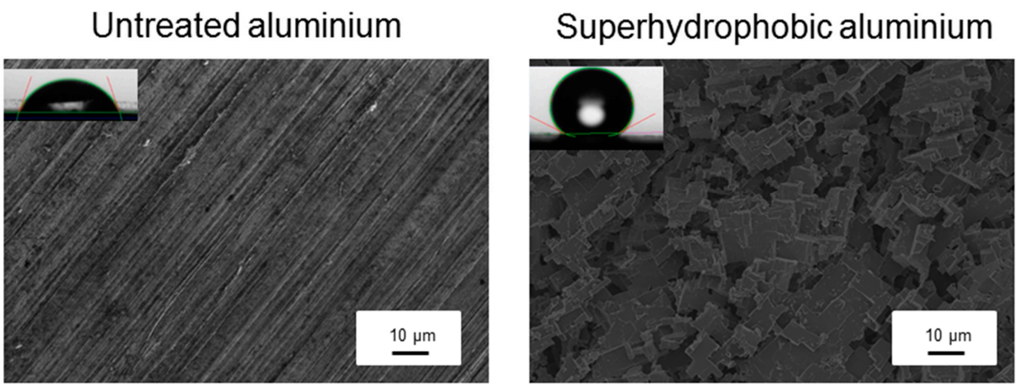

Figure 2.

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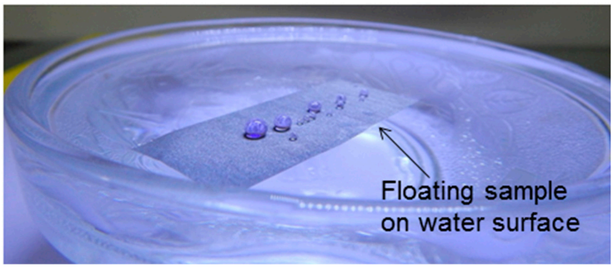

Figure 3.

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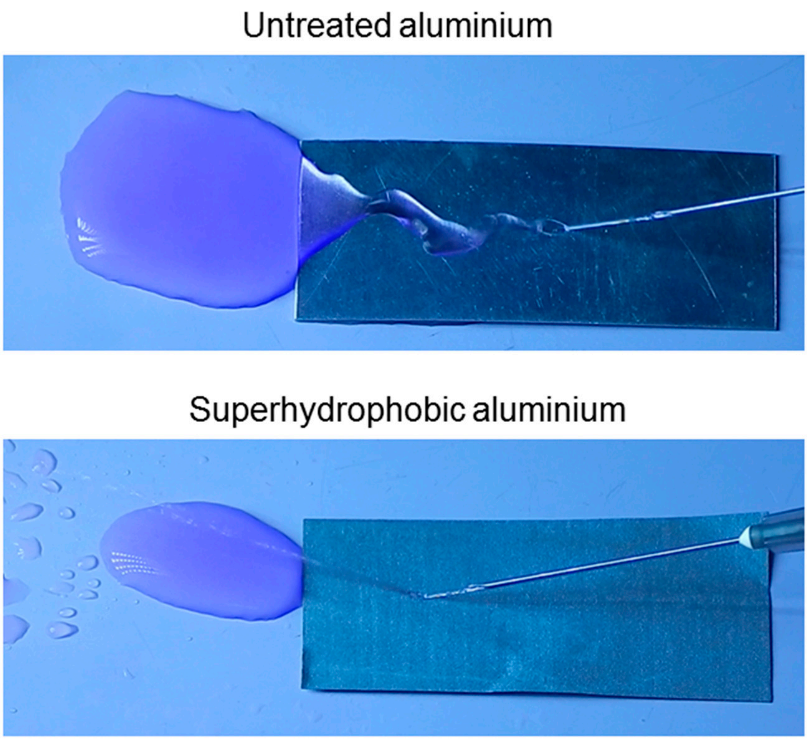

Figure 4.

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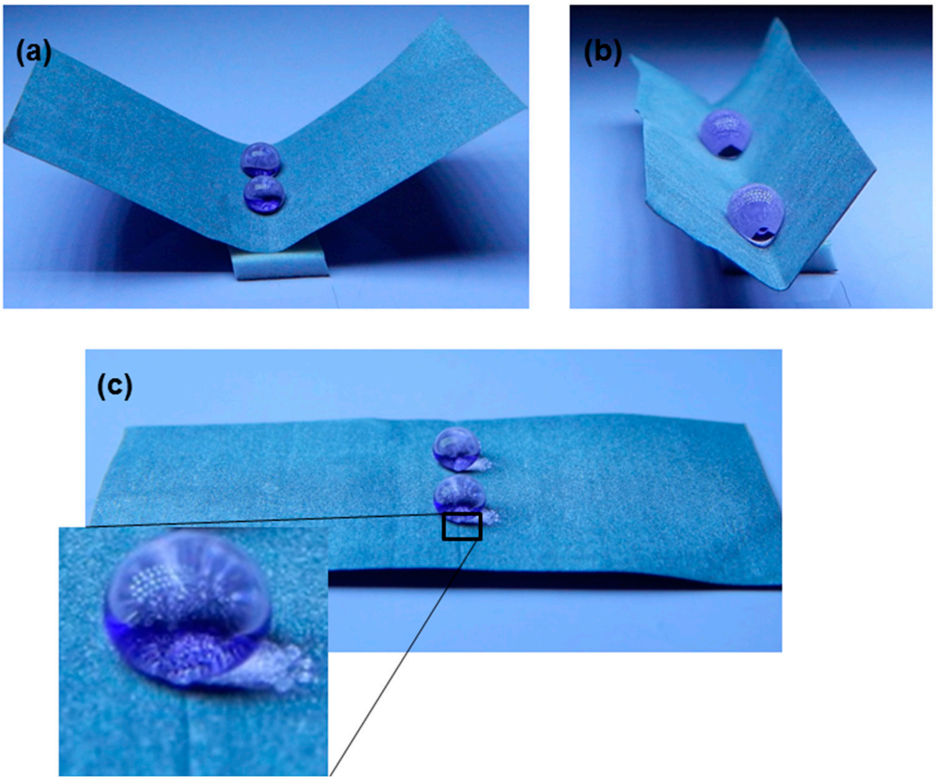

Figure 5.

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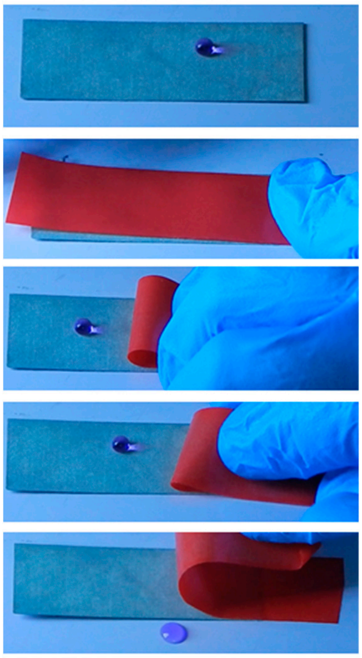

Figure 6.

Preprints (www.preprints.org) | NOT PEER-REVIEWED | Posted: 2 January 2017 doi:10.20944/preprints201701.0008.v1

Peer-reviewed version available at Biomimetics 2017, 2, 2; doi:10.3390/biomimetics2010002

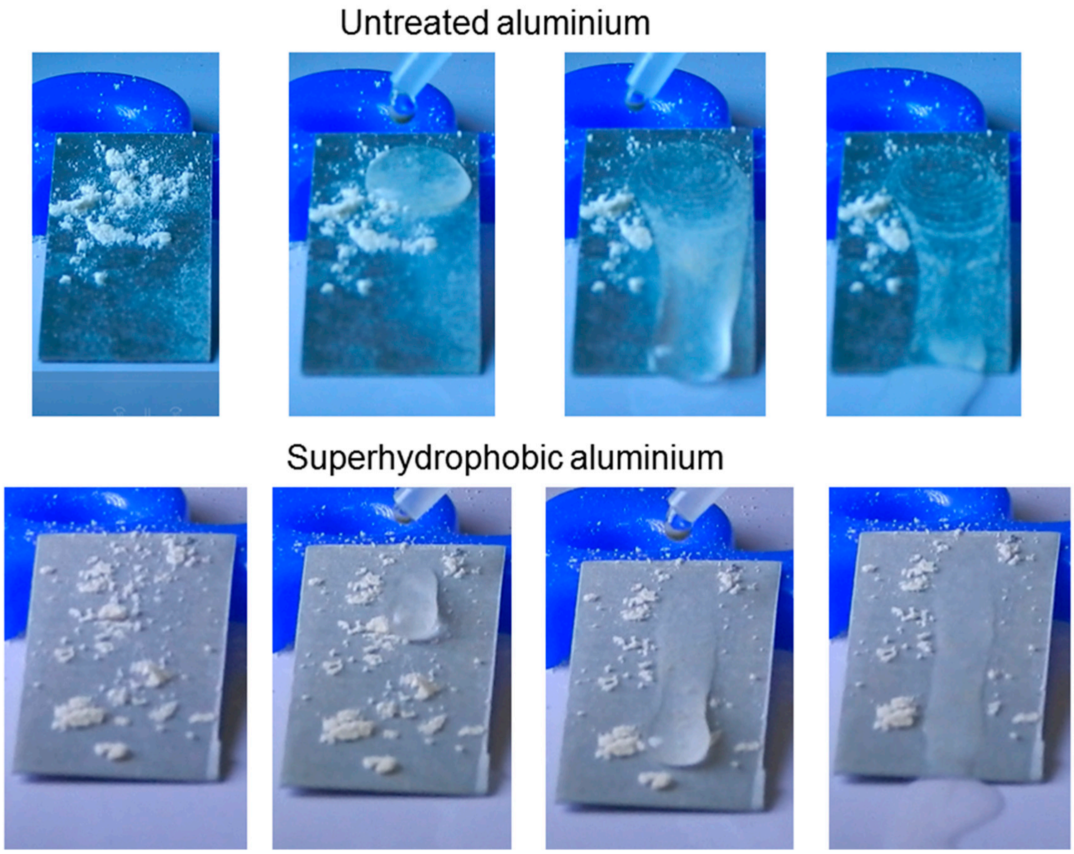

Figure 7.

Preprints (www.preprints.org) | NOT PEER-REVIEWED | Posted: 2 January 2017 doi:10.20944/preprints201701.0008.v1

Peer-reviewed version available at *Biomimetics* **2017**, *2*, 2; doi:10.3390/biomimetics2010002

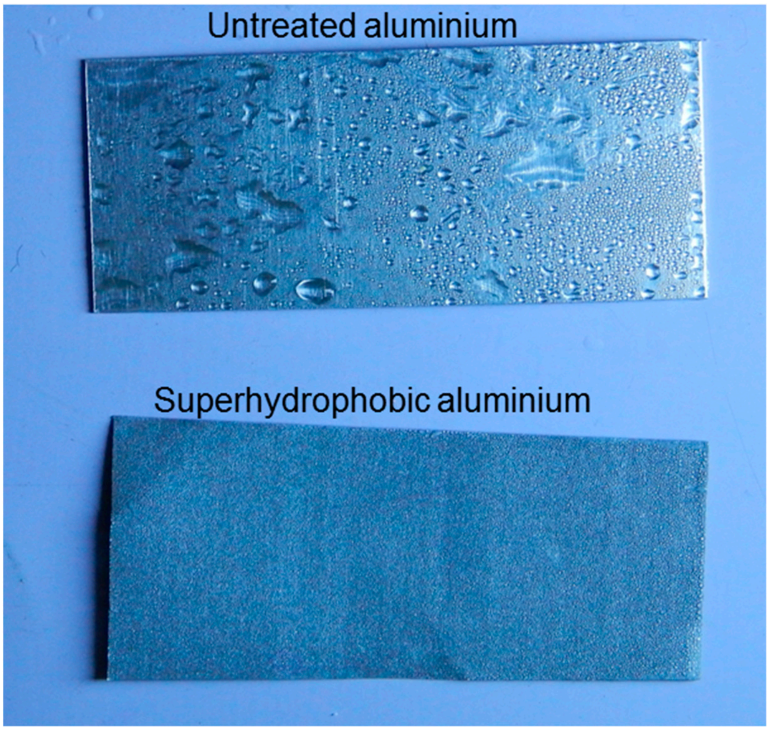

Figure 8.