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The role of hydrogen gas bubble in hydrophobic properties in mixed micro layer ($\text{Al}_2\text{O}_3+\text{Mg}$)

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ABSTRACT

Purpose: To find out more about the role of hydrogen gas bubbles in improving the hydrophobic nature of a layer, especially in the layers of microparticles Alumina (Al_2O_3) with Magnesium (Mg).

Design/methodology/approach: The method used is an experimental method by first conducting the SEM-Edx test, testing the content of the elements in the waxy layer and observing the topographic shape on the surface of the taro leaves. Then prepare a mixture of Alumina micro particles with Magnesium to investigate the hydrophobicity of the taro leaves. The mixed presentations between Alumina and Magnesium are: (0, 10, 20, 30, 40, 50, 60, 70, 80, 90 and 100%).

Findings: The results of this study found three conditions of the Alumina and Magnesium mix layer when in contact with a droplet, namely: Hydrophobic conditions occur when the surface structure of the rough mixed micro layer forms micro crevices, then bubbles of hydrogen gas fill it to form trapped gases. When droplets come in contact with the surface of the mixed layer the effect of the gas being trapped is very effective at creating hydrophobic properties. While the transition conditions occur when more and more hydrogen gas bubbles along with the increasing percentage of Mg and the opposite occurs in micro particle fissures. Bubbles fill the micro-gap space fully so that the tops of the micro particles are covered by bubbles. This causes the droplet surface tension to weaken, so the droplet contact angle decreases. Furthermore, hydrophilic conditions occur when the micro gap is getting narrower as the percentage of Mg increases and the formation of hydrogen gas bubbles increases. The high level of bubble density in the micro gap closes the peaks of the micro particles, which results in the surface tension of the droplet getting weaker. In this weak surface tension condition, the hydrogen bubble can break through the droplet surface tension and change its hydrophobic nature to hydrophilic.

Research limitations/implications: This research is limited to the hydrophobicity of Alumina and Magnesium materials, mainly to investigate the role of hydrogen gas in supporting the hydrophobic nature of taro leaves (*Colocasia esculenta*).

Practical implications: The practical implication in this study is the use of hydrophobic membranes which are widely applied to filtration.

Originality/value: Discovered the composition of a membrane mixture of Alumina (Al_2O_3) and Magnesium (Mg) to create hydrophilic and hydrophobic conditions.

Keywords: The role of hydrogen gas bubbles, Alumina, Magnesium, Micro particle peaks, Droplet surface tension weakened

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PROPERTIES

1. Introduction

The uniqueness of plants and animals is still much hidden in this nature. To uncover the secret must be studied in more detail and care. The behaviour of plants and animals when adapting to their environment is interesting to observe. Beginning in the 1990s biologists and materials began to study superhydrophobic properties in plants. Among the plants that have superhydrophobic properties are Lotus (*Nelumbo Nucivera*) and Taro (*Colocasia Esculenta*) which have a high angle of contact with water [1,2]. The surface of the leaves is very rough composed of papillose epidermal cells, which form papillae or micro-properties. Besides having a micro/nano scale roughness, the entire surface of the leaf is also coated with a layer of wax that has hydrophobic properties.

Many superhydrophobic properties currently being produced include: as a non-sticky material for the cooking equipment (Teflon), anti-wet paint, anti-dust glass, waterproof textile, etc. Materials that have hydrophobic properties are very useful, especially their ability to clean themselves (self-cleaning) of the impurities particles, so that it lightens the task of humans in cleaning it.

The main parameters that characterize the hydrophobic nature are the contact angle of the droplet when in contact with the leaf surface. The droplet contact angle depends on several factors, namely: surface energy, surface roughness and surface cleaning [3-6]. If a surface is wetted by liquid, it is called hydrophilic with a static contact angle between $0^\circ \leq 90^\circ$, whereas if the liquid cannot wet its surface is called a hydrophobic surface, with a contact angle value between $90^\circ \leq 180^\circ$.

The surface of superhydrophobic leaves has a high contact angle because gas bubbles fill the nano valleys. These gas bubbles result in smaller droplet contact with the leaf surface. Static contact angles and hysteresis contact angles of the Lotus leaves respectively around 164° and 3° [7,8]. Leaf drops of water remove contaminant particles from the surface when rolling, which leads to self-cleaning [9].

It has been investigated that all superhydrophobic leaves that are capable of self-cleaning consist an intrinsic hierarchical structure [10,11]. The rough surface structure forms an air bag, which results in the low contact area of the water droplet. The effect of the low contact plane results in a reduction in hysteresis contact angle, tilt angle, and adhesive strength [12,13]. The wavy surface texture forms an air bag that prevents water from actually touching its surface.

Hydrophobic nature research is not only done in plants and animals but has developed in other materials. Such as Alumina (Al_2O_3) powder research has been conducted to distinguish between Alumina which has hydrophobic and hydrophilic properties. An interesting finding from this study is that Alumina which has hydrophobic properties has nano gas bubbles on its surface when in contact with water. Moderate hydrophilic alumina based on the observation that no gas bubbles formed on its surface. This shows that the nano gas bubbles affect the hydrophobic/hydrophilic nature of the Alumina powder surface [14]. Recent studies have found a mixture of alumina and magnesium in creating hydrophilic, hydrophobic and superhydrophobic properties [15].

The effect of hydrophobic properties on the gas coating with Oxygen, Hydrogen and Fluorine have been carried out by [16]. This research was carried out on Ultra Diamond Nano particles. The results of the hydrophobic test show that: Oxygen coating shows hydrophilic properties with contact angles (42.3° - 51.4°), while the coating uses Hydrogen (78.5° - 83.9°) and Fluorine (101.8° - 103.9°) shows hydrophobic properties. The results of this study indicate that hydrogen gas has the potential to change the hydrophobic nature of a material.

Research on the mechanism of formation of hydrogen gas bubbles due to the superhydrophobic nature of taro leaves has been carried out by [17]. This research succeeded in uncovering the phenomenon of gas trapped on the surface of Talas leaves. The trapped gas test results show the hydrogen gas content resulting from the reaction between the elements of the surface layer of the Talas leaf and the H_2O droplet. The energy source obtained in this reaction process comes from

the surface tension of the droplet in contact with nanoparticles on the surface of the taro leaves. Gas trapped on the surface of superhydrophobic leaves has an important role in its characteristics [18]. To find out more about the role of Hydrogen gas bubbles in enhancing hydrophobic properties, a mixed micro particle layer was made by combining the hydrophobic elements Alumina (Al₂O₃) and Magnesium (Mg) elements to create Hydrogen gas bubbles.

2. Materials and methods

2.1. Material

The synthetic material used to examine the effect of Magnesium content on the hydrophobic nature of Taro

leaves is: Magnesium (Mg) powder analysis, with powder size (0.06-0.3mm) brand: KgaA, made in Darmstadt (Germany). Alumina Oxide (Al₂O₃) particle size analysis: 0.063-0.200 mm, brand: KgaA, made in: Darmstadt (Germany). These two ingredients are mixed with a percentage of Mg (0, 10, 20, 30, 40, 50, 60, 70, 80, 90 and 100%) and then a synthetic layer is made to mimic the behaviour of hydrogen gas bubbles due to reaction (Mg + H₂O) and their role in the hydrophobic nature of Taro leaves.

Based on the SEM-edx test on the Talas leaf, Figure 1a, the topographical form and the content of the elements in the waxy layer are shown in Figures 1b and 1c. The content of the elements on the entire surface (Fig. 1b) contains a lower element Magnesium than in the papilla (arrow) – Figure 1c.

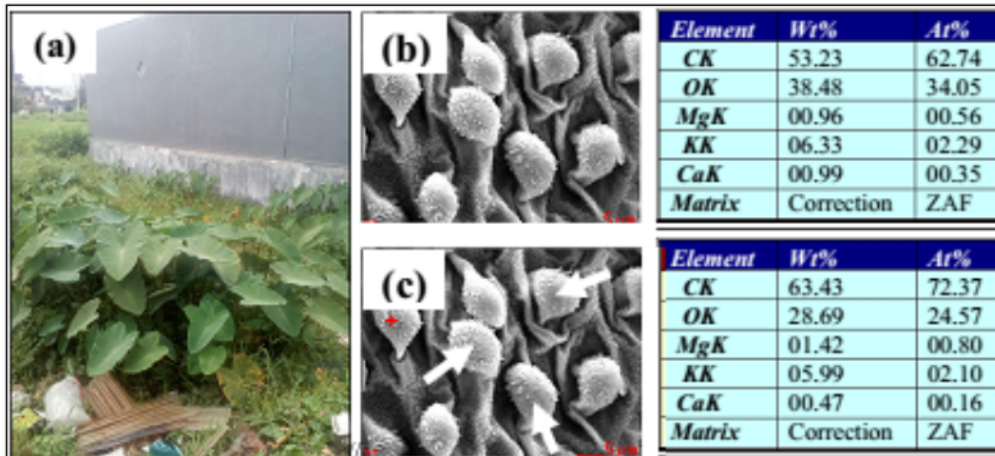


Fig. 1. a) Taro leaves and b) and c) form of topography and EDX results of Talas leaves

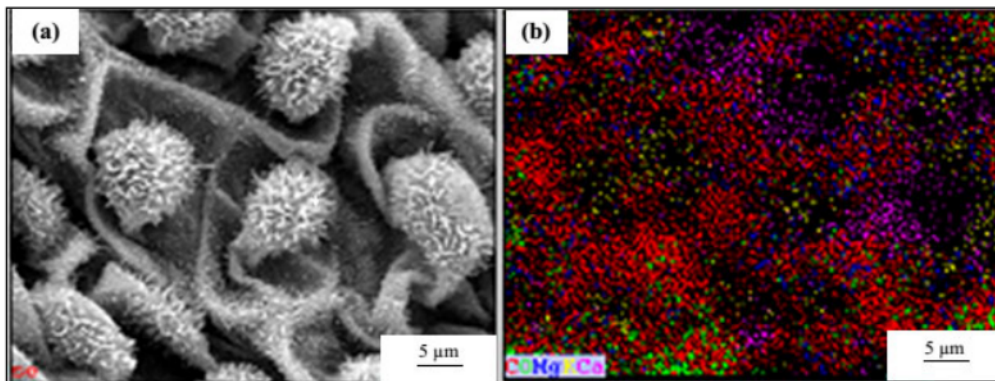


Fig. 2. Mapping test results on the surface of Talas leaves: a) mapped surfaces, b) mapping results

The mapping results in Figure 2a show the elements in the Talas leaves consisting of C, O, K, Mg and Ca. The distribution of the elements on the surface of the Talas leaf (waxy layer) is shown in Figure 2b. The element mapping results show that the Mg elements are evenly distributed throughout the surface of the waxy layer. This shows that the element Mg has an important role in the superhydrophobic nature of taro leaves.

To find out how the role of the element Magnesium in the superhydrophobic properties of taro leaves was made three layers of microparticles consisting of elements: Micro Alumina Analyst (Fig. 3a), Micro Magnesium Analyst (Fig. 3b) and Microparticles mix ($\text{Al}_2\text{O}_3 + \text{Mg}$) (Fig. 3c). This is intended to investigate the role of the elemental magnesium content in the superhydrophobic nature of the taro leaves shown in Figure 3d. Whereas the Alumina particle layer of Figure 3a and the Magnesium particle layer of the Figure 3b were used as a comparison in this study.

2.2. Research procedure

Measurement of droplet volume and contact angle

Droplet volume measurements are performed as in Figure 4, using tool (1), the droplet volume is varied from 1 to 5 ml.

The droplet contact angle measurements were made with the Microscope position (4) as shown in Figure 4. The results of the image are displayed on Notebook (6), then with software measurement the droplet contact angle measurements (2).

Hydrogen gas bubble observation

Droplet volume measurements were carried out as in Figure 4, using tools (1), the droplet volume varied (1-5 ml) dripped on the leaves of taro/micro particle layer (3). Taking pictures vertically on the droplet when the contact is made with the position of the Microscope (5) as in Figure 4. The image is displayed on Notebook (6), then with Image-J Software displayed on a mm scale.

Calculation of Mg fraction in mixtures ($\text{Al}_2\text{O}_3 + \text{Mg}$)

To adjust the number of Mg fractions of the Figure 3c (black arrow) in the Alumina layer (red arrow) in the Figure 3c (red arrow) is done by varying the amount of Mg/mm^2 in the Alumina area. Micro particles of Alumina and Magnesium weighed according to their percentage: (0: 100 mg, 10 mg: 90 mg, 20 mg: 80 mg, 30 mg: 70 mg, 40 mg: 60 mg, 50 mg: 50 mg, 60 mg: 40 mg, 70 mg: 30 mg, 80 mg: 20 mg, 90 mg: 10 mg and 100 mg: 0 mg). Calculation of percentage mix ($\text{Al}_2\text{O}_3 + \text{Mg}$) is done by first taking a picture with the

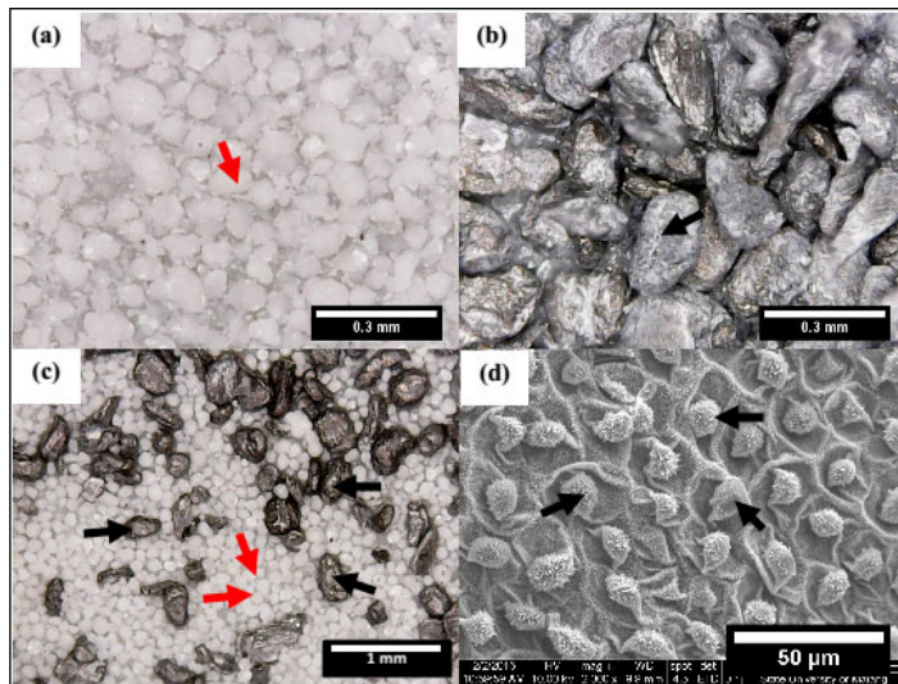


Fig. 3. Layer of micro particles (a). Alumina (Al_2O_3) (b). Magnesium (Mg) (c). Mix ($\text{Al}_2\text{O}_3 + \text{Mg}$) (d). Taro Leaves

position of a digital microscope (5) in Figure 4. Then the image results are analysed using Image-J Software, to determine the Mg fraction in the Alumina layer.

3. Results and discussion

The measurement results of contact droplet angles (1-5 ml) are shown in Figure 5. It is seen that the contact angle of the mixed layer ($Mg + Al_2O_3$) is greater than the contact angle of the Alumina and Magnesium layers. This phenomenon shows that the mixture between ($Mg + Al_2O_3$) gives a significant increase in hydrophobic properties in the micro particle layer.

In the Alumina and Magnesium layers, the contact angles formed are under the mixed layer. This indicates that there is an interesting event in mixing between ($Mg + Al_2O_3$). The increase in droplet contact angle in the mixed layer compared to Micro Alumina particles ranged (22-34%), with Magnesium microparticles ranged (133-149%) and with Taro leaves lower around (2-13%). These results indicate an increase in the hydrophobic nature of the mixed micro-particle layer has not been able to exceed the superhydrophobic properties of the taro leaves. The surface roughness factor at the nanoscale is the cause of taro leaves having higher superhydrophobic properties when compared to the mixed layer ($Mg + Al_2O_3$).

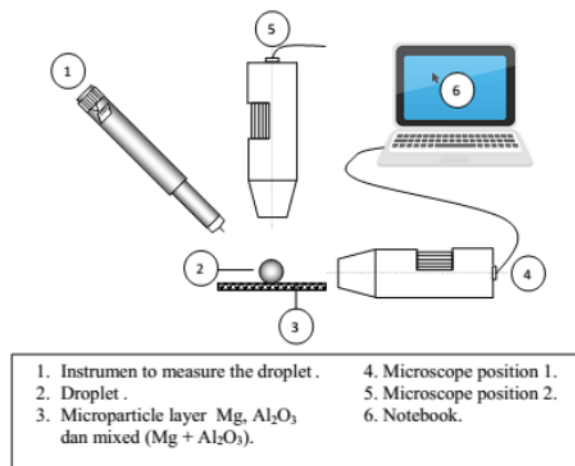


Fig. 4. Droplet contact angle measurement technique

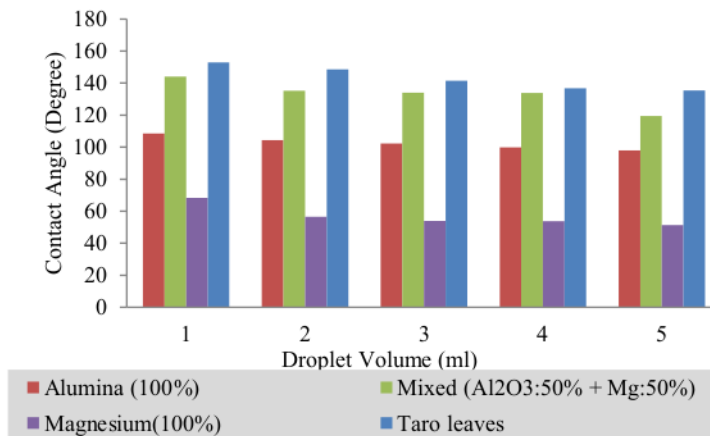


Fig. 5. Graph of the relationship between droplet volume and contact angle measurement

Figure 6 Shows droplet contact angles at various percentages of Magnesium in the Alumina layer. From Figure 6 it can be concluded that there is a certain Magnesium percentage that can create hydrophobic and hydrophilic properties. Point (1) Figure 6 shows that Alumina microparticles have hydrophobic properties while Magnesium microparticles, point (4) have hydrophilic properties.

There are four important points in Figure 6, namely, at point 1 (hydrophobic point) where the layer is composed of whole Alumina microparticles (Mg: 0%), point 2 is the maximum hydrophobic point (contact angle reaches 1440) in the Mg fraction: 50%, point 3 (critical point of hydrophobic to hydrophilic change) is achieved at the Mg: 70% fraction, and point 4 (the lowest hydrophilic point) in the Mg: 100% fraction.

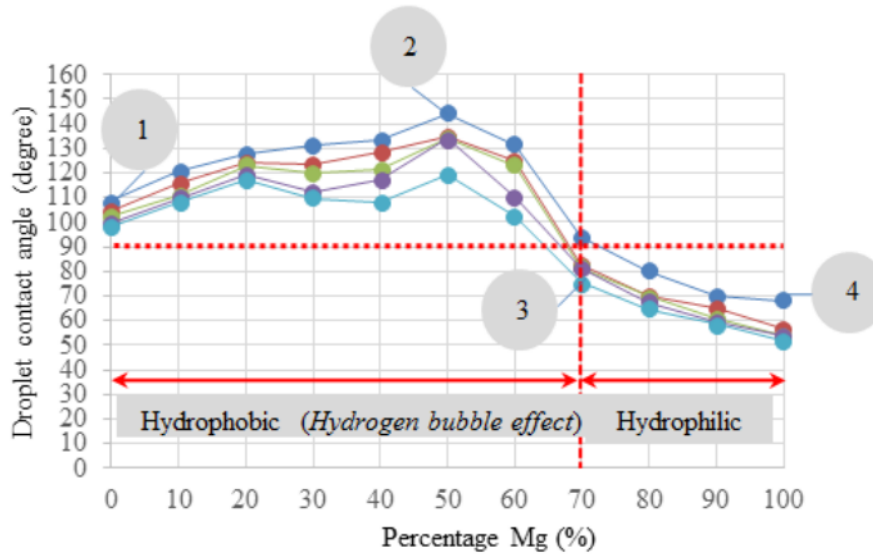


Fig. 6. Graph of the relationship between Magnesium variation in percentage droplet contact angle

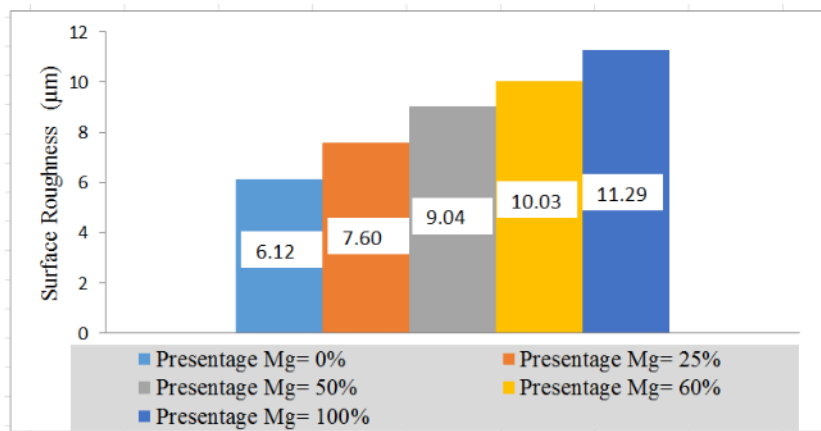


Fig. 7. Graph of the relationship between the percentage of Mg with respect to surface roughness value

Broadly speaking there are three regions formed in Figure 6: hydrophobic regions between 0% Mg to 60% Mg, transition regions at 70% Mg (point 3) and hydrophilic regions above 70%. The causes of these 3 regions are the effect of hydrogen gas bubble bearings resulting from the reaction of Mg and H_2O on a contact area between the droplet and the surface layer of microparticles.

Figure 7 shows the surface roughness at 5 Mg fractions that vary (0-100%) to the Alumina layer. It appears that increasing Mg increases surface roughness (6.12-11.29 μm). The hydrophobic nature of a material is largely determined by surface roughness. Hydrophobic surfaces become more hydrophobic and hydrophilic surfaces become more hydrophilic with increasing surface roughness values [19,20]. In mixed layer conditions (Mg + Al_2O_3) the role of Hydrogen gas bubbles can reduce the effect of roughness on the surface in creating hydrophobic properties. This is evidenced by the highest contact angle it does not occur at a maximum roughness rate but at a roughness rate between: (7.60-9.04 μm). This proves that the role of Hydrogen bubbles is quite significant in creating the hydrophobic nature of the mixed layer.

Figure 8, shows the relationship between surface roughness and contact angles at various percentages of Mg. It appears that surface roughness correlates very strongly with the contact angle. This means that the contact angle is determined by surface roughness. However, above 50% Mg the contact angle decreases with increasing surface roughness. This happens because the increase in surface roughness actually increases the surface tension which triggers the reaction formation of hydrogen gas so high that the grooves formed by the roughness are not able to

accommodate the gas bubbles so that the roughness peaks that are the source of the surface tension sink and reduce the contact angle formed as will be explained more detailed in the Figure 9.

The results of the 3-D surface analysis at the percentage of Mg: 0, 50, 70 and 100% showed different levels of roughness. The unmixed micro particle layer shows a more uniform surface with smaller grooves as shown in Figures 10a and 10d. This phenomenon changes when the micro particles in the mixed show different results where the shape of grooves gets bigger with the increasing percentage of Mg. Comparison results between grooves in Mg mix: 50%; Al_2O_3 : 50% with Mg: 70%; Al_2O_3 30% shows an increase in roughness when the percentage of Mg increases. The function of grooves in the hydrophobic nature is where the formation of hydrogen gas is trapped as shown in Figure 11. When the number of Hydrogen bubbles formed does not exceed the capacity of the grooves, the hydrophobic nature continues to increase and conditions change when the amount of gas formed exceeds the capacity of the grooves. The dimensions of the bubble enlarge and the number increases so that the contact angle of the droplet decreases as shown in Figures 9 (k-o) and 9 (p-t).

Superhydrophobic nature increases when the Hydrogen bubble does not exceed the capacity of grooves and the surface roughness effect is still dominant so that the surface tension is high and able to support the droplet above, this occurs at a percentage of Mg below 50%. When the percentage is above 50% Mg, high H_2 production makes the bubbles cover the roughness peaks so that the surface tension is lower and decreases the ultrahydrophobic to hydrophobic nature.

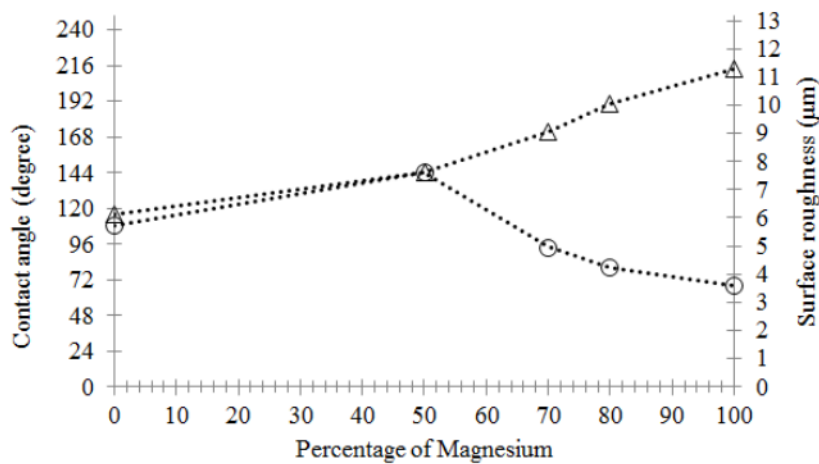


Fig. 8. Graph of the relationship of the percentage of Magnesium to the contact angle and surface roughness

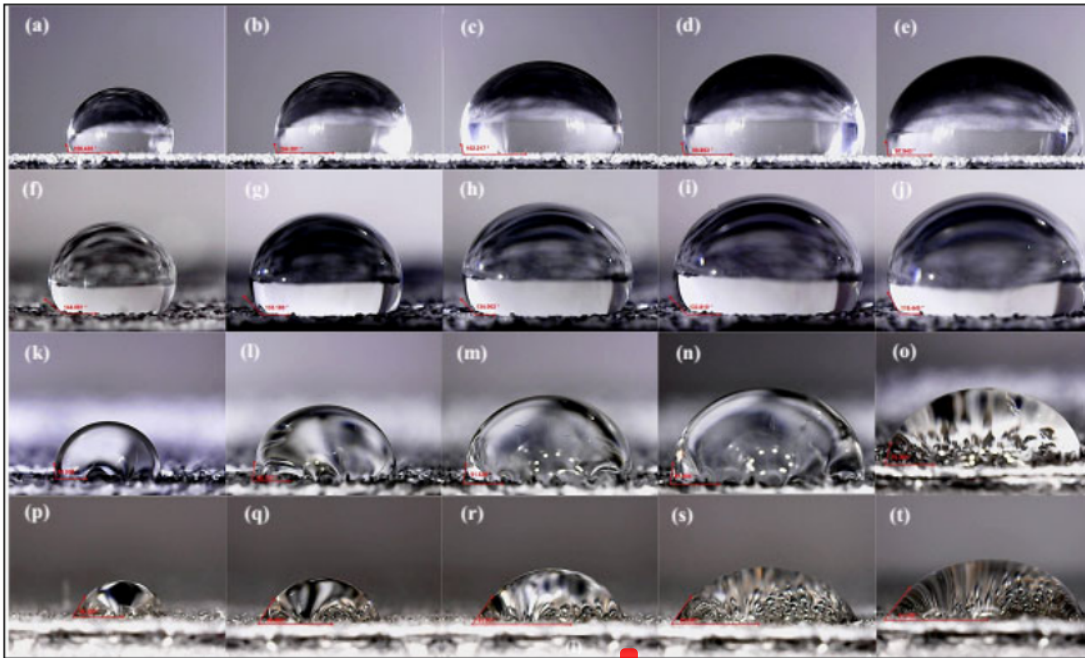


Fig. 9. Droplet test on the micro particle mix layer at the percentage of Mg: 0% (a-e), Mg: 50% (f-j), Mg: 70% (k-o), and Mg: 100% (p-t) in the droplet volume (1-5 ml)

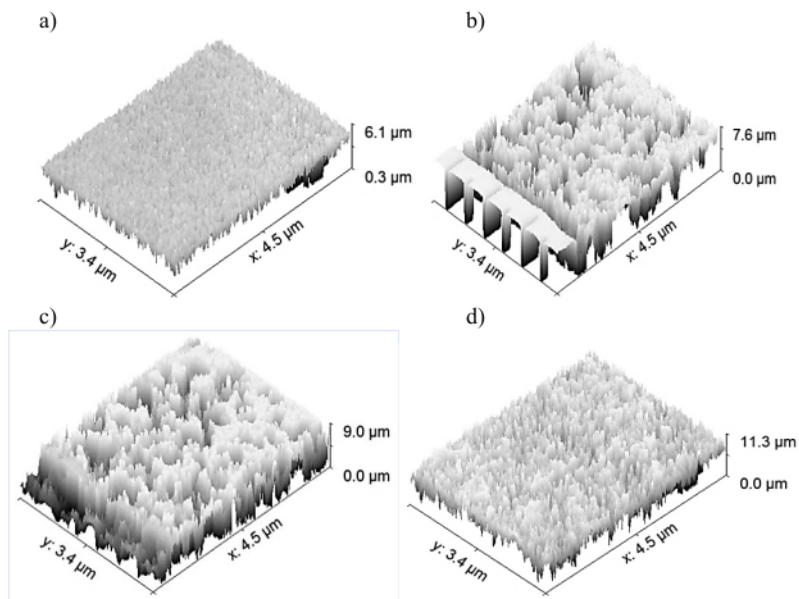


Fig. 10. Analysis of 3-D roughness: a) Mg: 0%; Al₂O₃: 100%, b) Mg: 50%; Al₂O₃: 50%, c) Mg: 70%; Al₂O₃: 30% and d) Mg: 100%; Al₂O₃: 0%

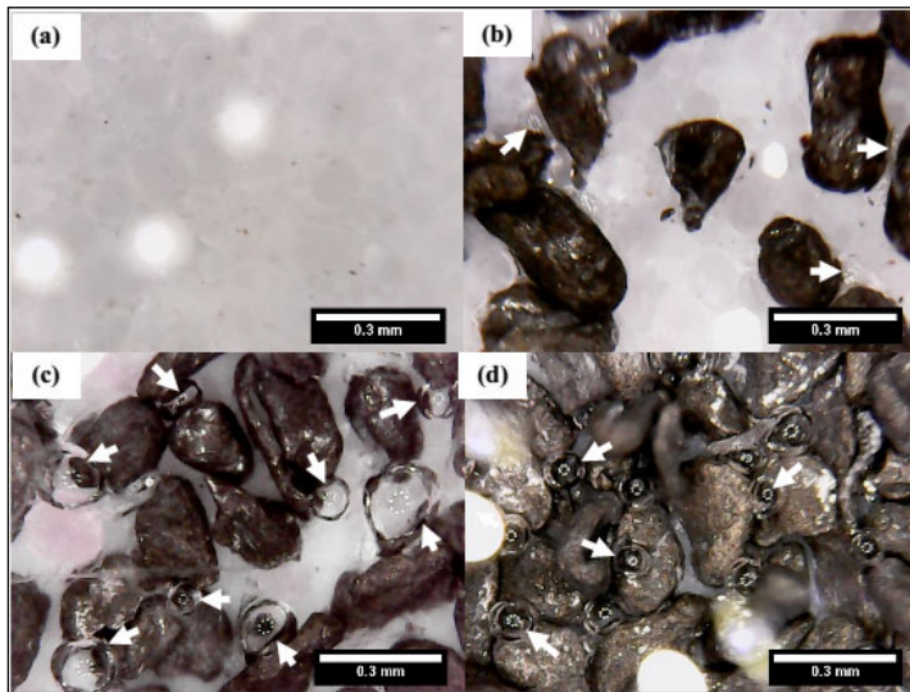


Fig. 11. Observation of 3ml droplet contact with the surface layer of micro particles using a digital microscope a) Mg Percentage: 0%, b) Mg: 50%, c) Mg: 70% and d) Mg: 100%

In the condition of Mg: 100% capacity of grooves decreases due to the uniform micro-particle size of Mg. In this condition, the hydrophobic nature decreases, due to the influence of the hydrogen bubble being more dominant compared to the dimension factor grooves. When grooves are no longer able to accommodate Hydrogen gas, bubbles form outside the grooves with a larger diameter so that they change their hydrophobic nature to hydrophilic.

When Mg and Al_2O_3 micro particles are not mixed, the surface texture forms tend to be stable, not varied and the fluctuations are low as shown in Figures 12a and 12d. This is because of the size of the micro particles that form the layer is uniform (the shape and size are almost the same), the shape of the peak that is not so sharp (sharp) compared to Figures 12b and 12c. This spiky texture forms an effect when the amount of hydrogen gas that is formed is still not too much, this spiky surface texture is effective for creating hydrophobic properties when the gas formed is still in the grooves. But when the amount of Hydrogen gas is more and more bubbles push out beyond the grooves and cover the entire surface of the grooves so that the bubble the effect is more dominant than the function of surface roughness, this

is the cause of the change in hydrophobic to hydrophilic properties in the mixed layer.

Figures 9a-e is the contact angle of the droplet at the percentage of Mg: 0%. From the picture, it can be seen that the Alumina layer has hydrophobic properties at an intermediate contact angle ($98-108^\circ$). Figure 9f-j is the contact angle of the droplet in the mixed layer with a percentage of Mg: 50%, the contact angle of the droplet is between ($119-144^\circ$) in volume (1-5 ml). The contact angle formed is the maximum contact angle as shown in Figure 6(2). In the right composition (Mg: Al_2O_3) the effect of the hydrogen gas bubble can make a significant contribution to the hydrophobic nature. Figures 9k-o is the contact angle in the transition region (the area of change from hydrophobic to hydrophilic) as shown in Figure 6(3) with a percentage of Mg: 70%. This picture shows the growth of Hydrogen bubbles on the base of the droplet which increases with increasing droplet volume. The hydrogen bubble is increasingly apparent in Figure 9o which results in a significant decrease in the contact angle. Figures 9p-t is the contact angle with the percentage of Mg: 100%. In this condition, the growth of the bubble is more clearly visible.

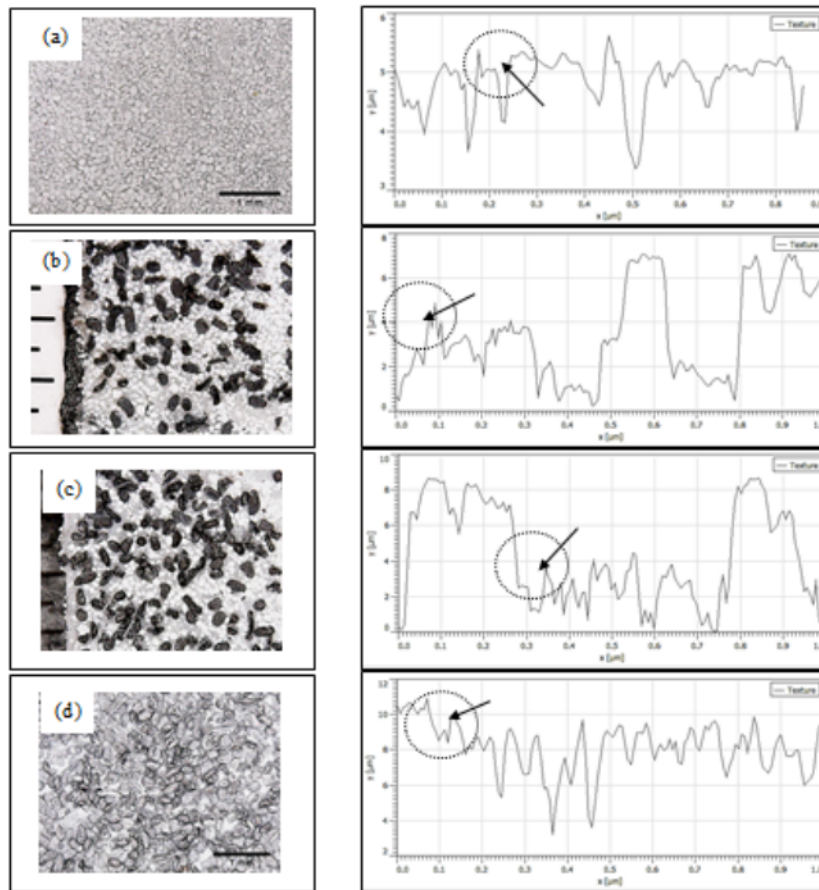
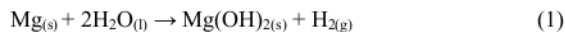


Fig. 12. Analysis of surface texture: a) Mg: 0%; Al₂O₃: 100%, b) Mg: 50%; Al₂O₃: 50%, c) Mg: 70%; Al₂O₃: 30% and d) Mg: 100%; Al₂O₃: 0%

Hydrogen gas bubble overgrowth results in a decrease in the contact angle of the droplet, so the surface properties change to hydrophilic. Hydrogen gas bubbles are formed due to the reaction between Mg and H₂O droplets following the reaction as follows:



The Magnesium element that comes in contact with water reacts to form Magnesium Hydroxide in the form of solids and releases Hydrogen gas in the form of bubbles. These bubbles play a role in the hydrophobic nature of the mixed microparticle layer (Magnesium + Alumina).

Figure 11a appearance of droplet contact on the surface of microparticles (Mg + Al₂O₃): percentage of Mg: 0%, bubbles do not form, while Hydrogen bubbles form at the

percentage of Mg: 50, 70 and 100%. The shape of the bubble varies in diameter and is spread evenly across the surface of the mixed layer.

Figure 12b hydrophobic conditions, the surface structure of a roughly mixed microlayer forming micro cracks, then a hydrogen gas bubble fills it to form a trapped gas. When droplet comes in contact with the surface of the mixed layer the effect of the gas being trapped is very effective at creating hydrophobic properties as shown in Figures 9f-j and 13a.

Figure 11c transition conditions, hydrogen gas bubbles more and more as the percentage of Mg increases and the opposite occurs in micro-particle fissures. Bubbles fill the micro-gap space fully so that the tops of the microparticles are covered by bubbles. This causes the surface tension in the droplet to weaken so that the contact angle of the droplet decreases as shown in Figures 9k-o and 13b.

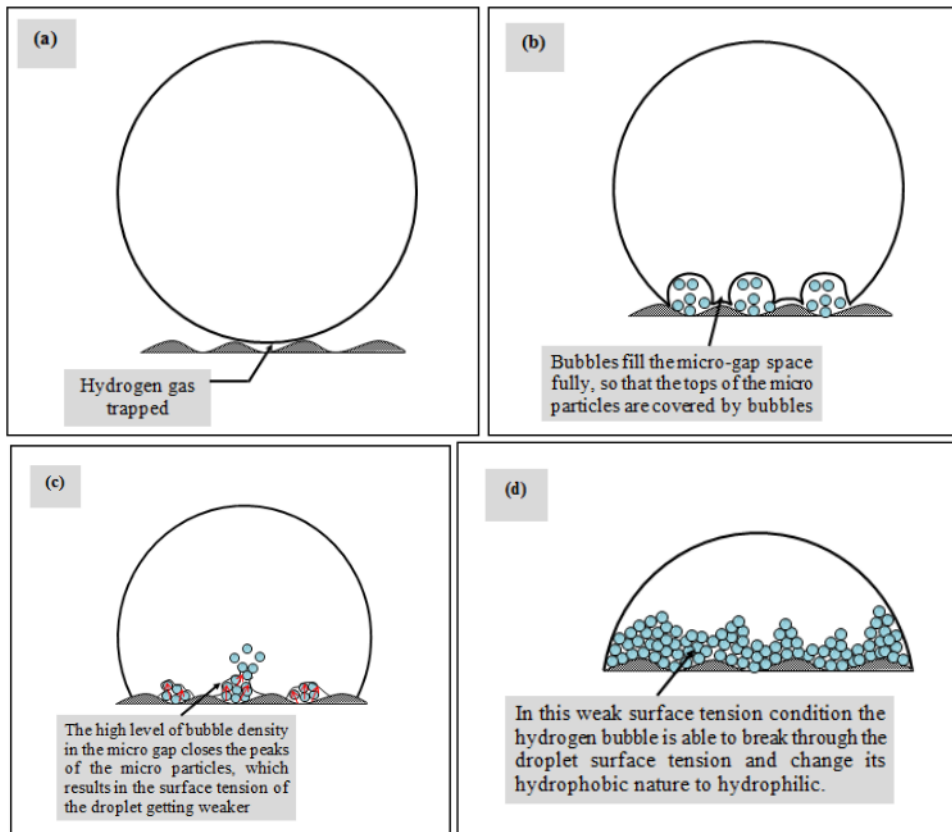


Fig. 13. The process of changing the nature of hydrophobic to hydrophilic in the mixed micro layer ($\text{Mg} + \text{Al}_2\text{O}_3$)

Figure 11d is a hydrophilic condition, the micro gap is getting narrower as the percentage of Mg increases as shown in Figure 12d and the formation of hydrogen gas bubbles increases. The high level of bubble density in the micro gap closes the peaks of the microparticles, which results in the surface tension of the droplet getting weaker. Under these weak surface tension conditions, the hydrogen bubble can break through the surface tension of the droplet of Figure 13(c) and change its hydrophobic nature to hydrophilic as shown in Figures 9p-t and 13d.

4. Conclusions

From the results of this study, it can be concluded that:

- a. Hydrophobic conditions, the surface structure of a roughly mixed microlayer forms microcracks, then a hydrogen gas bubble fills it to form trapped gas. When

droplets come in contact with the surface of the mixed layer the effect of the gas being trapped is very effective at creating hydrophobic properties.

- b. Transition conditions, hydrogen gas bubbles more and more as the percentage of Mg increases and the opposite occurs in micro-particle fissures. Bubbles fill the micro-gap space fully so that the tops of the microparticles are covered by bubbles. This causes the surface tension on the droplet to weaken so that the contact angle of the droplet decreases.
- c. Hydrophilic conditions, the micro gap is getting narrower as the percentage of Mg increases and the formation of hydrogen gas bubbles increases. The high level of bubble density in the micro gap closes the peaks of the microparticles, which results in the surface tension of the droplet getting weaker. In this weak surface tension condition, the hydrogen bubble can break through the droplet surface tension and change its hydrophobic nature to hydrophilic.

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