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RESEARCH ARTICLE

Fluorine-free Superhydrophobic Stearic Acid Modified Kaolin Nanoparticles for Self-cleaning Silicone Rubber Surface

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Abstract This study introduces fluorine-free nanoparticles to innovate superhydrophobic surfaces for self-cleaning applications, addressing environmental concerns linked to fluorine-based compounds. The method involves modifying kaolin nanoparticles with stearic acid and integrating them into silicone. The surface of stearic acid-kaolin nanoparticles exhibits a contact angle of 149.4° ± 3.3, while the resulting composite film exhibits a contact angle of 113.9° ± 5.8. The surface roughness of the silicone rubber surfaces increases proportionally with higher concentrations of stearic acid-modified kaolin nanoparticles. Field Emission Scanning Electron Microscopy (FESEM) analysis demonstrates the successful coverage of kaolin nanoparticles-stearic acid on resulting film. Self-cleaning performance is evaluated by simulating the deposition of graphite powder onto the surfaces and subsequently observing the behaviour when water is dropped on them revealing their remarkable self-cleaning properties.

Keywords: Superhydrophobic, Fluorine-free, Nanoparticle modification, Self-cleaning surfaces, Kaolin-stearic acid composite.

Introduction

In recent years, there has been a growing interest in the development of sustainable materials. Fluorinated superhydrophobic materials are highly water-repellent and have a wide range of potential applications, including self-cleaning surfaces, anti-corrosion coatings, and oil-water separation. However, the production and use of fluorinated chemicals has been linked to several environmental and health problems [1,2]. Superhydrophobic surfaces inspired by nature, such as lotus leaves, butterfly wings, fish scales, and water striders' legs, involve the incorporation of micro-nanostructures and low surface energy. A superhydrophobic surface is determined when the contact angle exceeds 150° and the sliding angle is lower than 10° [3-7].

There are two different wetting states that can exist depending on the way that a drop when contact the rough surface which called the Cassie-Baxter state and the Wenzel state [6]. In the Cassie-Baxter state, the liquid droplets rest on top of the roughness features of the surface, with trapped air beneath them. This state is characterized by water droplets being able to easily slide and roll off the surface due to its low contact angle hysteresis, typically less than 10°, resulting in self-cleaning properties known as the "lotus effect" [8]. However, depending on the surface design or external influences, the liquid droplets may undergo a transition to the Wenzel state. In the Wenzel state, the liquid infiltrates and fills the grooves or roughness features of the surface, eliminating the trapped air. This transition can happen when the surface is subjected to factors such as increased pressure, tilting, or vibration, which cause the liquid to be forced into the roughness grooves [8-12].

Silicone rubber is known for its exceptional flexibility, high thermal and chemical stability, ozone resistance, electrical insulating properties, high gas permeability, excellent hydrophobicity, physiological

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insensitivity and pronounced surface activity [13]. These qualities have led to its widespread use in both industrial production and everyday life. Silicone rubber can endure temperatures ranging from -50°C to 250°C, making it suitable for a wide range of applications in both hot and cold environments. Its resistance to ozone degradation also makes it an excellent choice for outdoor applications. Furthermore, silicone rubber is permeable to gases, making it an excellent choice for applications such as medical implants and membranes [14]. It is widely used in many fields due to excellent physical and chemical properties, including mechanics, sealing, interfacial and friction properties, among others. This versatility continues to attract many researchers who explore and characterize its constitutive model, friction and contact properties, new material preparation, sealing performance, and more, resulting in significant research productivity. Additionally, silicone rubber's main chain, composed of -Si-O- bonds, has higher chemical bond energy, which imparts better thermal stability compared to other elastomers with main chains composed of -C-C- bonds [15]. Commercial silicone rubber consists of two parts with a fixed mixing ratio. Part A contains vinyl groups as a crosslinker, part B contains Pt or Sn organometallic as a catalyst [16].

Recently, there has been a shift towards the development of fluorine-free, eco-friendly superhydrophobic surfaces. One promising alternative is stearic acid, a long-chain fatty acid that can be used to improve water repellency [17]. Stearic acid has been shown to significantly enhance the hydrophobicity of a variety of materials, including cotton fabric, metakaolin, and cement-based materials [18]. For example, Sharif et al. demonstrated that stearic acid polymerized with citric acid has created a durable superhydrophobic cotton fabric [19]. Yang et al. developed a stearic acid modified metakaolin to improve the resistance of cement-based materials [18]. Ahmad et al. introduced CeO2 nanoparticles and stearic acid to create a fluorine-free coating with self-cleaning properties, which enhance surface roughness and reduce surface sorption energy [20]. Research by Wang et al. also showed that the application of a alumina particles to silicone led to superhydrophobicity with water contact angle and rolling angle of 156.6° ± 1.1° to 5.1° ± 0.7° [14]. Badre et al. has modified ZnO nanowire films by dipping the substrate in stearic acid which resulted these films possess superhydrophobic properties and excellent stability for long-term storage conditions [21]. These results suggest that stearic acid is a promising fluorine-free alternative for the development of durable and sustainable superhydrophobic surfaces.

This study endeavors to create self-cleaning surfaces that are devoid of fluorine. We achieve this by combining superhydrophobic stearic acid-modified kaolin nanoparticles with silicone to create a self-cleaning surface. Various weights of kaolin nanoparticles are dispersed in a 1 wt% ethanol-stearic acid solution to produce superhydrophobic kaolin particles, which are then integrated with silicone to produce self-cleaning films. The characterization studies encompass SEM, contact angle measurements, AFM analysis, FTIR spectroscopy, and more.

Materials and Methods

Silicone rubber Elastosil LR 3003/60 which have two components (A and B) and curing agent were supplied by Immortal Green Industrial Sdn Bhd (Wacker, USA). Ethanol was obtained from Analytical laboratories (HmbG Chemicals, Germany). Stearic acid purchased from In Scent GMBH (Germany), and kaolin purchased from Kaolin (Malaysia) Sdn Bhd; N-heptane was obtained from VCHEM; Deionized water was prepared in the laboratory.

Preparation of Superhydrophobic Stearic Acid Modified Kaolin Nanoparticles

Kaolin nanoparticles (0.1 g) were weighted using a weighing scale and dispersed in 1 wt% ethanol-stearic acid solution to form stearic acid modified kaolin nanoparticles. Then, the solution was continuously stirred and refluxed at a temperature of 60°C for 8 hours. The solution was then centrifuged. After centrifugation, the pellet was rinsed with ethanol to remove the residual stearic acid from the solvent before being dried in an oven at a temperature of 50°C for 3 hours [4]. The stearic acid modified kaolin nanoparticles were prepared with different weights of dispersed kaolin from 0.1g to 0.4g: (Kaolin-0.1@SA), (Kaolin-0.2@SA), (Kaolin-0.3@SA), and (Kaolin-0.4@SA). Figure 1 shows the illustration of the preparation of superhydrophobic nanoparticles in this work.

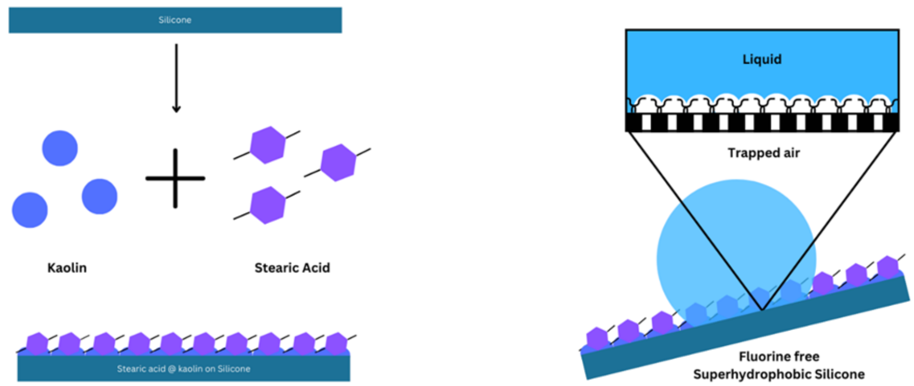

Figure 1. Illustration of the fabrication of fluorine-free superhydrophobic stearic acid modified kaolin nanoparticle.

Preparation of Stearic Acid Modified Kaolin Nanoparticles Silicone Film

Firstly, silicone rubber film was prepared using a 1:1:5 ratio of Elastosil A: Elastosil B: n-heptane. Afterward, 6 g of Elastosil A and Elastosil B been weighed and poured into a beaker. Then, 30 g of n-heptane was added to the beaker. The mixture was stirred with a glass rod and then with a magnetic stirrer until it was completely dissolved. Next, 1 wt% of stearic acid-modified kaolin powder was added to the dissolved solution and stirred continuously until it was completely mixed. The mixture was then poured onto a glass slide and cast to a 0.05 cm width using a casting knife. The mixture was left on the glass slide temporarily and then fabricated by drying in an oven for 2 to 3 hours at a temperature of 50°C [5]. Here, silicone films with different concentrations of the stearic acid-modified kaolin nanoparticles (5 wt%, 10 wt%, 15 wt%, and 20 wt%) were prepared.

Performance testing and characterization

The surface wettability of the sample was evaluated via contact angle method. Using deionized water, the volume of the droplets was 4 μL. A droplet was dropped on the three random points on the sample's surface. The average of the random points was calculated to obtain the contact angle value. standard deviation was then calculated. The surface wettability and physicochemical properties of the composite film were evaluated. FESEM was used to study surface morphological features of the prepared film. AFM was used to analyze and measure the surface topography of the films. FTIR was used to evaluate the functional group of the stearic acid modified kaolin nanoparticles. The graphite powder was used to simulate dust for self-cleaning properties. Before placing the sample in the petri dish, the surface was evenly sprinkled with graphite powder. The angle formed by the sample's surface and the bottom of the petri dish was approximately 10°. The sample's upper surface was then constantly doused with water using the dropper while pictures were being taken [5].

Results and Discussion

Surface Wettability of Stearic Acid modified Kaolin Particles

Surface wettability is a critical aspect of surface characterization for self-cleaning applications. Thereby, water contact angle analysis was carried out on difference samples such as the pristine kaolin, stearic acid-kaolin powder, and stearic acid-kaolin modified silicone film to characterize their surface wettability behaviour. A drop of water was dropped onto three different areas of each sample to determine the surface's contact angle. Figure 2(a-d) shows different water contact angle of stearic acid-kaolin powder prepared with different loading.

A droplet of water readily spreads and penetrates the kaolin powder. It is because the kaolin powder has high surface energy due to the present of hydroxyl groups on its surface, making it interact readily with polar substances like water. Meanwhile the hydrophobicity becomes evidence when the surface of kaolin particles is modified with stearic acid. With stearic acid modification (Kaolin-0.1@SA, Kaolin-0.2@SA, Kaolin-0.3@SA, Kaolin-0.4@SA), the droplets stay on the samples and form almost circle-like droplets. It is because the surface is incapable of forming strong bonds primarily because of hydrophobic nature of stearic acid's long alkyl (hydrocarbon) chain, which resulting in a lower energy return [5].

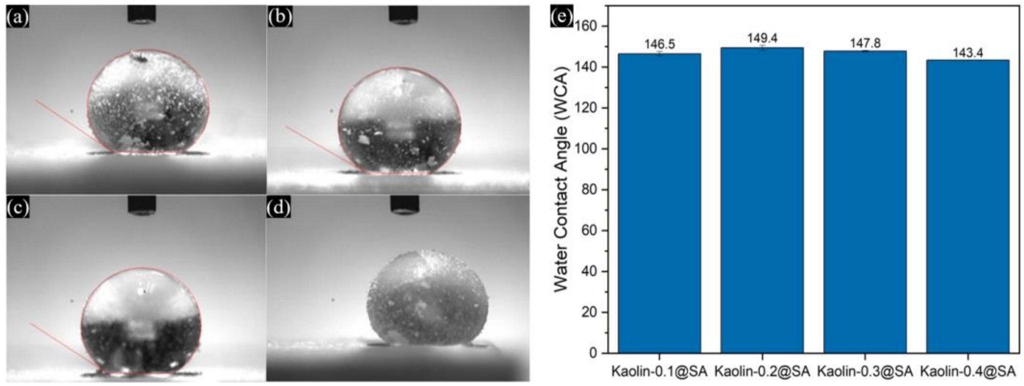

Figure 2. Different wettability across each sample in 1 wt% ethanol-stearic acid solution: (a) Kaolin-0.1@SA, (b) Kaolin-0.2@SA, (c) Kaolin-0.3@SA and (d) Kaolin-0.4@SA. (e) Water contact angle value of different samples

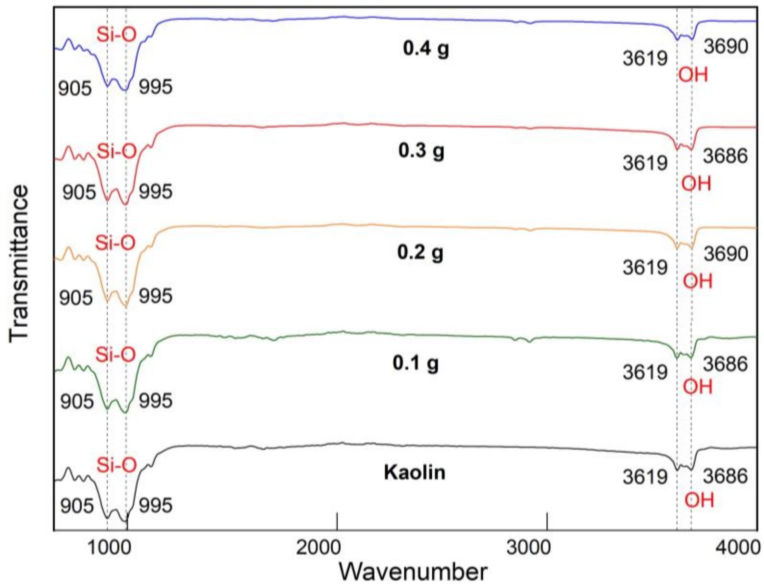

Figure 3. FTIR spectrum of the tested samples of kaolin powder with stearic acid

Modifying kaolin's surface with low surface energy of stearic acid resulted in greater resistance to high surface tension of water from penetrating into the powder [6]. Based on Figure 2(e), Kaolin-0.2@SA has the highest water contact angle value of 149.4° and Kaolin-0.4@SA has the lowest water contact angle value of 143.4°. Nevertheless, each of the samples show no significant difference in water contact angle value. Kaolin-0.3@SA was chosen to further composite with silicone to obtain self-cleaning film because of its higher preparation yield of stearic-kaolin powder in comparison to Kaolin-0.1@SA and Kaolin-0.2@SA, despite all three samples having comparable contact angles, with Kaolin-0.3@SA displaying a contact angle of 147.8°. Theoretically, even though Kaolin-0.4@SA has the highest weight of dispersed kaolin in 1 wt% ethanol-stearic acid solution, but in Figure 2(d), it shows unstable hydrophobicity. that at higher concentrations, stearic acid may not effectively coat the kaolin particles, leading to unstable hydrophobicity. Consequently, the Kaolin-0.4@SA sample was not chosen, instead, the Kaolin-0.3@SA sample was chosen.

Surface Chemistry of Stearic Acid modified Kaolin Particles

To determine whether the kaolin nanoparticles have been modified by the stearic acid, the FTIR analysis was carried out to identify the presence of chemical compounds and functional group in the samples. **Figure 3** shows the FTIR comparison between kaolin powder, Kaolin-0.1@SA, Kaolin-0.2@SA, Kaolin-0.3@SA, and Kaolin-0.4@SA. As can be seen from the pristine kaolin curve, peaks at 3200 to 3700 cm-1 and 830 to 910 cm-1 are ascribed as O-H and Si-O bond stretching vibration, respectively. For the 0.1g kaolin SA-modified kaolin curve, there is only O-H bond stretching presence at the peak with wavelength value 3425 cm-1. Followed by 0.2g kaolin, 0.3g kaolin and 0.4g kaolin, the wavelength values are 3371 cm-1, 3363 cm-1 and 3348 cm-1, respectively with the same O-H bond stretching. The wavelength value decreases when the amount of dispersed kaolin increases. In addition, compared to the pristine kaolin, there are no Si-O bond presence in all SA-modified kaolin samples [7]. It results from interactions between hydroxyl groups on the surface of the samples and destabilisation of the silicon dioxide (Si-O) double bond. The double bonds of the carboxylic group take on partial double bond characteristics when the stearic acid molecule interacts with the kaolin surface, forming a resonance-stabilized carboxylate group [7]. Therefore, the FTIR analysis proven the kaolin nanoparticles have been successfully modified via stearic acid. As shown in **Figure 4(n)**, the FTIR spectrum generates a different wavelength value of each sample's membrane. Almost all the samples show the same functional groups present at the peaks which are O-H and C-O. The difference of each sample is their different wavelength value of each peak. Increasing the concentration of stearic acid-modified kaolin leads to an increasing wavelength value at peaks.

Surface Wettability of Kaolin@Stearic Acid@Silicone Film

The fabrication of self-cleaning silicone film was prepared with different concentration of superhydrophobic stearic acid modified kaolin nanoparticles (1wt%, 5wt%, 10wt%, 15wt% and 20wt%). **Figure 4(a-f)** indicates water contact angle values for various samples, including the pristine film and those containing different concentration of superhydrophobic stearic acid kaolin nanoparticles. There is no significant difference in the wettability of each sample. The pristine membrane is made of inherently hydrophobic silicone. That is the reason why the pristine film is capable to achieve water contact angle exceeding 90°, with a specific water contact angle of 113.2°. The highest water contact angle value recorded is 113.9° at a concentration of 15wt%. Meaning that, none of the aforementioned films achieve the anticipated superhydrophobic surfaces. It could be due to the insufficient surface roughness of the surfaces. Film with 15wt% (SA/K15 film) was chosen as the best surface with hydrophobicity since it exhibits the highest contact angle among other concentrations.

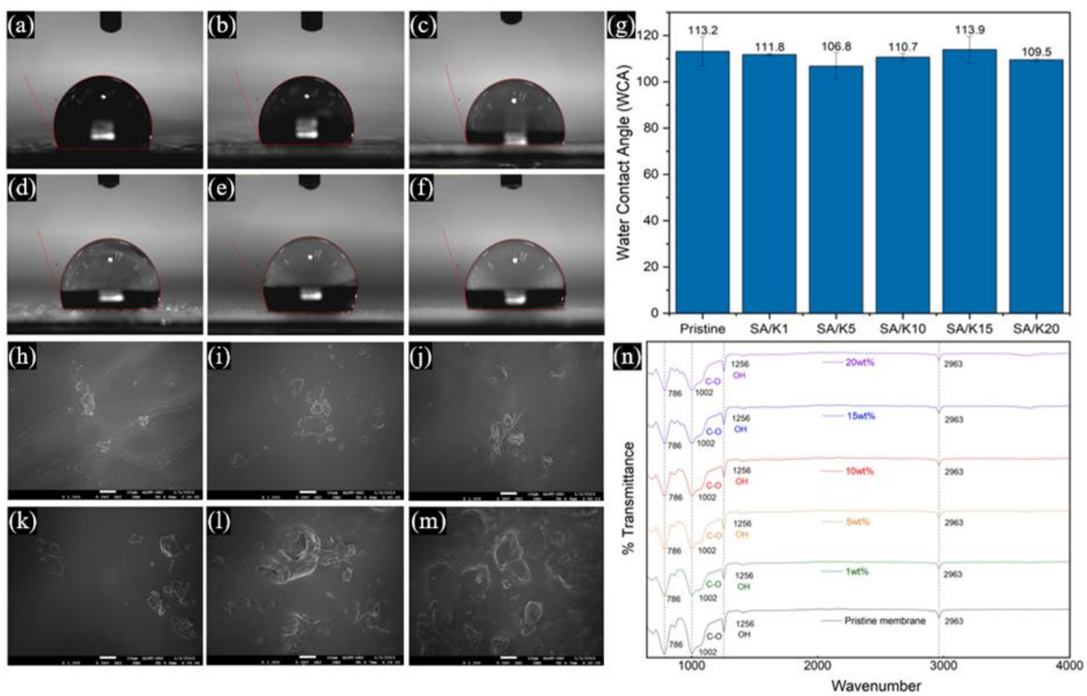

Figure 4. Wettability produced by the samples membrane: (a) Pristine membrane, (b) SA/K1, (c) SA/K5 (d) SA/K10, (e) SA/K15 and (f) SA/K2O. (g) Water contact angle values of the samples. FESEM images of sample membrane: (h) Pristine membrane, (i) SA/K1, (j) SA/K5 (k) SA/K10, (l) SA/K15 and (m) SA/K2O. (n) FTIR spectrum of superhydrophobic silicone rubber surface and pristine membrane

Surface morphology of Kaolin@Stearic Acid@Silicone Film

Surface morphology of the samples was investigated by using Field Emission Scanning Electron Microscopy analysis (FESEM) as shown in Figure 4(h-m). The introduction of superhydrophobic particles results in the induction of surface roughness. The surface exhibits increasing roughness as the particle loading increases, creating valley-like structures (refer to Figure 4(h-m)). The distribution of stearic acid-modified kaolin particles on the film's surface were improved at greater concentration. The distribution of the nanoparticles is well-uniform at concentration of 20 wt% as compared to other concentrations. With higher concentration, more superhydrophobic particles present in the solution, more areas on the film's surface are exposed to the superhydrophobic nanoparticles, forming valley and ridge like structure. This structure promotes slippery surface because the presence of air gaps between the rough surface prevents the droplets from penetrating the gaps.

Surface roughness of Kaolin@Stearic Acid@Silicone Film

Surface roughness is one of the significant parameters in creating self-cleaning surfaces. When surfaces are rougher, they become more slippery and water droplets on top tend to become almost spherical. Atomic Force Microscopy (AFM) used to measure the surface topography by generating high-resolution nanoscale images. In this experiment, silicone films with different concentration of superhydrophobic stearic acid modified kaolin nanoparticles were measured with AFM to discover the roughness of the surfaces. The surface roughness of the films was distinguished with different concentration of stearic acid-kaolin modified nanoparticles (1wt%, 5wt%, 10wt%, 15wt% and 20wt%) as shown in Figure 5. Among all the measured samples, pristine membrane without stearic acid-kaolin modified has the smallest value of roughness which is 1.878 nm. Despite the smooth surface, the inherent hydrophobic nature of silicone is the reason behind the contact angle exceeding 90°. The value of roughness increases as the concentration of stearic acid-kaolin modified nanoparticles increases due to the increase of surface irregularities.

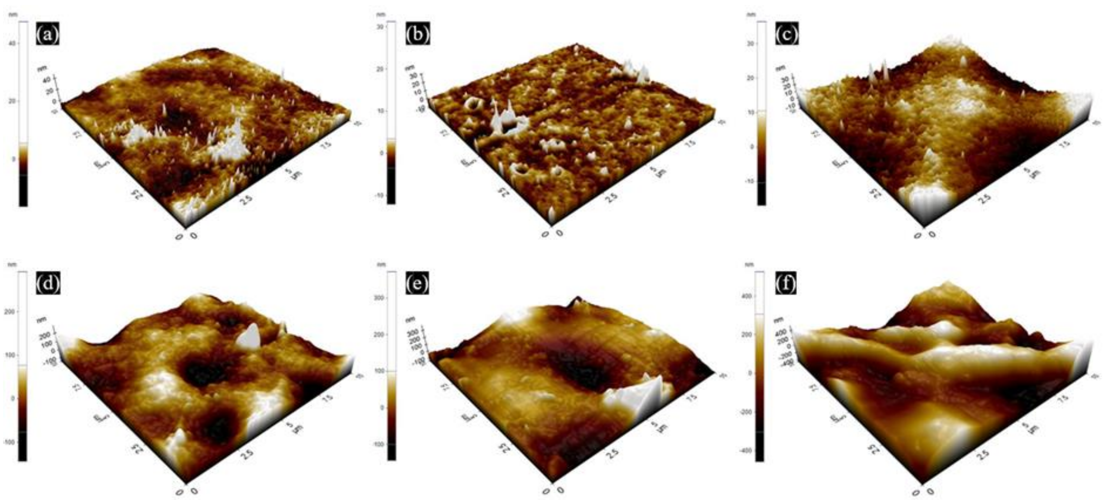

Figure 5. The surface roughness of the films: (a) Pristine membrane, (b) SA/K1, (c) SA/K5 (d) SA/K10, (e) SA/K15 and (f) SA/K2O

Self-cleaning Performance of Kaolin@Stearic Acid@Silicone Film

In this experiment, the self-cleaning effectiveness was evaluated by evenly dispersing contaminant particles (specifically graphite powder) onto the membrane. As shown in **Figure 6**, water droplets entrained dust particles on the surface due to adhesion forces was observed, showcasing the ease with which rolling droplets dragged these particles [11]. The pristine membrane was covered with graphite powder, and the sample was then inclined at an angle of 6° above horizontal followed by applying 30 drops of water. Due to the surface's inherent hydrophobicity, water droplets immediately carried a small amount of graphite powder as they rolled from the top to the bottom of the sample, as seen in **Figure 6(a)**. Under the same conditions, when water droplets were applied to the stearic acid-kaolin modified silicone film covered with graphite powder, the water droplets also rolled down the surfaces. In the process, the droplets effectively removed particles from the surface of the stearic acid-modified kaolin@silicone films, resulting in a more thorough cleansing of the dispersed powder compared to the pristine sample. This demonstrates that the films exhibit superior self-cleaning performance compared to the pristine membrane. The quantity of surface protrusions due to surface irregularities coming from kaolin particles as well as the low surface energy of the stearic acid and silicone rubber promote self-cleaning [10]. As seen in **Figure 6**, a droplet of water rolls down the surface, removing some of the graphite powder. The self-cleaning ability was enhanced with the increased loading of superhydrophobic stearic acid-modified kaolin nanoparticles. The irregularities of the superhydrophobic particles on the silicone film makes the surface rough, entrapping air underneath the drop, which induced self-cleaning abilities. Therefore, the stearic acid-kaolin modified silicone films have proven to have better self-cleaning performance than the pristine membrane.

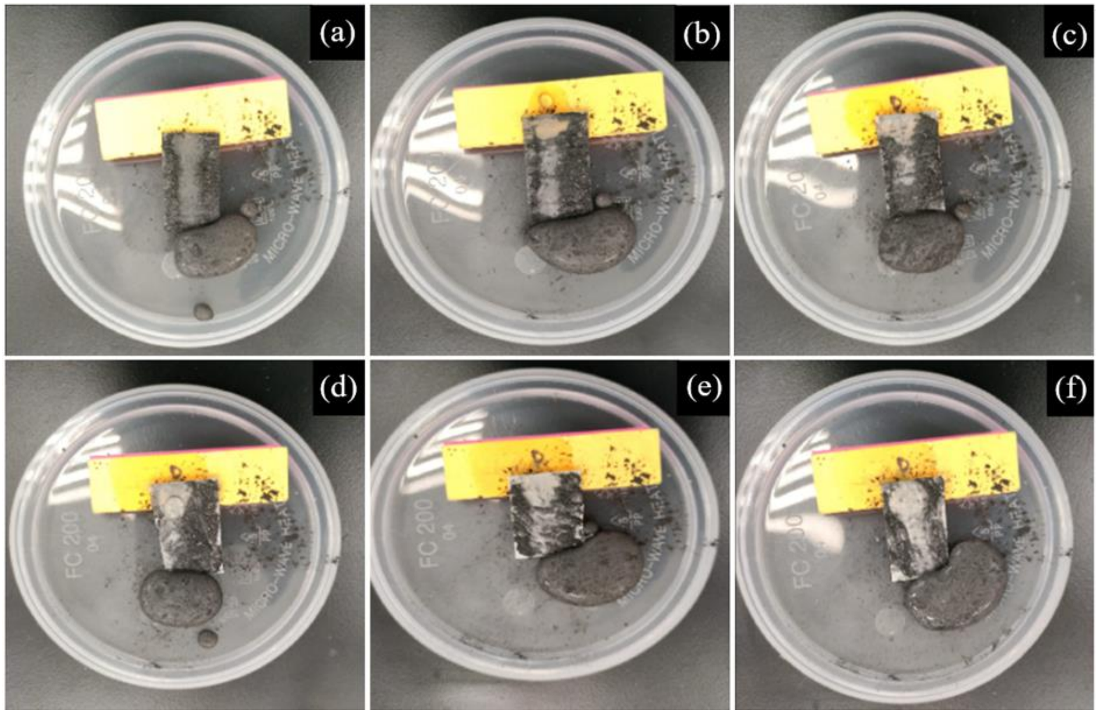

Figure 6. Self-cleaning performance of samples after 30 drops of water dropped: (a) pristine membrane, (b) SA/K1, (c) SA/K5, (d) SA/K10, (e) SA/K15 and (f) SA/K20

Conclusions

In summary, the modification of kaolin nanoparticles using stearic acid was successfully employed to achieve superhydrophobic properties with contact angle of 149.4° ± 1.2. The superhydrophobic particles were then embedded in silicone films to impart self-cleaning properties. The resulting silicone rubber film exhibited rough surfaces. Consequently, the surface displays self-cleaning properties of sample SA/K15 showed as the best configuration of the film with the highest contact angle of 113.9° ± 5.8. Water droplets effortlessly slide down the surface of the silicone film coated with stearic acid-modified kaolin particles, efficiently dislodging any debris adhering to it. To enhance future outcomes, implementing a two-step spray method to ensure an even dispersion of stearic acid-kaolin modified nanoparticles onto the silicone film is recommended.

Conflicts of Interest

The author(s) declare(s) that there is no conflict of interest regarding the publication of this paper.

Acknowledgment

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