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Synthesis and Characterization of a Silica-based Hydrophobic Coating using Sol-Gel Method Applied on Various Substrates

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Abstract: We synthesized and characterized a silica based hydrophobic coat using sol-gel method applied on various substrates. The hydrophobic solution was prepared by mixing tetrahydrofuran (THF), deionized water, and isopropyl alcohol (IPA) with volumetric ratio of 1:3:4 at 60^0 C, addition of 0.01M HCl or NaOH, Polydimethylsiloxane (PDMS) and Tetraethylorthosilicate (TEOS) at different weight ratios, under 7-hr stirring at 60^0-80^0C. The hydrophobic solution was then applied on various substrates, and characterized using scanning electron microscope, image-processing program, and contact goniometry. It was observed that at certain PDMS-TEOS weight ratio, silica-filler addition, and pH-level yielded greater contact angle up to 135^0 and smaller roll-off angle about 25^0-27^0. Surface analysis also correlates the effect of coating to the substrate and its hydrophobicity performance. These results suggest that it is possible to synthesize a better performing silica-based coating that has near-superhydrophobicity characteristics compared to commercial hydrophobic coat.

Key Words: Hydrophobicity; Sol-Gel Method; Nanotechnology; Roughness; Image J

1. Introduction

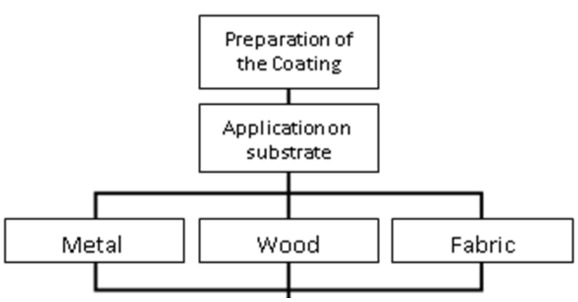
Hydrophobicity is defined as the property of a surface to “repel” water, although the repulsion-like effect is due to the absence of attraction between the water and the surface in contact. Surfaces that have extreme anti-wetting properties are known as superhydrophobic. Superhydrophobic surfaces occur in nature. Much research in recent years has focused on mimicking the “The Lotus Effect” – a natural hydrophobic phenomenon observed in lotus leaves.

Synthesizing coatings with characteristics exhibiting the lotus effect can have many possible applications, from conservation science to structural integrity of various colossal infrastructures exposed to harsh weather conditions in the Philippines.

To provide a practical solution to the ever persistent and pervasive variations in humidity and temperature, the proponents will attempt to design and formulate a silica-based hydrophobic coating applied on various substrates using the sol-gel method, which has previously shown effectivity for preparing thin silica films. In this paper, we examine the effect of silica-fillers, TEOS-PDMS weight ratios, and acid-base nature of the solution on the hydrophobicity performance of the coating by determining contact and roll-off angles and correlating anti-wetting properties to its surface morphology and particle characteristics.

2. METHODOLOGY

Figure 1 below is a framework showing how the synthesis and characterization of hydrophobic coating. The coating was prepared by mixing THF, deionized water, and isopropyl alcohol IPA with volumetric ratio of 1:3:4 at 60^0 C. It was followed by an addition of 0.01M HCl or NaOH. TEOS, with 1:1 molar ratio with THF, and PDMS were added in the solution with 60:40, 70:30, and 80:20 weight ratios respectively. The sol-gel was then applied on various substrates - metal, wood, and fabric. These substrates were characterized using scanning electron microscope for surface morphology, image-processing program for particle characterization, and contact goniometry for determining contact and roll-off angle.



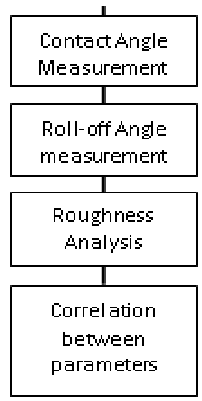


Fig. 1. Flowchart of the experimental work

The roll-off angle measurement setup was provided in figure 2. In the determination of the roll-off angle, the substrates would be placed on a flat surface. This flat surface is to be constructed from a hinged apparatus, where its angle of incline will be varied. A single drop with volume of 0.5 centiliters was delivered by the dropper will be applied on both substrates.

Figure 2. The apparatus used in determining the Roll-Off angle.

3. RESULTS AND DISCUSSION

3.1 Particle Analysis

It was mentioned in the Introduction that silica-fillers, PDMS-TEOS weight ratios, and acid-base nature of the sol-gel may determine the hydrophobicity performance of the silica-based coating. The results were divided into five parts as follows: particle characterization, roughness profile, contact angle, roll-off angle, and correlation of roughness. The processed image used for particle analysis can be seen in Fig. 3 (below). For the metal substrate, there was a higher particle count of size 27-30 microns at 70:30 ratio (w/ and w/o filler) alkaline-induced coating compared to other ratios (see Fig.4).

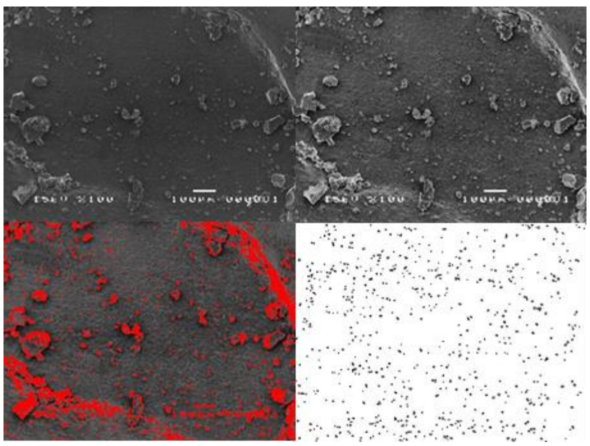


Figure 3. From the top-left: a. Original SEM Image b. contrasted SEM image c. false-colored SEM image d. Processed image showing individual particles.

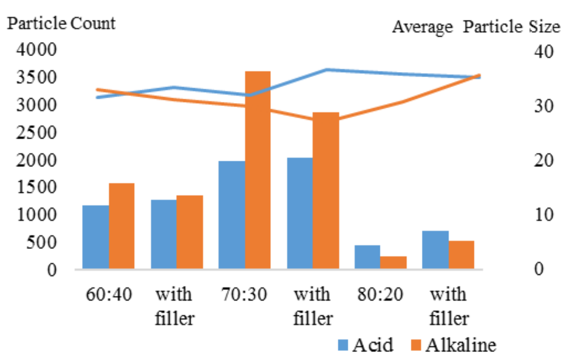


Figure 4. Graph showing particle count (bar graph) and average particle (line graph) size of acid vs alkaline induced coats.

For the wooden substrate, it shows similar particle count and size when compared to the metal substrate. However, Table 1 reports that for the fabric substrate, there was a higher particle count of size 28-29 microns at 70:30 ratio (w/ and w/o filler) acid-induced coating.

Table 1Table 2Results of particle analyses on fabric substrate treated with the acid induced

	pH 2		
Ratio	Count	Total Area (10^{-6} m^2)	Average Size (10^{-6} m^3)
60:40	687	18500.21	26.93
with filler	1234	37393.75	30.30
70:30	1024	29081.25	28.42
with filler	1721	50612.50	29.41
80:20	881	25100.12	28.49
with filler	1491	41143.75	27.60

3.2 Roughness characterization

The roughness profile was illustrated from the SEM images using an image-processing program as displayed in Fig. 5. The images show the difference between the surfaces uncoated substrates. As presented, there was a higher mean roughness profile of coated surface compared to untreated ones. Figure 7 reports the numerical data for the roughness profile.

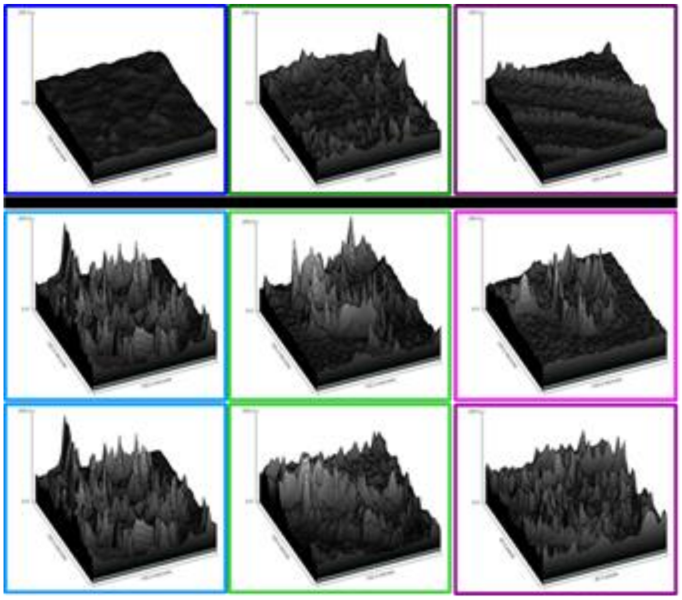


Figure 5. Processed images for mean roughness profile. First row: untreated metal, wood, and fabric substrates. Second row: coated substrates at 70:30 ratio (w/o filler) Third row: coated substrates at 70:30 ratio (w/ filler). For coated metal and wood, the coatings are alkaline induced. For the coated fabric substrate, the box in pink shows the roughness profile for an acid-induced coating and the other one for alkaline-induced coating.

The data suggested that there was a general increase in the Rq and Ra values, with the exception of the 60:40 acidic samples and 70:30 alkaline sample who did not exhibit a significant difference with the addition of the fused silica filler. In terms of the kurtosis, there seems to be a general decrease in its value, suggesting that the addition of the silica of coated and uncoated substrates. As presented, there was a higher mean roughness profile of coated surface compared to untreated ones. Table 2 reports the numerical data for the roughness profile. The data suggested that there was a general increase in the Rq and Ra values, with the exception of the 60:40 acidic samples and 70:30 alkaline sample who did not exhibit a significant difference with the addition of the fused silica filler. In terms of the kurtosis, there seems to be a general decrease in its value, suggesting that the addition of the silica filler induced more ordered structures on the substrate.

pH 12						
Ratio	Rq	Ra	Rku	H.Peak (10^{-6} m)	L. Valley (10^{-6} m)	T.H (10^{-6} m)
60:40	69.37	65.37	2.11	245	-15	260
with filler	74.20	69.45	1.97	245	-17	262
70:30	83.30	78.83	1.74	245	-12	257
with filler	82.87	79.59	1.60	245	-24	269
80:20	73.59	70.86	1.74	245	-26	271
with filler	78.63	76.67	1.52	245	-31	276

Table 2, Results of particle analyses on fabric substrate treated with the acid induced

3.3 Contact Angle Analysis

For the contact angle test, the results were compared to a commercial product, Crep Protect. For the metal, the optimum average contact angle was achieved under the Alkaline induced coating with ratio of 80:20 with added 5wt% fused silica. It achieved an average contact angle of 114.767 °, rivaling the average contact angle of Crep Protect with an average of 111.197°. For the wooden all samples exhibited hydrophobic contact angles with a minimum contact angle of 111.903 ° and a maximum of 135.287 ° for the sol-gel and 141° for the Crep Protect. For the Fabric, the optimum contact angle was achieved through the Acid induced coat with added fused silica. It achieved an average contact angle of 131.743 º degrees, which outperformed the Crep Protect of a contact angle of 117.497 º degrees

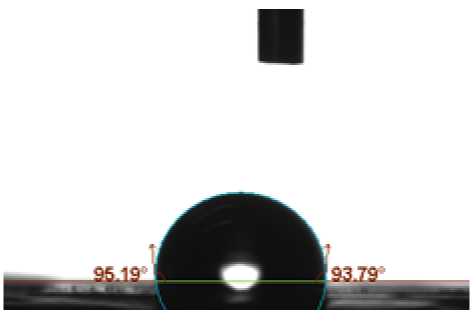


Figure 6. A photograph of the contact angle showing 95.19 and 93.79 degrees. The larger the angle, the better hydrophobicity

3.4 Roll-off Angle Analysis

The roll-off angle characterization revealed the average Roll-Off angles taken from 6 trials. Crep Protect was used as a standard with an average of 44.83. For the Metal, the optimum roll-off angle of an average25.83 degrees was achieved under the Acid induced coat with ratio of 60:40, without the fused silica. For the Wood sample, the optimum was achieved under 2 sample. The Acid induced 70:30 ratio with added fused silica of an average roll-off angle of 34.83 and the Alkaline induced 60:40 ratio without fused silica of an average 85.33. Crep Protect yielded an average roll-off angle of 31 degrees. Lastly for the fabric, the optimum was achieved under the acid induced 80:20 ratio without fused silica filler. It achieved an average roll-off angle of 29.67, which is quite close to Crep Protect of an average roll-off angle of 27.33 degrees.

3.5 Correlation between parameters

There is a significant correlation between wetting properties and arithmetic peaks and valleys (Ra) values. However, these correlations differ for various substrate and pH-level. that for the metal substrate under the acidic coating, that there is a moderate correlation of 0.635 between the contact angle and the corresponding Ra value, however for the Roll-off angle, the data points to a strong correlation value of 0.834 suggesting that the Ra values have a strong influence on the roll-off angles of the metal substrates under alkaline coatings. For wood substrate, there is a weak correlation for all pH-level between the wetting properties and Ra values. Looking at the fabric substrate under the acidic coatings, the data points to a moderately strong correlation between the Ra values and the contact angle, while the roll-off angles exhibited a moderately weak correlation between the Ra values for the acidic coatings.

4. CONCLUSIONS

The paper attempts to synthesize a silica-based hydrophobic coating using the sol-gel method. The sol-gel; with different TEOS-PDMS weight ratios, silica-fillers, and pH-level; was then applied on various substrates - metal, wood, and fabric. In this study, five different investigations were conducted: particle characterization for particle count and mean size, surface morphology for the mean roughness profile (Rq, Ra, Rku), contact goniometry for determining contact and roll-off angles, and solving for correlation between wetting properties and the arithmetic mean of peaks and valleys (Ra) values. We found out that at 70:30 TEOS-PDMS ratio, the hydrophobic performance of the coating exhibits the most. For the said weight ratio, the alkaline-induced coating is the effective one compared to the acid-induced one. There's a significant difference in the hydrophobicity between coating with and without silica-fillers. One result for the contact angle approaches 134^0, showing a near-superhydrophobic effect. In addition, the synthesized coating outperforms the commercial product, therefore indicates the possibility of production that can compete against known brands.

The data gathered provides valid data for the development of hydrophobic coatings and suggests that this method may lead to creating superhydrophobic coatings or even surfaces. For any future studies in the continuation of this research, the researcher recommends the inclusion of various other substrates that may be susceptible to compromise and damage under with the exposer of moisture and water such as concrete, piping, and roofing. The long term effects of such exposure and the long term study of the sol-gel method may also prove significant in the presrvation of such materials and improve the integrity of said materials and contribute to better methods. Aspectc outside of this particlar study's scope and limitation would also include other tests such as the integrity of the coating itself, for long and short term study such as scratch tests, mechanical tests, wash tests, and elemental composition.

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