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**Rafał Kozera[1,2*], Bartłomiej Przybyszewski[1,2], Katarzyna Ziętkowska[2], Anna Łabęda[2], Anna Boczkowska[1,2], Bogna Sztorch[3], Robert E. Przekop[3], Christian W. Karl[4], Monika Pilz[5]**

[1] *Technology Partners Foundation, ul. Bitwy Warszawskiej 7A, 02-366 Warszawa, Poland*
[2] *Faculty of Materials Science and Engineering, Warsaw University of Technology, ul. Woloska 141, 02-507 Warszawa, Poland*
[3] *Centre for Advanced Technologies, Adam Mickiewicz University in Poznan, ul. Uniwersytetu Poznańskiego 10, 61-614, Poznań, Poland*
[4] *SINTEF Industry, Department of Materials and Nanotechnology, Forskningsveien 1, 0373 Oslo, Norway*
[5] *SINTEF Industry, Department of Process Technology, Forskningsveien 1, 0373 Oslo, Norway*
[*] *Correspondence:* rafal.kozera@technologypartners.pl

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# ANTI-ICING AND HYDROPHOBIC PERFORMANCE OF CHEMICALLY MODIFIED WATERBORNE POLYURETHANE HYBRID COATINGS

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**The problem of ice is evident in many industries, such as aviation, transport and energy. The accumulation of ice not only causes monetary losses, but also threatens safety. In the aviation industry, ice build-up leads to changes in aerodynamics as well as damage to parts and sensors. This in turn contributes to emergency landings, flight cancellations, the need to replace parts, and increased energy consumption. Ice build-up can be prevented by using active or passive systems. Due to the cost, time-consuming nature and environmental disadvantages of using active systems, the development of anti-icing coatings is becoming increasingly popular. In this work, an air spraying method was used to fabricate the sample. A waterborne polyurethane paint was applied to aluminum substrates. Modification of these coatings with functionalized organosilicon compounds was done. Compounds with the same core were used. The organosilicon compounds contained functional groups that direct hydro- and icephobic properties of the surface. Roughness, wettability (contact angle and roll-off angle) and ice adhesion force measurements were taken. Lower roll-off angle values were obtained for each of the chemical modifications. The reduction was more than 75%. On this basis, it can be concluded that the hydrophobic properties of the coatings were improved. A decrease in the ice adhesion force values was obtained, which demonstrated the favorable effect of the icephobic properties of the coatings. This reduction for two of the types of modification was more than 50% compared to the reference sample. The results of the roughness, wettability and ice adhesion were also linked, showing what relationships exist between them.**

**Keywords: anti-icing coatings, waterborne polyurethane, polysiloxanes, ice adhesion, chemical modifiers**

## INTRODUCTION

The problem of ice is noticeable in many areas, including the transport and aviation industry. This phenomenon is observed, for example, when ice accumulates on composite structures such as wind turbines, bridges, tanks, elements of energy installations and on airplanes. Ice accumulated on those components can lead to numerous undesirable consequences. It is important to acknowledge that it is usually more beneficial to prevent damage instead of repairing it. Surface engineering facilitates implementing such tactics by improving the properties of the surface of a material, which is known to be more vulnerable than the inside of the element [1]. The enhanced characteristics may include mechanical properties, corrosion and wear resistance, thermal or electrical conductivity and insulation [2, 3] as well as hydrophobicity and icephobicity [4].

Each surface should be designed in accordance to the element's requirements and working environment [5]. The hydrophobicity and icephobicity of a surface are desirable properties in many applications, including aviation [6], insulators [7], bridges [8] and the wind energy sector [9]. The latter industry experiences power losses up of to 30% of output power because of the fact that the materials used for wind turbine blades are not fully resistant to unfavorable conditions the elements operate in [10]. The scale of the loss depends highly on the location of the wind farm and there are attempts to model the turbine's environmental conditions and predict damages [11]. One of the threats is rain induced erosion of the surface [12]. It occurs when there is an impact of a water droplet on an element rotating at high-speed, such as a blade. The main negative effect of the occurrence is damage to the leading edge, which causes dangerous fatigue damage and a decrease in efficiency [13]. What is more, ice accumulation on wind turbine blades may lead to damage as harmful as structural degradation, elevated loads and decreased aerodynamic efficiency. In cases of neglect and hesitation in the removal of ice, the process may not only cause power loss, but also safety issues and even definite stoppage of operation [14, 15].

The roughness of a surface is frequently recognized as a crucial factor affecting its hydrophobic and icephobic properties [16]. The proper design of a surface may allow alteration of the wettability characteristics. Overall, rough surfaces tend to exhibit high wettability contact angles [17, 18], however, in the literature, achieving a "proper" level of roughness is advocated, rather than aiming for it to be extraordinarily high [19, 20]. On the other hand, a decrease in ice adhesion strength is usually observed for smoother surfaces [21, 22].

Low wettability is mainly owing to the presence of air pockets. This can be mainly explained using the Cassie-Baxter model. When the ambient temperature drops below the freezing point of water, water molecules begin to condense on the surface of the nanostructured superhydrophobic surface. These small water droplets, in turn, effectively prevent the formation of air pockets. This can lead to a change in wettability at low temperatures. There are studies in which a group of superhydrophobic coatings was prepared using the sol-gel method [23]. The value of the contact angle grew with increasing the silica content, from 50 to 165° (for 20% by weight of the additive). Wettability was shown to change along with a change in ambient temperature. It was observed that a reduction in the wettability angle was greater for the samples with the higher CA at room temperature (the highest roughness). Moreover, the sample surface at low temperatures exhibited very high adhesion of frozen water droplets. At room temperature, on the other hand, the droplet easily slipped off the surface. The high adhesion of droplets at subzero temperatures was due to wetting of the air pocket surface by moisture condensation. The effect of the higher water/solid contact was greater ice adhesion strength than on the glass substrate to which the coating was not applied. It was recognized that an important issue in the development of icephobic materials is to keep its wettability as low as possible at low temperatures. In other work focused on the wettability of solid surfaces at temperatures below room temperature, the profiles of water droplets on superhydrophobic surfaces were recorded [24]. A modified arrangement of ZnO nanotubes on the surface was performed. It was concluded that at -10°C the value of the contact angle decreases by about 8° compared to the measurement at room temperature. In turn, another paper noted that the surface free energy also changed under the influence of low temperature [25].

Nevertheless, the relation between the hydrophobicity and icephobicity of surfaces has not been clearly established yet. Some studies [7, 26, 27] prove that in many cases hydro- or superhydrophobic materials also exhibit anti-icing properties. On the other hand, there are papers proving that water-repellence does not automatically lead to minimized ice adhesion or an extended freezing delay time [28, 29]. Different approaches to ice accretion on superhydrophobic surfaces are discussed by Lin et al. in review [30], with a conclusion that there is room for investigation of the relation between water-repellency and anti-icing properties in order to develop valuable superhydrophobic and icephobic coatings.

Innovative materials that aim to obtain specific properties, including hydrophobicity, increased mechanical strength, and thermal stability arouse considerable interest both among the scientific community and among industrial specialists. One of the modification possibilities is the introduction of organosilicon compounds, i.e. silanes, polysiloxanes, silsesquioxanes, into the polymer matrix. The main chain of polysiloxanes consists of repeating [-Si-O-Si-] units, which contribute, among others, to the thermal stability and elasticity of the material. The Si-O bond is longer than the C-C bond, and the Si-O-Si bond angle is about 143°, which ensures flexibility of the chain. Adding appropriate functional groups to the side chain makes it possible to modify the properties of polysiloxanes [31]. A properly selected functional group can introduce various changes, such as modifying chemical reactivity, improving mechanical properties, regulating solubility, or controlling thermal stability. For example, alkyl or fluoroalkyl groups can change hydrophobicity, and reactive groups can influence the interactions between the molecule and the polymer.

Feng et al. reported on the synthesis of a series of polysiloxane-modified polyurethanes (Si-PU) using 2,4-toluene diisocyanate (TDI), polydimethylsiloxane with a dihydroxybutyl group (DHPDMS), polytetramethylene glycol (PTMG) and 1,4-butanediol (BDO). The research shows that an increase in the DHPDMS content resulted in an increase in the water contact angle and a decrease in surface tension. Si-PU showed good surface and mechanical properties with a DHPDMS content of 5% [32].

In a study by Wang et al., polyurethane based on polysiloxane was developed by means of chemical copolymerization. Using polydimethylsiloxane with a hydroxyl group-containing two carbamate groups at the ends of the polymer chains, 4,4′-dicyclohexylmethane diisocyanate (HMDI) and 1,4-butanediol, a series of thermoplastic polysiloxane polyurethanes (Si-TPU) were obtained. Si-TPU showed good microphase separation and higher mechanical properties [33].

Santiago et al. described polyurethane-siloxane thermosetting copolymers of various compositions, obtained from a trimer of isophorone diisocyanate, poly(caprolactone) triol and poly(dimethylsiloxane) terminated with a hydroxyl group. These materials were cast onto aluminum surfaces. The conducted research shows that the water contact angle increased with the percentage of siloxane [34].

The aim of this paper is to investigate how the addition of the authors’ chemical modifiers from the group of polymethylhydrosiloxanes influences the properties of waterborne polyurethane coatings dedicated for composite protection. The uniqueness and novelty of this work are the modification of resin with polysiloxanes functionalized with two types of olefins. The reactive group (allyl 2,2,3,3,4,4,5,5-octafluoropentyl ether) is capable of interacting with the polymer matrix and the non-polar groups (hexyl or octyl) are responsible for giving the modified surface a hydrophobic/icephobic character. This is an attractive and innovative approach in the context of polymer matrix modification. The investigated characteristics include roughness, wettability at room temperature and ice adhesion strength.

## MATERIALS AND METHODS

### Chemical modifications

The chemical modification of the waterborne polyurethane coatings was performed with in-house synthesized chemical modifiers from the group of multifunctionalized organosilicon compounds (MOD). In this work, polymethylhydrosiloxanes were used and functionalized with two different functional groups with the same molar ratios. Pure polymethylhydrosiloxane without functional groups was also utilized for comparison purposes.

### Synthesis and analysis of chemical modifiers

The procedure of MOD1/2 wt%, MOD2/2 wt%, MOD3/2 wt%, MOD4/2 wt%, MOD5/2 wt%, MOD6/2 wt% synthesis in accordance with the methodology described in our previous work [35], was used in this work. The obtained products were identical in appearance and spectroscopic characteristics to those obtained in the referenced work. Unmodified polysiloxane (PHD992) was used as the reference sample. Nuclear magnetic resonance (NMR) spectra for $^{1}H$, $^{13}C$, and $^{29}Si$ were conducted at 25°C using Bruker Ascend 400 and Ultra Shield 300 spectrometers. $CDCl_3$ was employed as the solvent, and the chemical shifts are presented in ppm, referencing the residual solvent ($CHCl_3$) signals for $^{1}H$ and $^{13}C$.

## Preparation of samples

The samples used for the investigations were fabricated by adding 2 wt% of one of the chemical modifiers to the waterborne polyurethane matrix. The coatings were deposited by spraying using a pressured gun. The topcoat consisted of Aerowave 5001 base (AkzoNobel), curing solution 6002 (AkzoNobel) and water. The proportions were 100:33:20 by weight.

Each chemical modifier was assigned a letter symbol. The compositions, i.e. core type, functional groups, and their ratio to each other are listed in Table 1.

TABLE 1. **Composition of coatings and chemical modifiers (PHS – polymethylhydrosiloxane, OFP – allyl 2,2,3,3,4,4,5,5-octafluoropentyl ether, HEX-hexyl group, OCT-octyl group)**

| No. | Sample description | Core | Olefin 1 | Olefin 2 | Molar ratio $O_1$:$O_2$ |
|---|---|---|---|---|---|
| 0 | REF | - | - | - | - |
| 1 | MOD1 | PHS 992 | OFP | OCT | 01:04 |
| 2 | MOD2 | PHS 992 | OFP | OCT | 01:02 |
| 3 | MOD3 | PHS 992 | OFP | OCT | 02:02 |
| 4 | MOD4 | PHS 992 | OFP | HEX | 01:04 |
| 5 | MOD5 | PHS 992 | OFP | HEX | 01:02 |
| 6 | MOD6 | PHS 992 | OFP | HEX | 02:02 |

### a. Roughness

The roughness of the sample surfaces was investigated by means of an optical profilometer Sensofar S Lynx in interferometric mode. An objective with magnification of 20x/0.40 was utilized. The Ra parameter describing surface roughness was evaluated. The final values are the average of three different measuring points on the surfaces.

### b. Wettability

The wettability of the fabricated sample surfaces was studied by characterization of the contact angle (WCA) and roll-off angle (RoA), both conducted at room temperature. The sessile drop method was employed with the application of a 5 µl droplet. The values were analyzed by means of a Goniometer OCA15 and SCA software from DataPhysics Instruments (DataPhysics, Filderstadt, Germany). The final WCA and RoA values are the average of five different measuring points on the surfaces.

### c. Ice adhesion

For the ice adhesion (IA) tests, specimens with dimensions 100 x 25 mm were prepared. After the fabrication process, the study materials were mounted in a developed holder, which was then filled with water. For 24 h, the samples were kept at -10°C and afterwards they were mounted in the grips of the testing machine Zwick/Roell Z050. From each material with a different chemical composition, 6 specimens were fabricated. The conditions during the tests were the same, i.e. room temperature and a relative humidity of 50%. The details of the ice adhesion test procedure was described in the previous paper [36]. The final ice adhesion was determined from five measurements.

## RESULTS

In Table 2 are shown the values of the roughness parameters along with their standard deviation values. As can be observed, the values do not change significantly, while the highest values were obtained by the MOD3 and MOD6 samples.

TABLE 2. **Ra values for reference and modified surfaces**

| Sample | Ra [µm] | SD [µm] |
|---|---|---|
| REF | 0.25 | 0.02 |
| MOD1 | 0.17 | 0.04 |
| MOD2 | 0.13 | 0.01 |
| MOD3 | 0.22 | 0.05 |
| MOD4 | 0.13 | 0.03 |
| MOD5 | 0.14 | 0.02 |
| MOD6 | 0.28 | 0.02 |

In Fig. 1 the results of the wettability contact angle measurements are displayed. It can be seen that the most of the results are similar to the reference values, and only the surfaces after modifications MOD3 and MOD6 show a noticeable increase in comparison to the reference samples.

Fig. 2 presents the roll-off angle results for all the modified samples and the reference material. In this case, all the samples exhibited significantly lower roll-off angles with the lowest values recorded for the MOD2 and MOD4 samples, indicating good mobility of droplets on the modified samples and a positive chemical effect of all the applied functional groups. On the other hand, less improvement was recorded for samples MOD3 and MOD6, showing the influence of the 02:02 molar ratio between the functional groups as the least effective in comparison to the other applied molar ratios, despite the increase in their wettability and roughness.

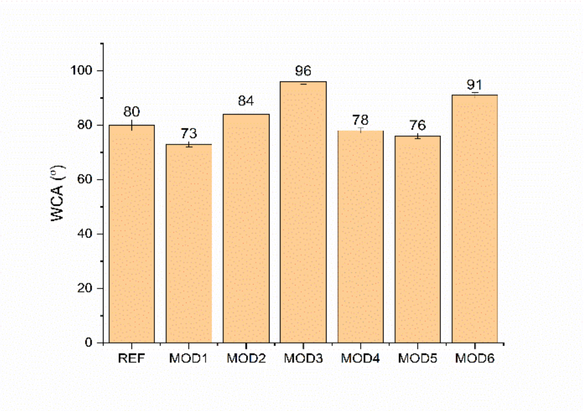

Fig. 1. Results of wettability tests measurements before (REF) and after modifications (MOD1-MOD6)

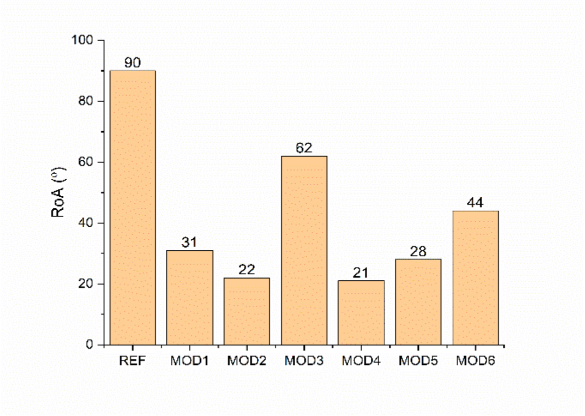

Fig. 2. Results of roll-off angle measurements before (REF) and after modifications (MOD1-MOD6)

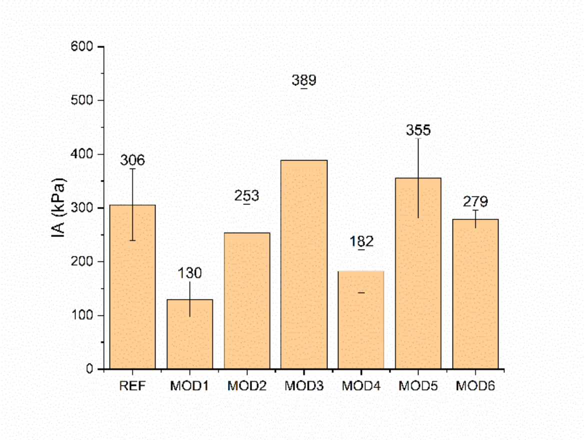

Fig. 3 Results of ice adhesion measurements before (REF) and after modifications (MOD1-MOD6)

Fig. 3 presents the results of the ice adhesion tests. It can be observed that as in the previously presented cases, also in this case, the greatest chemical modifications resulted in a significant drop in the ice adhesion values. In the case of MOD1 and MOD4, the values were lower more than 50% in the case of MOD1 in comparison to the reference sample, i.e. 130 and 182 kPa respectively. The ice adhesion values also indicated that modifications MOD3 and MOD5 not only resulted in the largest values among the tested samples but were also higher than the reference sample (REF).

## DISCUSSION

### Relationships between roughness and wettability/ice adhesion

Surface roughness can play an important role in the coating's ability to achieve hydrophobicity and icephobicity [37]. Figure 4 shows the obtained relationships between these properties for the developed surfaces. In analyzing the graphs, it is not possible to determine direct correlations correctly for all the samples, but only certain trends: as the surface roughness increases, the values of water contact angles (Fig. 4a) and ice adhesion (Fig. 4b) also rise. The lack of clear correlations may be because the Ra values for individual coatings do not differ significantly from one another. Therefore, it is not possible to determine how roughness affects wettability and ice adhesion for the produced surfaces. Nonetheless, it should be noted that, according to the available literature, the lower the roughness of hydrophobic surfaces, the lower the values of ice adhesion [28, 38, 39].

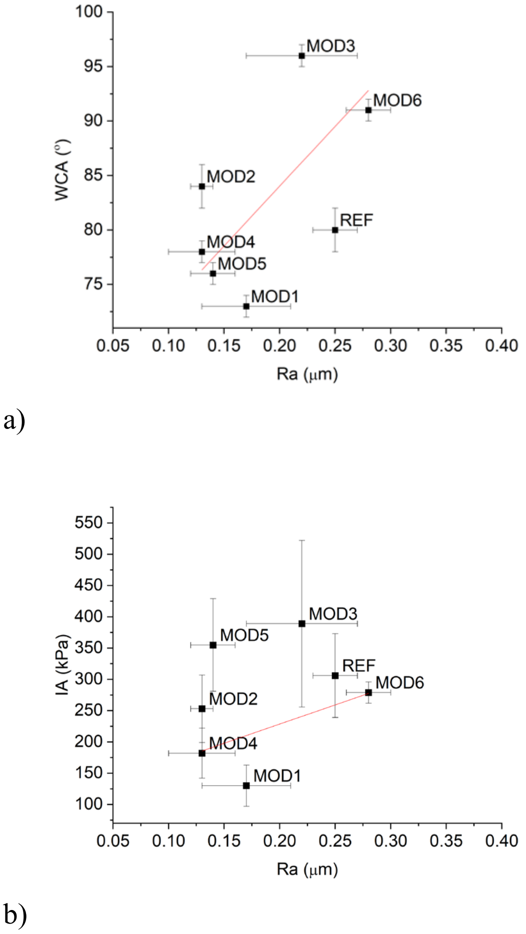

Fig. 4. Relationships of (a) WCA; (b) IA as a function of Ra

A similar trend can be seen in Figure 4b. Therefore, when designing super- and hydrophobic surfaces, it should be kept in mind that frost may be deposited in the surface micro/nanostructure, which increases the contact area between the substrate and the overcooled droplets, and consequently causes ice anchoring. As a result, nucleation velocity and ice adhesion (a decrease in anti-icing performance) increase through the formation of a mechanical connection. Ling et al. [40] demonstrated that ice adhesion on a hierarchical superhydrophobic surface can be up to 67% higher than for a polished copper substrate. In 2023 [41] and in 2024 [42] it was also observed that the highest reduction in IA compared to a reference polymer surface was obtained for the surface with the lowest roughness. Bharathidasan et al. [39], Susoff et al. [28] proved that a smooth, slightly hydrophobic surface can be a potential solution to obtain low ice adhesion values, while surfaces with a developed texture can exhibit low icephobic performance.

## Relationship between wettability and ice adhesion

One method for designing icephobic surfaces is to achieve enhanced water contact angles (WCA) [43-45]. However, it has been proven in recent years that surfaces with WCA < 90° (hydrophilic nature) are also able to record high anti-icing performance, such as low IA values (around 100 kPa) [46]. Therefore, in studies of icephobic surfaces, the focus should be on determining the parameters describing anti-icing properties, not hydrophobic properties. Figure 5 shows the relationship between IA and WCA for the produced samples.

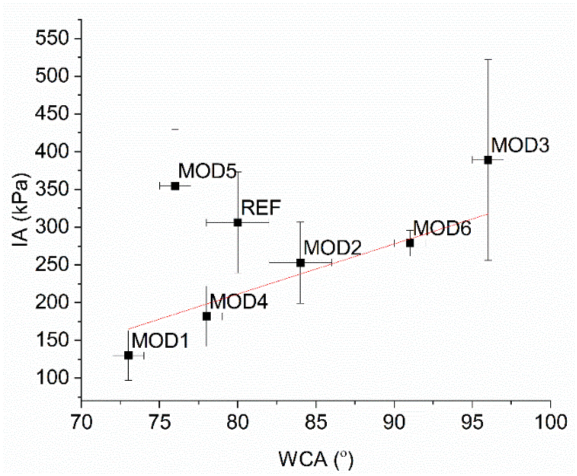

Fig. 5. Relationship of IA as a function of WCA

For most of the developed coatings, excluding the reference sample and the MOD5 sample, there is a correlation that the higher the WCA value, the ice adhesion also increases. The coating with the lowest IA value obtained the lowest WCA value. A similar relationship was observed in work [47]. This proves that the achievement of hydrophobicity is not synonymous with the achievement of icephobicity. According to the available literature, organosilicon compound-based surfaces with reduced ice adhesion (IA reaches values lower than 20 kPa) are characterized by water contact angles of 80–110°, thus icephobic surfaces can be both hydrophilic and hydrophobic surfaces [46].

## CONCLUSIONS

A primer was applied by air spraying, followed by the waterborne polyurethane topcoat. Multifunctionalized organosilicon compounds were used as chemical modifiers. Investigations were conducted to measure the roughness, wettability and ice adhesion strength. The following conclusions were drawn from the tests:

- Roughness: No major differences in surface roughness were observed. The highest values were obtained for the MOD3 and MOD6 samples, in which the molar ratio of OFP:OCT and OFP:HEX was 02:02, respectively.
- WCA: As with the roughness results, no significant differences were observed in the contact angle, with the highest values being obtained for samples MOD3 and MOD6.
- An RoA lower than that of the reference sample was reported for all the samples with the lowest values recorded for the MOD2 and MOD4, indicating good mobility of droplets on the modified samples.
- A decrease in ice adhesion strength was noted for most samples. The largest was obtained for the MOD1 modified sample. It was over 50% compared to the reference sample.
- Listing a single principle that unambiguously links hydrophobicity to icephobicity is a challenging task. The achievement of hydrophobicity is not synonymous with the achievement of icephobicity.

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