

Flame-sprayed polyethylene on polydimethylsiloxane as a composite material with ice-phobic properties

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ABSTRACT

Passive anti-icing coatings are a promising strategy to mitigate ice accumulation without external energy input. In this study, a bilayer composite material combining a soft polydimethylsiloxane (PDMS) elastomer of different thicknesses with a flame-sprayed polyethylene (PE) top layer was developed and evaluated for icephobic performance. Surface morphology, wettability, interlayer adhesion, and micromechanical properties were systematically characterized. Ice adhesion strength was measured using both push-test and centrifugal adhesion test methods under controlled icing conditions.

The results show that the icephobic performance of the composites strongly depends on the thickness of the PDMS sublayer. While thin PDMS layer provide limited improvement compared to PE alone, composite with thicker PDMS layer significantly reduces ice adhesion, outperforming both pure PDMS and pure PE coatings. Accelerated aging led to partial degradation; nevertheless, the composite maintained low ice adhesion and superior performance over single-layer systems.

These findings demonstrate that PE–PDMS bilayer architectures offer a robust and tuneable approach for designing durable, passive anti-icing surfaces.

1. Introduction

Surface icing is a major concern for several applications, from naval efficiency [1] to aircraft safety [2,3] and the resilience of energy system [4,5]. The accumulation phenomena of snow and ice on surfaces result from a complex interplay of environmental parameters such as temperature, humidity, wind speed, and the chemical-physical properties of the substrate [6–8]. In the absence of a universal icing mitigation strategy, research efforts are focused on the development of diverse solutions aimed at both delaying accumulation and facilitating its detachment from the surface [9,10]. Passive anti-icing coatings represent a promising approach to face this problem without additional energy consumption.

Among the different coatings under development, superhydrophobic [11,12], lubricated [13], polymeric and elastomeric [14] surfaces are the most studied and promising approaches to reduce ice adhesion. Superhydrophobic coatings typically combine surface texturing with low-energy layers to minimize wettability and easing water removal.

Many works deal with their effectiveness as anti-icing solutions in different fields, generally revealing a pronounced hindering of freezing phenomena [15,16]. However, several studies have shown that their effectiveness can vary highly depending on variables such as micro/nano scale patterning or environmental conditions during icing/deicing [17–19]. Furthermore, in terms of long-term durability, the formation and detachment of ice on these surfaces may induce progressive wear of the surface texturing, thereby compromising their icephobic performance [20].

Lubricated coatings reduce ice adhesion through the presence of a lubricating fluid layer that hinders the formation of a stable bond between the ice and the substrate. The lubricant is typically confined within a porous layer beneath the surface, which must exhibit chemical affinity with the lubricant to ensure stable confinement and prevent its expulsion from the coating. Extremely low ice adhesions have been reported for lubricated coatings (< 10 kPa) [21]. However, their primary limitation is the progressive depletion of the lubricant [22], which is exacerbated by repeated icing/de-icing cycles.

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Polymeric coatings act through different mechanisms depending on their chemical-physical nature [23] but in general their low surface energy reduces ice adhesion facilitating its detachment under minimal mechanical force. For this reason, long-carbon-chain polymers are considered effective candidates for anti-icing coatings. A notable example is polyethylene (PE), which exhibits an intrinsically low surface energy (30–35 mN/m), imparting hydrophobicity and helping to reduce ice adhesion [24,25]. In addition to these surface properties, PE is inexpensive, chemically stable, and exhibits excellent mechanical performance. Furthermore, its melting temperature ($T_{\text{melt}} = 128\text{ }^{\circ}\text{C}$) makes it particularly suitable as a feedstock powder for the deposition of coatings via thermal spraying [25]. All these features make PE a highly versatile material, suitable for the creation of anti-icing coatings on various types and shapes of surfaces [26–28].

Another important class of polymers used for anti-icing applications is that of elastomers [29,30]. The mechanism of ice detachment is related to their mechanical properties, in particular to the low elastic modulus and hardness, which enable surface deformation under minimal external loads [31,32]. Indeed, when ice adheres to an elastomer, the application of a shear or normal force induces an interfacial fracture at the ice–surface boundary [33]. This fracture propagates efficiently across the interface due to the low elastic modulus of the elastomer, thereby facilitating ice detachment. The key mechanism underlying this behaviour is the ability of the soft elastomer to deform locally, generating stress concentrations that initiate and drive the propagation of interfacial cracks. This process significantly reduces the effective adhesion strength between the ice and the substrate, and the ice shedding occurs through elastic energy release [34].

A commonly used elastomer is polydimethylsiloxane (PDMS) [35,36] which represents a promising anti-icing material not only because of its elasticity but, thanks to the presence of methyl groups along the siloxane backbone, it also shows an intrinsic hydrophobic nature. PDMS is commercially available as Sylgard™ 184 formulation, a bicomponent kit where a vinyl terminated prepolymer (component A) and a cross-linking agent containing Si–H functional groups (component B) are mixed to obtain a non-toxic, easy to use and stable siloxane materials [37]. By fine tuning the mix ratio between A and B component, the curing temperatures and times it is possible to change the elasticity of the final materials and, consequently, the anti-icing properties [38–40]. In a previous work [41] we deeply studied Sylgard™ 184 formulation to find the ideal parameters to obtain best anti-icing properties, resulting in a PDMS coating prepared with A:B ratio of 30:1. This elastomeric coating evidenced a very high anti-icing performance thanks to low hardness which facilitate the detachment of the ice from the surface. However, the high concentration of prepolymer results in a highly adhesive coating surface which leads to a reduced anti-icing performance compared to what would be expected based on its elastic modulus. Thus, although the coating already exhibited excellent anti-icing properties, further improvements are still possible.

Starting from our findings, this study aims to present a possible solution to enhance the icephobicity of PDMS through composite materials obtained by applying a PE layer onto a soft PDMS elastomer via thermal spraying. The resulting PE@PDMS composite material seeks to overcome the intrinsic stickiness of the bare elastomeric surface, while maintaining, or even enhancing, its hydrophobic behaviour. The anti-icing performance of the composite is systematically investigated, revealing improved performances, depending on the thickness of the elastomer, compared to the individual non-combined PDMS and PE materials.

2. Experimental

2.1. Materials and sample preparation

Aluminium alloy (6082 and 6061) flat samples ($50 \times 50 \times 2\text{ mm}$ and $30 \times 60 \times 2\text{ mm}$) were used as substrates. The aluminium plates were

sandblasted and cleaned with acetone before their use.

Sylgard184® (Syl184) was purchased from DowSil™.

Low-density polyethylene (LDPE) powder was purchased from Plascoat® Europe BV with an average particle size of $150\text{ }\mu\text{m}$, as given by the powder supplier.

The PDMS samples were prepared by mixing the prepolymer and cross-linking agent in 30:1 weight ratio. After stirring for 30 min, PDMS was degassed for at least 1 h in vacuum and then cast on the aluminium substrate to obtain different thickness coating using molds. Molds made of PMMA, with dimensions of either $30 \times 60 \times 5\text{ mm}$ or $50 \times 50 \times 5\text{ mm}$, were used based on the size of the aluminium substrate. The samples were cured in oven at $100\text{ }^{\circ}\text{C}$ for 1 h, obtaining elastomeric coatings of about 1 mm (pdms_1) and 5 mm (pdms_5) thickness. Once cured, the samples were kept in a dry box.

PE@PDMS composite materials were prepared by flame spray deposition of a PE layer on pdms_1 and pdms_5, generating the samples pe@pdms_1 and pe@pdms_5 respectively. Flame spray process (Casto-Dyn DS 8000, Castolin) was automatically operated with a robot (ABB IRB 4400/60). An oxygen-acetylene flame was used with the gas pressure of 4 bar and 0,7 bar, respectively. The optimized parameters used are detailed in Table 1, giving rise to a homogeneous PE coating of about 1 mm of thickness. The coated samples were then allowed to cool to room temperature and stored in a desiccator.

For a comparison purpose, a 1 mm thickness layer of PE was directly deposited on grit-blasted (Al_2O_3 grits, Mesh 36) aluminium alloy substrate via flame spraying (PE_FS).

A PDMS layer of 2.5 mm of thickness (pdms_2.5) and the respective composite material (pe@pdms_2.5) was also prepared and the relevant results are reported in the Supplementary Material.

2.2. Characterization

The static water contact angle (SCA) measurements were carried out using the DSA30 and DSA100 (Krüss, Hamburg, Germany) with the method of the sessile drop, using a $5\text{ }\mu\text{L}$ volume of water, at $23 \pm 2\text{ }^{\circ}\text{C}$ and a relative humidity (RH) of $50 \pm 5\%$. The measurements were replicated at least five times for each sample. Dynamic contact angles and the resulting hysteresis (H) and roll-off angle (RO) measurements were performed at $23 \pm 2\text{ }^{\circ}\text{C}$ and RH of $50 \pm 5\%$ with the tilting plate method using a $25\text{ }\mu\text{L}$ volume of water. Five replicates were performed on each sample. For SCA, H and RO average and standard deviation of data are reported.

The optical microscopy images were collected using metallographic Nikon EPIPHOT 30.

The Atomic Force Microscope (AFM) images were collected with Nanosurf Pure Drive microscope in wave mode acquisition analysis. The images were processed by the free software Gwiddion with the following correction: plane level, polynomial background subtraction, fix zero value, align raws by polynomial method.

Surface roughness parameters (R_a and R_q) were calculated from AFM images by averaging ten line-profiles per map. The FT-IR characterizations were conducted with the FT-IR Alpha 1 (Bruker) spectrometer with ATR apparatus (diamond crystal was used as internal reflection element).

Adhesion of the PE layer on composite materials was evaluated following the ASTM-D3359 Standard Test Methods for Rating Adhesion by Tape Test. Basically the test consists in scratching the surface with a

Table 1
Experimental conditions for flame spraying of PE.

Spray distance (mm)	300
Traverse speed (mm/s)	500
Pre-heating temperature ($^{\circ}\text{C}$)	100
Number of layers	4
Post-heating temperature ($^{\circ}\text{C}$)	200

specific scratcher, peeling off the coating with a tape characterized by a certified adhesive force and visually evaluating the adhesion of the coating to the substrate after the test performed.

Mechanical properties of PE and composite materials were measured by instrumented indentation using a FISCHERSCOPE® HM2000 equipped with a Vickers indenter. Tests were carried out using the following parameters:

- Maximum applied load ($F_{\max}$) 600.000 mN applied over 60 s, following a loading law with constant $d\sqrt{F}/dt$.
- Creep time: 60.0 s at maximum load.
- Unloading curve: Same as the loading segment (symmetric unloading).
- Method for tangent fitting: Power Law Method applied to the unloading curve.
- Load range used for fitting: 20.00%–100.00% of $F_{\max}$, with Epsilon = 0.75.

With this technique, the indenter is pressed into the material under a continuously increasing load and then unloaded, while the corresponding indentation depth is recorded in real time. From the geometry of the indenter and the depth of penetration, the Martens Hardness (HM) can be derived. Additionally, nanoindentation provides other key parameters such as creep displacement and the Indentation Modulus (EI), which can be related to the Young's Modulus by accounting for the Poisson's ratio of the material. It also allows evaluation of the elastoplastic response of the surface, by measuring the following energy contributions:

W_e : Energy dissipated during deformation (plastic/viscous work).

W_r : Elastic energy recovered upon unloading.

W_t : Total energy required to indent the material, calculated as.

$$W_t = W_e + W_r. \quad (1)$$

Ice adhesion was evaluated measuring the force needed to detach ice from the coated surfaces respect to a reference material. The measurements were carried out by means of two different equipment and applying two different methods. The first is the push-test method by using a homemade apparatus described elsewhere [28,42]. During this analysis the ice was formed on the surface of the samples in a $10 \times 10 \times 10$ mm mold filled with deionized water cooled at -15°C for 2 h. The sample surface was kept at a constant temperature by means of a Peltier cell, and the experiment was carried out in less than 30 s. A mechanical probe, moved with a constant speed of 0.3 mm/s, detached the ice from the surface by applying a tangent force. Thanks to a load cell connected to the probe, the peak force F needed to detach the ice from the sample was registered. The ice adhesion strength (τ) in shear values is calculated as:

$$\tau = \frac{F}{A} \quad (2)$$

where A is the surface of the specimen in contact with the ice.

The second method used to evaluate the ice adhesion is the Centrifugal ice Adhesion test (CAT) [28] at Tampere University. To carry out this experiment the ice is grown on the specimen surfaces inside an "icing wind tunnel" a system capable of producing various types of ice with different LWC (Liquid Water Content) and droplet size by adjusting parameters such as flow velocity, water supply rate, pressure, and temperature. Once the ice growth phase is completed, the samples are left overnight in the chamber. In these experiments, the temperature of -10°C , droplet speed of 25 m/s and ice type of mixed-glaze ice was used. Iced area was 30×30 mm with the ice thickness ~ 10 mm. After this period the specimens are fixed on a metal bar inside a centrifuge equipment. The bar is then rotated with constant acceleration until ice detachment occurs. The bar is weighed before and after the test to measure the mass of ice removed (m). The maximum centrifugal force

can be measured as follows

$$F_{\text{centrif}} = m\omega^2 = lwh\rho\omega^2 \quad (3)$$

where l , w , h and ρ are respectively the length, the width, the height, and the density of the ice cube, r is the radius of the bar and ω is the rotation speed at the time of detachment. The shear stress (τ) is estimated as the ratio between the centrifugal force (F_{centrif}) calculated at the time of ice detachment and the ice-solid interfacial area (A) using the following equation

$$\tau = \frac{F_{\text{centrif}}}{A} \quad (4)$$

With push-test method a total of four tests on four different specimens for each sample were performed, while with CAT one test was conducted on each of three parallel specimens per sample. Average values and standard deviations are reported.

Accelerated aging treatments on samples were performed by applying the procedure described in Supplementary Material. Briefly, the samples were placed in a climatic chamber and tested on four different stages:

- Stage A: thermal cycling from -30°C to $+80^\circ\text{C}$ for 24 h.
- Stage B: UV radiation exposure for 71 h.
- Stages C: wide-spectrum lamp irradiation for 23 h.
- Stage D: spraying with demineralized water for 23 h.

3. Results and discussion

3.1. Morphological evaluation

The composite materials feature a homogeneously distributed undulated PE layer with a smooth tactile finish, completely covering the underlying PDMS coating, as shown in Fig. 1. Although some black spots can be observed at the PE/PDMS interface, these do not indicate polymer interpenetration. They are due to small plastic residues that adhered laterally to the elastomeric coating during the thermal spraying deposition process [43].

The surface morphology of the samples investigated using optical microscopy reveals an irregular structure, attributed to the impact of molten microparticles during the thermal spraying process. Fig. 2 shows a representative image of pe@pdms_1 , where it is evident that the surface is not perfectly smooth, but rather characterized by depressions and superficial defects. The same features were observed for PE_FS reference and for the other composites.

AFM images of the composite coatings show a surface morphology independent of the PDMS layer thickness. Representative AFM maps of pe@pdms_1 are reported in Fig. 3 (additional optical and atom force microscopy images for the other samples and for the PE_FS are provided in the Supplementary Material). AFM analysis reveals the presence of a densely packed distribution of spheroidal agglomerates across the surface (Fig. 3a). The observed agglomerates exhibit lateral dimensions up to approximately $5\ \mu\text{m}$ and are composed of smaller nanometre-scale substructures with poorly defined shape (Fig. 3b), resulting in an overall irregular surface texture (Fig. 3c).

Surface roughness analysis confirms this qualitative observation; as summarized in Table 2, the R_a and R_q of the PE_FS reference and of the composite coatings remain very similar, with variations within the experimental standard deviation. This demonstrates that neither the presence nor the thickness of the PDMS sub-layer significantly influences the surface roughness of the composite coating, confirming that the surface morphology of the composites is governed exclusively by the flame-sprayed polyethylene layer.

Since the PE layer did not penetrate the elastomeric matrix but coated the surface, forming a sandwich-like composite structure, it is essential to evaluate the adhesion between the two materials. For this

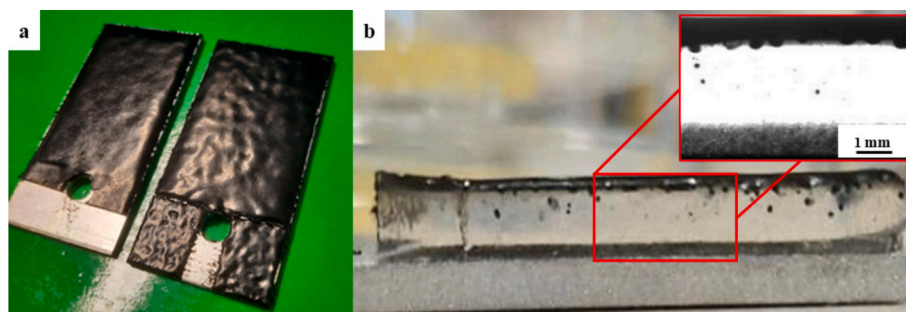


Fig. 1. Pe@pdms samples. a) Top view of pe@pdms_1. b) Lateral view of pe@pdms_5, highlighting the difference in the thickness of PE layer compared to the underlying PDMS.

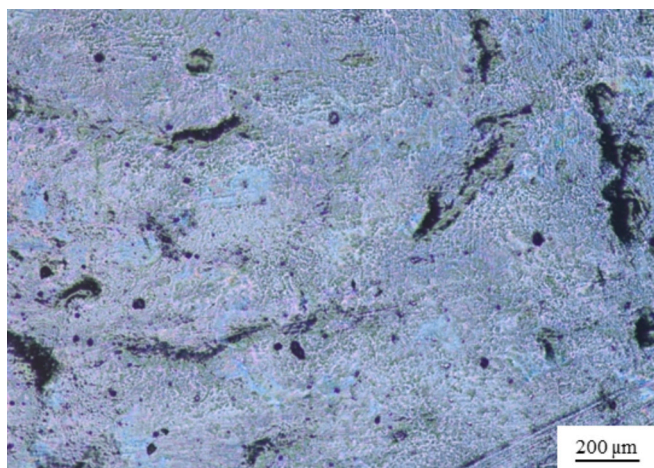


Fig. 2. Optical-microscope image of pe@pdms_1.

purpose, a tape test was performed as described in the Experimental Section. As shown in Fig. 4a, the PE layer remains strongly adhered to the PDMS surface even after the removal of the adhesive tape, with no evidence of delamination or damage. In Fig. 4b, we can also observe that the scratches penetrated down to the underlying elastomer layer, as indicated by the bright areas within the black cross. Although some PE material was removed, as evidenced by residues on the tape, the coating remains firmly adhered to the surface despite the mechanical stress.

The strong adhesion observed between PDMS and PE can be investigated analysing the characteristic of the elastomeric layer. Preparing PDMS coatings with an excess of prepolymer (30:1 ratio) led to a sticky surface, also explained by the accumulation of unreacted vinyl siloxane groups [41]. These free pendant chains are both chemically reactive and physically deformable, enabling multiple adhesion mechanisms. Firstly, at deposition temperatures ($\sim 200^\circ\text{C}$), PE undergoes mild thermal degradation, thus, generating radicals that react with vinyl groups of PDMS, resulting in covalent PE–PDMS bonds at the interface. This mechanism is consistent with the findings of Gent and Ahagon [44], who demonstrated that adhesion between rubber and vinyl-functionalized substrates increases with the surface density of vinyl groups, due to radical-mediated grafting during curing. More recent studies [45,46] confirm that radical grafting of vinyl-functionalized silanes onto PE is effective at similar temperatures ($\sim 200\text{--}220^\circ\text{C}$), although in those cases the reaction is initiated by organic peroxides during reactive extrusion. In the present study, although no initiators are added, the thermal and mechanical conditions of the spray process (high temperature, impact, and possible oxidative environment) could be sufficient to generate radicals on PE chains, enabling grafting reactions. In addition, physical mechanisms such as chain interdiffusion, capillary wetting, and mechanical interlocking — facilitated by the low crosslink density and soft

nature of the under-cured PDMS — likely contribute synergistically to the overall adhesion. These effects, originating from the presence of mobile surface oligomers, enhance interfacial contact and promote entanglement at the PE–PDMS interface [47].

3.2. Microhardness and mechanical properties

In our previous work [41] hardness values were obtained using a Shore A durometer, which is suitable for measuring the viscoelastic properties of gels or soft materials like PDMS, but not for plastics. Since PE is a plastic material and the pe@pdms samples are composed of a soft elastomeric bulk covered by a rigid PE layer, a more reliable mechanical characterization of these complex structures was carried out using the nanoindentation tester [48,49]. This technique provides access to key mechanical parameters, including Martens Hardness (H_M), which quantifies the material resistance to penetration by accounting for the reversible elastic component as well as any irreversible deformation, whether due to true plasticity or time-dependent viscoelastic effects. The Indentation Modulus (E_t) represents the local elastic stiffness and indicates how resistant the material is to reversible deformation under load. The technique measures creep displacement, capturing the time-dependent deformation under constant load. Nanoindentation also enables assessment of the elasto-plastic response via the energy balance during indentation, namely the energy dissipated during deformation (W_e), the elastic energy recovered (W_r), and the total indentation energy ($W_t = W_e + W_r$). The results are summarized in Table 3:

As expected, PE_FS exhibit the highest values of H_M and E_t , together with the lowest creep and energy-related parameters, indicating a relatively stiff, dimensionally stable material. Introducing a 1 mm thick PDMS sub-layer causes a strong reduction of both H_M and E_t , reflecting the lower elastic stiffness and viscoelastic nature of PDMS. Increasing the PDMS thickness to 5 mm does not result in significant changes in H_M and E_t compared to the thin hybrid material.

The elastic recovery ratio W_r/W_t is the fraction of energy recovered during unloading compared to the total indentation energy. PE_FS exhibits a value of 0.51, whereas the composites range between 0.34 and 0.35, showing a comparable response to each other. This indicates that PE has a higher fraction of elastic recovery, while composites dissipate more energy during mechanical loading, leading to viscoelastic-type deformation. The partition of elastic and dissipated energy during indentation is summarized in Fig. 5.

Creep provides information on the viscoelastic dissipation mechanisms that cannot be captured by the purely thermodynamic parameters. Again, the trend is confirmed: pe@pdms_5 exhibits a value more than 1.5 higher with respects to pe@pdms_1 and about 3 times higher with respect to PE_FS. The higher creep observed in pe@pdms composites indicates a larger contribution of viscoelastic relaxation, in contrast to the more dimensionally stable behaviour of PE. These results highlight that the presence and the thickness of the PDMS sub-layer predominantly govern the viscoelastic contribution to the overall mechanical response, while the PE coating primarily influences the plastic

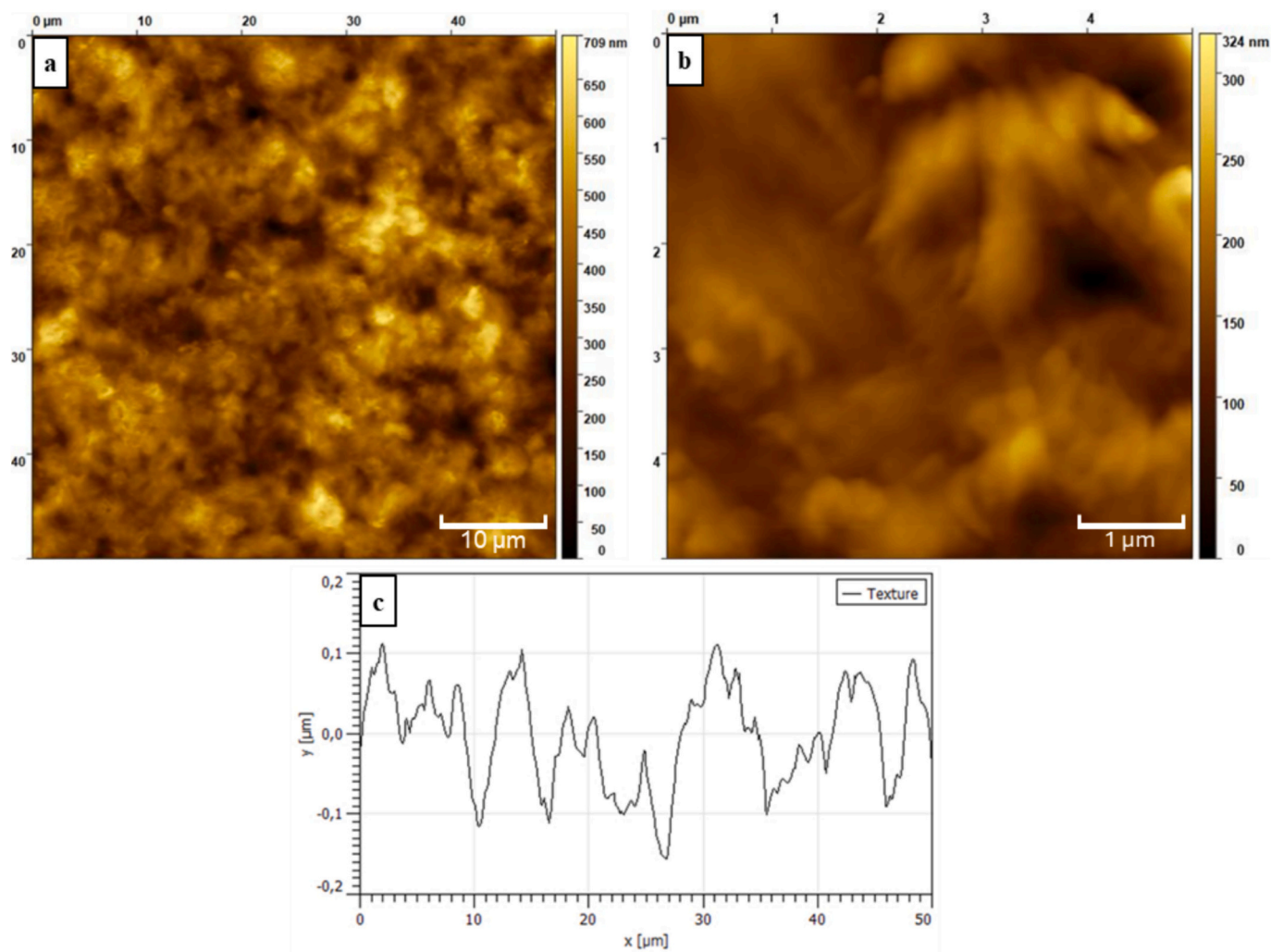


Fig. 3. AFM maps of pe@pdms_1 sample a) 50 × 50 μm area and b) 5x5μm area. c) surface texture.

Table 2

Arithmetic mean roughness (R_a) and root mean square roughness (R_q) of the PE reference and composite coatings.

Sample	R_a (nm)	R_q (nm)
PE_FS	83 ± 21	100 ± 24
pe@pdms_1	67 ± 11	82 ± 13
pe@pdms_5	70 ± 14	87 ± 16

Table 3

Micromechanical properties of PE_FS and composite materials.

Sample	HM (N/mm ²)	E_t (GPa)	Creep (μm)	W_c (μJ)	W_r (μJ)	W_t (μJ)	W_r/W_t
PE_FS	15.55	0.25	3.67	3.76	3.89	7.65	0.51
pe@pdms_1	4.74	0.06	6.64	9.75	5.03	14.78	0.34
pe@pdms_5	5.15	0.05	10.98	10.71	5.79	16.50	0.35

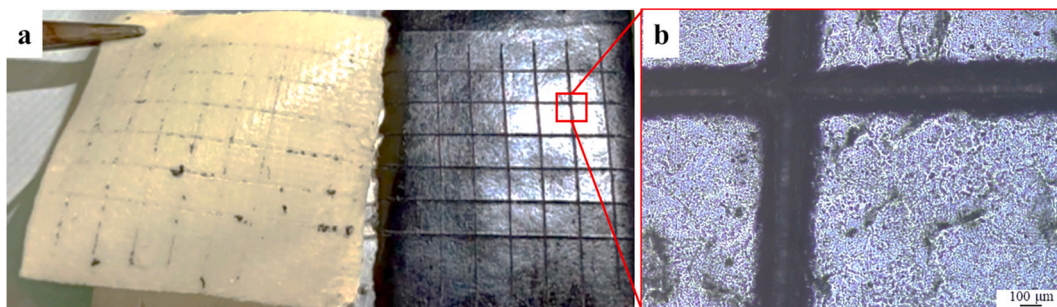


Fig. 4. a) Scratched pe@pdms_1 sample after tape test. b) Optical image of pe@pdms_1 highlighting the scratched area. The grey region represents the PE layer, while the thick black cross lines indicate the scratch. Within the lines, brighter areas, corresponding to the underlying elastomer, are visible.

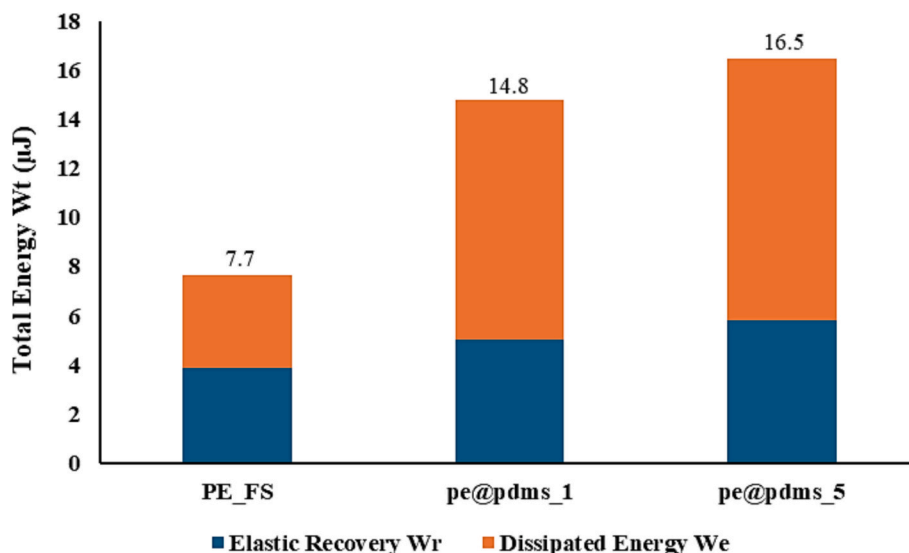


Fig. 5. Partition of elastic recovery energy (W_r) and dissipated energy (W_e) on the total energy (W_t) during nanoindentation of PE_FS and pe@pdms composites.

component captured by HM.

3.3. Wettability

As previously observed [41], PDMS in 30:1 ratio exhibits a relatively high static contact angle, being approximately 127° . However, the low water droplets mobility on the surface does not allow the measurement of H and RO with the tilting plate method (dash in Table 4). This effect is attributed to the high elasticity of the material that contributes to local deformations under the droplet, raising the droplet meniscus and altering the apparent contact line. This leads to an increased static contact angle due to capillary pressure, as described by Style et al. [50,51] At the same time, these deformations can also promote droplet pinning, thereby influencing the wettability.

PE showed a lower static contact angle ($SCA = 100^\circ$) being however characterized by a remarkable mobility of the droplet on the surface (approximately $H = 27^\circ$ and $RO = 38^\circ$). As expected, the composite materials evidenced the same hydrophobic behaviour of PE with only small differences basically within the standard deviation (Table 4). This indicates that the surface wettability of the composites is governed exclusively by the outer PE layer, confirming that the PE and PDMS layers remain structurally and functionally distinct rather than interpenetrated.

3.4. Anti-icing properties

According to Chaudhury and Kim [31], the ability of elastomeric coatings to facilitate ice removal can be described, for a generic rigid slab, by a model that strongly depends on the physico-chemical properties of the material. In this model, the shear stress (τ) required to detach a rigid body from a polymer surface is directly proportional to the shear modulus (μ) and the work of adhesion (W_a), and inversely proportional to the coating thickness (h), following the relation:

$$\tau \propto \sqrt{\frac{\mu \cdot W_a}{h}} \quad (5)$$

The correlation with the shear modulus, which is related to the elastic modulus through the Poisson's coefficient, indicates that the detachment process depends on the mechanical properties of the material. The work of adhesion is the energy required to overcome the chemical and physical interactions between the two interfacing surfaces, and it is linked to the water droplet mobility on the surface [52]. Consequently, the shear stress does not depend solely on the properties of the polymer coating but also on the nature of the counter surface [33]. In the case of ice or snow, additional chemical interactions, such as hydrogen bonding, or physical interactions arising from surface roughness and defects can influence the adhesion.

Since the icephobicity assessment can be also influenced by the experimental set-up of the analysis [42], the anti-icing properties of composite samples, pe@pdms_1 and pe@pdms_5, were evaluated using both *push* and *centrifugal test*. These two techniques were chosen to examine material responses under different icing conditions and mechanical strain.

To quantify the effect of polymer multilayer architectures on ice-adhesion strength, both tests were performed on the two composite samples and compared with PE_FS and with the corresponding bare PDMS layers (pdms_1 and pdms_5).

3.5. Push-test

Ice adhesion results with the push-test method are reported in Fig. 6.

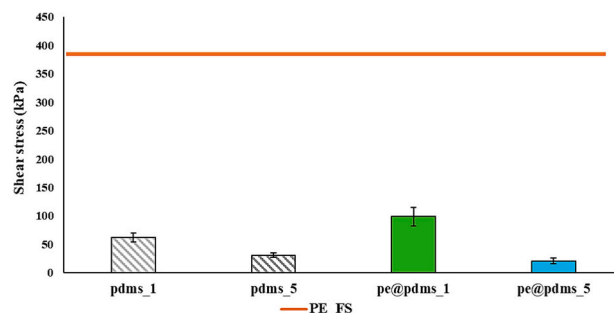


Fig. 6. Anti-icing properties of pdms_1, pdms_5 and composite materials. Orange line represent PE_FS used as reference.

Table 4

Wettability measurements for the composite materials, the bare pdms and the flame-sprayed polyethylene (FS_PE) are reported.

	pdms_1	pdms_5	PE_FS	pe@pdms_1	pe@pdms_5
SCA ($^\circ$)	126.7 ± 0.4	125.6 ± 0.8	100.9 ± 1.3	99.1 ± 1.1	98.7 ± 0.6
H ($^\circ$)	–	–	27.4 ± 2.0	33.7 ± 2.5	30.6 ± 2.4
RO ($^\circ$)	–	–	38.5 ± 2.2	40.7 ± 1.8	38.8 ± 0.5

The shear stress measured for PE_FS is approximately 380 kPa. Both *pdms_1* and *pdms_5* show lower ice adhesion with values strongly influenced by coating thickness: a reduction of nearly 50% is observed when increasing the thickness of PDMS from 1 mm (*pdms_1*) to 5 mm (*pdms_5*). Although the ice-composites interface is identical to that of ice-PE_FS, the shear stress values for composites materials are remarkably lower than those observed for pure PE_FS. As in PDMS, composites display a thickness-dependent response; however significant differences between elastomers and composite samples suggest the coexistence of both mechanical and surface-related contributions.

Micromechanical properties (Table 3) provide the first key to interpreting these trends. PE_FS shows high Martens Hardness ($HM \approx 15.5$ GPa), a relatively high indentation modulus ($E_I \approx 0.25$ GPa), low creep (≈ 3.6 μm) and a large elastic recovery ratio ($W_r/W_t \approx 0.5$ GPa), confirming its rigid and predominantly elastic character. The introduction of a PDMS sub-layer drastically alters the mechanical response: both *pe@pdms_1* and *pe@pdms_5* exhibit a strong decrease in HM (≈ 4.7 and 5.2 GPa, respectively) and E_I (≈ 0.05 and 0.06 GPa) with a remarkable increase in creep (≈ 6.6 and 11 μm), reflecting a more deformable and dissipative structure, enabling stress relaxation and interfacial cavitation during ice detachment [34]. These micromechanical mechanisms effectively reduce the stress required to remove the ice layer in push-test experiments, even though PDMS is not directly exposed at the surface (Fig. 7).

Within the composites, an increase in PDMS thickness further enhances these effects, leading to a reduction of the shear stress. The PDMS-to-PE thickness ratio emerges as the key parameter in optimizing anti-icing behaviour. Thus, *pe@pdms_1*, composed of two layers of equal thickness, exhibits lower icephobicity than pure *pdms_1*, indicating that in this configuration the stiffness of the PE layer dominates over the elasticity of PDMS. Nonetheless, *pe@pdms_1* performs significantly better than pure PE, highlighting that the soft PDMS sub-layer still positively contributes to its anti-icing behaviour, even if partially constrained by the rigid PE shell. In contrast, *pe@pdms_5* exhibits a drastically reduced shear stress, outperforming that of *pdms_5*. Based on its mechanical parameters (Table 3), this composite undergoes pronounced local deformation under strain, enabling effective energy dissipation and reducing interfacial ice adhesion. In these thicker configurations, the influence of the mechanical rigidity of PE becomes negligible, and the overall response is governed by the thick PDMS layer. The trend is confirmed by the results obtained for *pe@pdms_2.5* (see Supplementary Material).

It is however worth asking why the ice adhesion of *pe@pdms_5* is even lower than that of *pdms_5*. If the mechanism were governed solely by PDMS thickness, *pdms_5* would be expected to exhibit the same anti-icing performance as the *pe@pdms_5* composite. The observed deviation confirms that mechanical properties alone does not fully describe the adhesion mechanisms on elastomeric surfaces. PDMS prepared at a 30:1

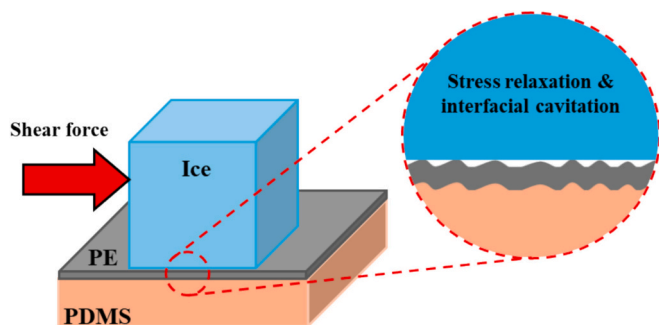


Fig. 7. Schematic comparison of ice detachment mechanisms on *pe@pdms* composites. The deformable elastomeric sub-layer (orange) enables local stress relaxation and promotes interfacial cavitation with a reduction in the stress required for ice removal.

ratio is known from the literature to exhibit pronounced surface tackiness [41] and we observed a negligible droplet mobility (see Wettability Section). These properties influence interfacial adhesion, giving rise to a high W_a towards ice [53,54]. At the same time, the PE outer layer provides a non-tacky surface with lower W_a that avoids the adhesive behaviour typical of high-ratio PDMS. Thus, the combination of soft and thick PDMS layer and a mechanically stable, non-adhesive PE interface yields the most favourable anti-icing performance.

Overall, these results show that the anti-icing performance of PE-PDMS multilayers is governed by a balance between bulk mechanical and surface characteristics. Thin PDMS layers introduce some degree of stress relaxation but are constrained by the intrinsic stiffness of PE. In contrast, thicker PDMS layers enable an efficient deformation-driven debonding mechanism while benefiting from the non-tacky surface of the PE coating, achieving optimal ice-release behaviour.

3.6. Centrifugal adhesion test (CAT)

To confirm the observed icephobic trend under different conditions, the samples were also tested with centrifugal adhesion test (CAT). In Fig. 8 are reported the ice adhesion strengths (in shear mode) for composites compared with pure PE and PDMS.

PE_FS, also with this technique, exhibited the highest ice adhesion strength values, while the composites showed reduced ice adhesion, with *pe@pdms_5* consistently outperforming both PE and PDMS.

It should be noted that absolute numerical values differ between techniques, reflecting intrinsic differences in experimental setups, ice type, sample size, and loading conditions [42]. The push-test applies a localized mechanical displacement, whereas the CAT exerts centrifugal forces across the entire ice-substrate interface. Despite the differences in the absolute values obtained with the two techniques, the overall trend is preserved across testing conditions, except for *pe@pdms_1* which shows slightly lower values than *pdms_1* in the CAT testing. This agreement validates the reliability of the measured anti-icing behaviour and supports the conclusions drawn regarding the performance of the composites.

3.7. Surface properties and ice-adhesion

In Table 5 are summarized the results of morphological and micro-mechanical analysis, as well as the wettability and icephobicity measurements.

The ice adhesion of PE-PDMS composites can be rationalized by considering surface morphology, mechanical response and wettability. Regarding morphology and roughness, optical and atomic force microscopy analysis reveal a comparable morphology of the PE top layer for *pe@pdms_1* and *pe@pdms_5*, indicating that surface texturing plays a secondary role in governing the adhesion trends.

According to the observed mechanical properties the introduction of

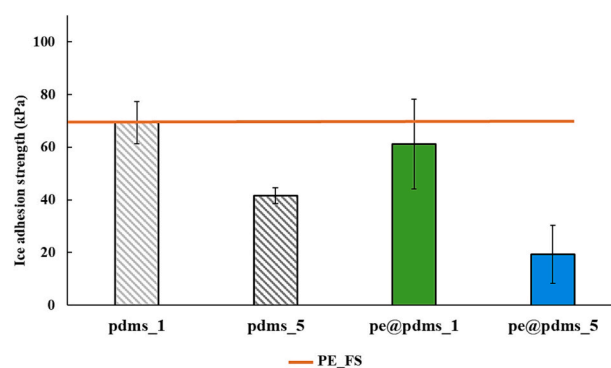


Fig. 8. Ice adhesion strength values from Centrifugal Adhesion Test (CAT) for *pe@pdms* composites compared with pure PE_FS and PDMS.

Table 5

Summary of the main properties of composite samples and reference (PE_FS). % red is the percentage of ice adhesion reduction respect to PE_FS.

Features	Samples		
	PE_FS	pe@pdms_1	pe@pdms_5
HM (N/mm ²)	15.55	4.74	5.15
E _t (GPa)	0.25	0.06	0.05
Creep (μm)	3.67	6.64	10.98
W _r /W _t	0.51	0.34	0.35
Thickness pe/pdms (mm)	1/0	1/1	1/5
R _a (nm)	83 ± 21	67 ± 11	70 ± 14
R _q (nm)	100 ± 24	82 ± 13	87 ± 16
SCA (°)	100.9 ± 1.3	99.1 ± 1.1	98.7 ± 0.6
H (°)	27.4 ± 2.0	33.7 ± 2.5	30.6 ± 2.4
RO (°)	38.5 ± 2.2	40.7 ± 1.8	38.8 ± 0.5
Shear stress (push test), %red Push-test	385.5 ± 73.8	98.7 ± 16.4	20.7 ± 5.2
Shear stress (CAT)	69 ± 7.0	61 ± 17	19 ± 11
%red CAT	–	11.6	72.5

a PDMS sub-layer beneath PE leads to a strong reduction in elastic modulus and a marked increase in viscoelastic dissipation (creep), which promote stress relaxation and interfacial cavitation during ice detachment. Increasing PDMS thickness from 1 to 5 mm amplifies these effects, consistently lowering the measured shear stress.

Regarding wettability, PE outer layer exhibits a certain degree of water mobility upon tilting, whereas water droplets remain pinned on the surface of bare PDMS. Consequently, even if the receding contact angle (θ_{rec}) was not measurable through tilting plate method, systematically lower values of θ_{rec} are to be expected on PDMS with respect to PE.

A commonly accepted [14,55,56] equation correlating θ_{rec} to W_a is:

$$W_a \approx \gamma_w (1 + \cos \theta_{rec}) \quad (6)$$

where γ_w is the surface tension of water. According to this equation higher θ_{rec} leads to lower W_a .

Thus, according to Eqs. (5) and (6), the synergistic combination of a low shear modulus provided by PDMS layer and a low W_a at the PE interface, explains the enhanced icephobicity of pe@pdms_5 compared to bare PDMS of comparable thickness and to PE.

3.8. Accelerated aging test (AC)

The composite materials pe@pdms were subjected to accelerated aging in environmental chambers (AC) to evaluate their durability. This procedure simulates prolonged outdoor exposure under harsh environmental conditions [41,57]. The samples were subsequently analysed by optical microscopy and compared with untreated references to identify

possible surface modifications. Fig. 9 reports the optical micrographs of a representative pe@pdms_1 sample before (a) and after (b) aging. The untreated surface appears relatively homogeneous, with a compact texture and only minor irregularities, whereas the aged sample clearly exhibits multiple cracks and elongated scratch-like features, indicative of mechanical degradation induced by accelerated weathering. Since the same behaviour is consistently observed across all tested composites, further data relating to other samples are provided in the Supplementary Material.

FT-IR ATR spectra were collected on the composite samples. Fig. 10 shows the spectrum of pe@pdms_1, representative of all pe@pdms composites, since the layers are simply stacked rather than interpenetrated. As a result, the spectral features are essentially identical across the composites. No significant differences or unexpected signals were detected, indicating the absence of chemical degradation. The only exception was a broad band above 3000 cm⁻¹ after AC, which was attributed to surface water uptake (orange spectrum in Fig. 10).

Wettability was assessed after the AC test, before and after a washing step, and compared with the values recorded before aging. Fig. 11 reports the results for pe@pdms composites and pure PE. All samples exhibit a degradation of the hydrophobic properties, as evidenced by decreasing static contact angles and increasing roll-off and hysteresis angles. Among the tested materials, polyethylene shows the most pronounced deterioration, with dynamic properties significantly compromised: the water contact angle decreased to ~90°, while contact angle hysteresis and roll-off angles markedly increased. The composites also displayed a deterioration in wettability, with RO and H angles raising up to 60° and static contact angle stabilizing around 90°, consistent with a weakly hydrophilic surface.

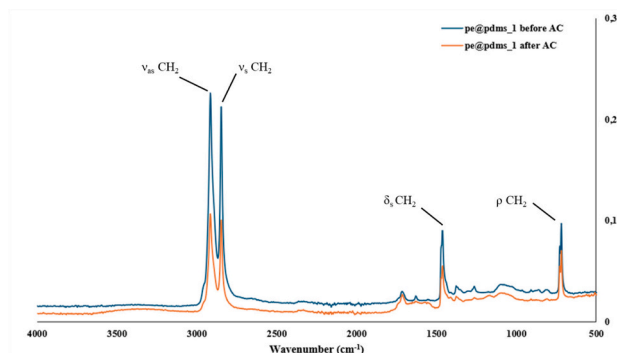


Fig. 10. FTIR-ATR of pe@pdms coating before and after the AC test. C–H stretching: 2915 cm⁻¹ (ν_{as} CH₂), 2847 cm⁻¹ (ν_s CH₂). C–H bending: 1461 cm⁻¹ (δ_s CH₂), 718 cm⁻¹ (ρ CH₂). Above 3000 cm⁻¹, water physisorbed on the surface.

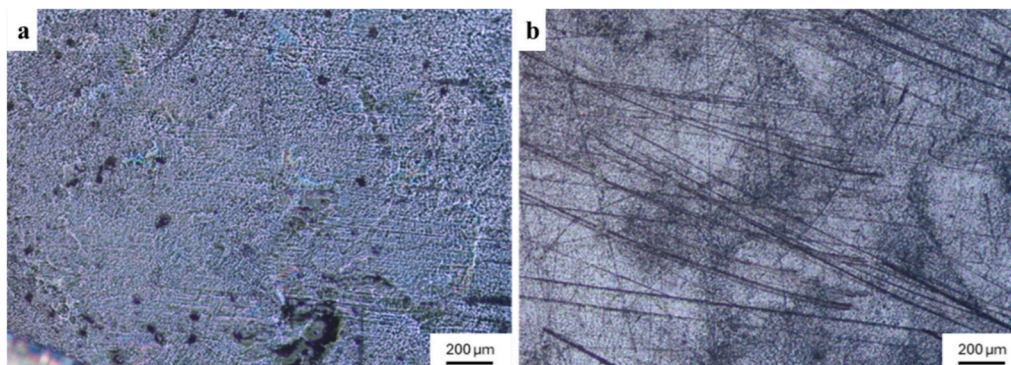


Fig. 9. Optical micrographs of a representative pe@pdms_1 sample before (a) and after (b) accelerated aging in environmental chambers. The untreated surface appears relatively homogeneous, with a compact texture and only minor irregularities, whereas the aged sample shows multiple cracks and elongated scratch-like features, indicative of mechanical degradation and embrittlement of the PE layer under prolonged exposure.

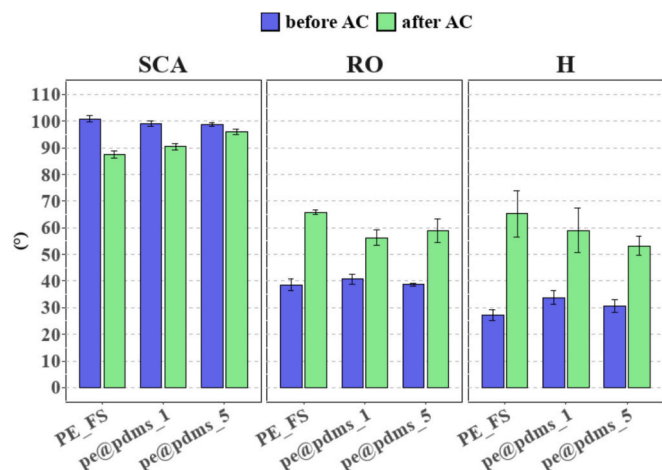


Fig. 11. Wettability of pe@pdms composites and PE_FS before and after accelerated aging (AC) and subsequent washing. Aging reduces hydrophobicity in all samples, with PE_FS showing the most pronounced degradation (SCA < 90°), transition to hydrophilic, while composites retain slightly higher contact angles (> 90°) and lower hysteresis/roll-off values.

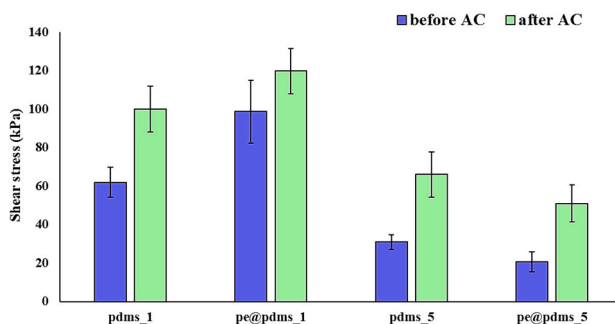


Fig. 12. Shear stress trough push-test after aging test (green bars) compared to pristine sample (purple bars). PE_FS before AC = 385 ± 74 kPa, PE_FS after AC = 518 ± 89 kPa. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

The shear stress values measured with push-test after aging test are reported in Fig. 12. The push-test was chosen because of the lower data dispersion observed with respect to CAT, especially on composite coatings.

Following the aging treatment, all the tested samples exhibited a specific deterioration of their anti-icing properties. For PE the shear stress value reaches 518 kPa likely because of the worsening of the surface homogeneity due to the embrittlement and to the partial loss of dynamic hydrophobic properties. For the PDMS samples (pdms_1 and pdms_5) shear stress increases owing to the increase of elastic modulus following aging, as observed in a previous study [41].

For the composite coatings, the observed deterioration of the icephobic performance is thus attributed to the combined effect of the increased wettability of the surfaces (reduce SCA and increased H and RO) as well as the increase of elastic modulus. A greater relative increase is observed in pe@pdms_5 compared to pe@pdms_1, which might be due to the more substantial contribution of the increased elastic modulus of the underlying PDMS layer to the overall ice adhesion.

Despite the deterioration of anti-icing properties, the pdms@pe_5 sample retains relatively good performance compared to both the pristine state and the reference without PE (pdms_5). Its shear stress values remain lower than those of pure PE, indicating that the bilayer structure contributes to mitigate the effects of surface degradation and loss of hydrophobicity.

Moreover, it is worth noting that after AC, the composites exhibited

no visible signs of detachment between the PE and PDMS layers. This observation confirms that interfacial adhesion remains stable and effective even after aging, indicating that the applied conditioning protocol does not compromise the structural integrity of the PE–PDMS interface. Such stability further supports the reliability of the composite system under tested aging conditions.

4. Conclusions

Bi-layer composite materials were fabricated through a rapid, cost-effective, and scalable two-step process, in which a casted PDMS layer was coated by a PE layer applied through thermal spraying. The resulting pe@pdms composites were systematically evaluated for their anti-icing performance and compared to the individual components.

The results demonstrate that the icephobicity of the composites is strongly influenced by the thickness of the PDMS layer. When the PDMS is thin, the composite exhibits better performance than PE alone but worse than that of PDMS. As the PDMS thickness increases, the elastic contribution becomes dominant, mitigating the rigidity of the PE layer and leading to enhanced ice detachment. In these configurations, the dynamic wettability imparted by PE becomes more effective, resulting in superior anti-icing properties compared to the pure components PE and PDMS. These findings highlight that a clear synergistic effect can only be appreciated when the PE layer is significantly thinner than that of PDMS (e.g., fivefold lower thickness), like in pe@pdms_5, where the beneficial contribution of the layered architecture is maximized. Notably, the Centrifugal Adhesion Tests generally agreed with the results obtained from the push-tests, showing consistency across different experimental methodologies. The findings confirm that the intrinsic stickiness of soft elastomers like PDMS can increase the adhesion between ice and surface. The application of a hydrophobic top layer with appreciable roll-off and hysteresis angles, such as PE, introduces a functional synergy: the soft substrate enables interfacial stress release, while the rigid, low-energy coating reduces ice–surface adhesion. This layered design thus represents a rational and innovative approach to decouple and optimize the mechanical and surface contributions to icephobicity.

Aging tests confirmed the robustness of the composite architecture: although both hydrophobicity and elasticity were partially lost, the composites maintained excellent anti-icing performance, with shear stress values below 100 kPa. Pe@pdms_5 preserved favourable anti-icing behaviour even after prolonged exposure, outperforming the uncoated reference.

This study highlights the critical importance of identifying the dominant mechanism – mechanical deformation versus surface characteristics – under specific structural conditions. The ability to tune the balance between these effects by adjusting the layer architecture offers a powerful strategy for designing next-generation anti-icing surfaces.

The obtained results support the potential of pe@pdms systems as effective and durable passive anti-icing coatings. The key challenge for the industrial scale-up of these materials is the development of approaches that retain their icephobic performances at micrometric thicknesses. Such an advancement would significantly reduce practical barriers to industrial applications, including cost, added weight, and fabrication constraints.

CRediT authorship contribution statement

Alessandro Casali: Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Marcella Balordi:** Writing – original draft, Supervision, Resources, Project administration, Funding acquisition. **Heli Koivuluoto:** Writing – review & editing, Supervision, Resources, Project administration, Methodology, Investigation, Conceptualization. **Paolo Gadia:** Methodology, Investigation, Data curation. **Giorgio Santucci de Magistris:** Writing – review & editing, Visualization, Data curation. **Paolo Pelagatti:** Writing – review & editing, Supervision,

Resources, Project administration, Funding acquisition.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Alessandro Casali reports financial support, administrative support, article publishing charges, and travel were provided by Italian Ministry of the Environment and Energy Safety. Alessandro Casali reports financial support, administrative support, equipment, drugs, or supplies, and travel were provided by Italian Ministry of Education and Merit. Alessandro Casali reports a relationship with Ricerca Sul Sistema Energetico RSE SPA that includes: non-financial support. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.porgcoat.2026.110283>.

Data availability

Data will be made available on request.

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