

Article

Anti-Icing Performance of Base Formulations for Coatings for Aeronautical Applications

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Abstract

Icing on surfaces presents a significant challenge across various technological fields, with added weight and operational interference impacting aircraft wings, wind turbine blades, and electrical equipment. While electrothermal methods effectively accelerate ice melting through the Joule effect, their substantial cost and energy consumption necessitate careful consideration. In the past two decades, anti-icing and icephobic coatings have garnered significant interest due to advancements in nanomaterials and a deeper understanding of ice nucleation and adhesion. These coatings are designed to prevent water droplet adhesion, delay freezing, and/or reduce ice adhesion, without the need for additional energy. Moreover, the combination of active Ice Protection Systems with passive coatings could prevent ice formation with an improved energy efficiency and reduced CO₂ emissions. Aligned with this aim, this study focuses on the development of anti-icing coatings, detailing the selection and characterization of the materials used in the coating formulations, including their chemical and physical properties. Subsequently, the experimental results are presented and analyzed, focusing on the characterization and anti-icing performance of the fabricated coatings through a series of tests. Finally, it concludes with a discussion of the key findings, useful for the formulation of advanced anti-icing coatings mainly for aeronautical applications.

Keywords: anti-icing coatings; superhydrophobic coatings; passive Ice Protection Systems (IPS); aeronautic; ice prevention; freezing delay time



Academic Editor: Ludmila B. Boinovich

Received: 6 February 2026

Revised: 19 February 2026

Accepted: 26 February 2026

Published: 28 February 2026

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1. Introduction

Ice accumulation on aircraft surfaces during flight poses a major safety and performance risk, as it alters aerodynamic behavior, reduces lift, increases drag, and may compromise critical onboard sensors, potentially leading to a loss of control [1]. In severe cases, ice accretion can cause structural damage or mechanical failure, further emphasizing the need for effective ice protection strategies [2,3]. Currently, aviation mainly relies on active Ice Protection Systems (IPSs), which are effective but energy-intensive and associated with increased operational and environmental costs [4,5]. To improve efficiency, growing research focuses on integrating passive superhydrophobic, anti-icing, and icephobic coatings that reduce ice nucleation, delay freezing, and lower ice adhesion [6,7]. When combined with active systems, these coatings can decrease energy consumption and operational demand by limiting ice accumulation and facilitating removal [8–16].

Superhydrophobic surfaces (SHSs), with micro/nanostructures, exhibit a low wettability and strong self-cleaning behavior. This performance arises from the Cassie state, where water droplets rest on an air layer trapped within the surface roughness, resulting in a solid–liquid–air interface governed by a low surface energy and hierarchical texture [17–19]. Due to their water-repellent nature and reduced ice adhesion, SHSs have been widely explored as icephobic coatings [20,21]. However, their effectiveness diminishes under extreme environmental conditions, e.g., at temperatures lower than $-12\text{ }^{\circ}\text{C}$ [15]. Consequently, to broaden the applicability of surface coatings as passive IPSs, research has expanded beyond the study and optimization of SHSs. Increasing attention is being directed toward the development of anti-icing and icephobic coatings specifically designed to inhibit or delay ice nucleation and freezing processes, rather than solely relying on water repellency. These alternative approaches aim to improve performance under extreme environmental conditions and enhance the reliability of passive anti-icing strategies.

In line with this approach, the present study addresses the early stages of developing a functional anti-icing coating specifically designed for aeronautical applications. The investigation focuses on identifying suitable base formulations to serve as polymeric matrices for the subsequent incorporation of hydrophobic/icephobic silica-based functional fillers. To this end, two distinct matrix systems were selected: (1) a bifunctional silicone–epoxy resin crosslinkable with amino–silane hardeners, and (2) a hydroxyl-functionalized polytetrafluoroethylene (PTFE) resin crosslinkable with di-isocyanate compounds. Various hardener types and different resin-to-hardener molar ratios were explored to identify the optimal formulations capable of curing at room temperature, thereby facilitating practical application and scalability.

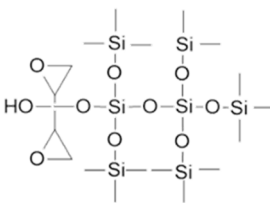
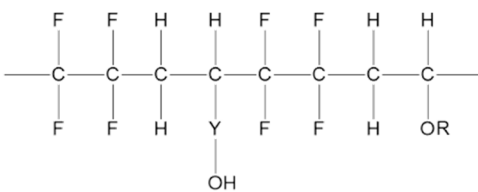
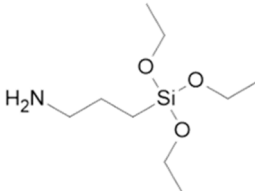
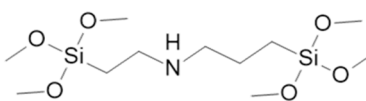
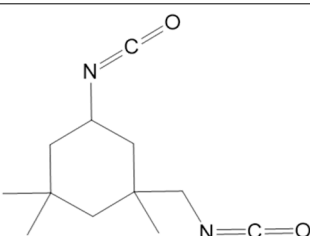
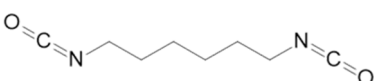
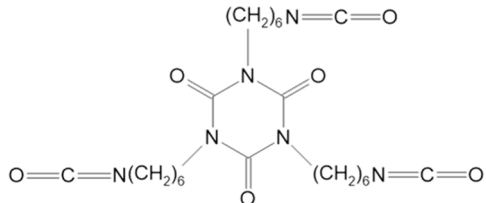
The curing behavior of the coatings was evaluated using Fourier Transform Infrared Spectroscopy (FT-IR). After curing, the coatings were characterized for their thermal and mechanical properties through Dynamic Mechanical Analysis (DMA) and Thermogravimetric Analysis (TGA). Surface properties were assessed by measuring the water contact angle at room temperature, as well as by calculating the surface free energy (SFE). Durability was investigated using a standardized cutting-and-tape adhesion test. Finally, the functional performance of the polymeric matrices was evaluated in terms of the freezing delay time (FDT).

2. Materials and Methods

2.1. Materials

The main components for formulations were SILIKOPON EF (Epoxide Equivalent Weight, EEW = 450 g/mol) (EF), purchased from Evonik, Essen, Germany, and ZEFFLE GK (Hydroxyl Equivalent Weight, OH-EW = 891 g/mol) (Z), provided by Daikin Chemicals, Osaka, Japan. As for the hardeners, DYNASILAN AMEO (Amine Hydrogen Equivalent Weight, AHEW = 110 g/mol) (AM), DYNASYLAN 1124 (AHEW = 341 g/mol) (DY) and TEGO® Cure 100 (TC) were purchased from Evonik, Essen, Germany, Isophorone diisocyanate (Isocyanate Equivalent Weight, NCO-EW = 111.14 g/mol) (ID) was provided by Acros Organics, Geel, Belgium, 1,6-Diisocyanate hexane (NCO-EW = 84.1 g/mol) (DH) was purchased from Aldrich, St. Louis, MO, USA, and Desmodur® Ultra N 3300 (NCO-EW = 193 g/mol) (D33) was supplied by Tokyo Chemical Industry, Tokyo, Japan. The chemical structures of the mentioned materials are reported in Table 1.

Table 1. Name, chemical structure, abbreviation, and main function of the chemicals used in the anti-icing formulations.

Name	Chemical Structure	Abbreviation	Main Function
SILIKOPON EF		EF	Main component
ZEFFLE GK		Z	Main component
DYNASYLAN AMEO		AM	Hardener
DYNASYLAN 1124		DY	Hardener
TEGO® Cure 100	Not available	TC	Hardener
Isophorone Diisocyanate		ID	Hardener
Diisocyanate Hexane		DH	Hardener
Desmodur® Ultra N 3300		D33	Hardener

2.2. Specimen Fabrication and Cleaning

In total, 44 stanned steel specimens, approximately $110 \times 30 \times 0.6$ mm in dimension, purchased from RS Components, were used as substrates, both as a reference and to apply the coatings.

To create a consistent surface roughness, the specimens were abraded using P80 sandpaper. Subsequently, they underwent a multi-stage cleaning process involving the

following solvents to remove any residual metal dust and impurities: Metaflex SP 1050, AkzoNobel Aerospace Coatings, Sassenheim, The Netherlands, deionized water, isopropyl alcohol, and ethanol.

2.3. Preparation of Coatings

The developed coatings formulations are reported in Table 2, each varying the concentration of curing agent.

Table 2. Developed coating formulations with ingredients' amount in grams and wt% for TC samples. The recommended and stoichiometric quantities of each reagent are in *italics* and **bold**, respectively.

ID Sample	EF	AM	DY	TC (wt% vs. EF)
EF-AM 2.0	10.00	2.00	-	-
EF-AM 2.2		2.22		
EF-AM 2.4		2.44		
EF-AM 2.6		2.60		
EF-DY 2.0	10.00	-	2.00	-
EF-DY 3.8			3.85	
EF-DY 5.6			5.60	
EF-DY 7.6			7.58	
EF-DY 9.6			9.38	
EF-TC 5	10.00	-	-	5
EF-TC 10				10
EF-TC 15				15
EF-TC 20				20
EF-TC 30				30
ID Sample	Z	DH	ID	D33
Z-DH 0.8	10.00	0.80	-	-
Z-DH 0.9		0.94		
Z-DH 1.1		1.10		
Z-ID 1.0	10.00	-	1.00	-
Z-ID 1.2			1.25	
Z-ID 1.5			1.50	
Z-D33 2.0	10.00	-	-	2.00
Z-D33 2.2				2.17
Z-D33 2.5				2.50

Coatings were prepared by mixing the solutions for 10 min with a magnetic stirrer at 300 rpm at room temperature and then poured onto the substrates and cured at room temperature. Following the curing process, the coating thickness ranged from 300 to 1000 μm , while the surface roughness (R_a) was measured between 0.1 and 0.3 μm .

2.4. Characterization

DMA was performed with a Triton Tritec 2000 (Triton Technology Ltd., Keyworth, UK) in tension mode on samples prepared in 15×8 mm rectangular molds and let cure at room temperature for one week before being tested. Samples were submitted to a $10^\circ\text{C}/\text{min}$ temperature ramp from between -30°C and -20°C up to around 60°C above the glass transition temperature, at 1 Hz frequency and 0.01 mm displacement.

Glass transition temperature (T_g) was considered as the maximum in the $\tan\delta$ curve, or as the point of flex in the modulus curve when the $\tan\delta$ peak was not well defined. The

glassy modulus (E'_g) was taken at $T_g - 50$ °C; the rubbery modulus (E'_r) was set at two times the value of T_g in degrees Celsius.

TGAs were performed with a TGA Q500 (TA Instruments, New Castle, DE, USA), submitting samples to a temperature ramp starting from 40 °C up to 750 °C at 10 °C/min in air atmosphere. The theoretical residual mass at 750 °C, corresponding to the $\text{SiO}_3/2$ moieties expected from the oxidative degradation of the crosslinked hardener, was calculated as follows:

$$\text{Res. Mass @750 } ^\circ\text{C (air)} = m_{\text{SiO}_3/2} / m_{\text{Tot}} \times 100 \quad (1)$$

with the following stoichiometric moles: $n_{\text{SiO}_3/2} = n_{\text{AM}} = 2 n_{\text{DY}}$. The additional contribution arising from the degradation of EF, which cannot be quantitatively predicted a priori, was assessed through Thermogravimetric Analysis (TGA) performed in air.

FT-IR analyses in attenuated total reflectance (ATR) mode were performed using an FT-IR IS50 from Thermo Fisher Scientific SPA (Segrate, Milan, Italy) on the single components and on the prepared samples, both right after their mixing and after one week of room temperature curing.

Surface roughness was characterized using a SAMA SA6260 roughness meter (S.A.M.A. Italia S.r.l., Viareggio, Italy) in compliance with ISO 4288 standards [22].

The cured coatings' thickness was determined using a PosiTector 6000 (DeFelsko Inspection Instruments, Ogdensburg, NY, USA), operating on the magnetic induction principle.

Contact angle measurements were performed at room temperature in compliance with the ASTM D7490-13 standard [23], using water (H_2O), diiodomethane (CH_2I_2) and formamide (HCONH_2) as standards.

The polar and dispersive components of the reference liquids are listed in Table 3. Tests were carried out by depositing ten drops of 3 μL of each liquid on the sample surface.

Table 3. Surface energy components of the reference liquids.

Liquid	Formula	Room T Surface Energy [mN/m]	Dispersive Component [mN/m]	Polar Component [mN/m]
Water	H_2O	72.8	26.4	46.4
Diiodomethane	CH_2I_2	50.8	50.8	0
Formamide	HCONH_2	57.0	22.4	34.6

Surface free energy (SFE) was calculated by implementing the Owens–Wendt (OW) method [24–26] with a nonlinear programming problem developed at CIRA, described in Equations (2) and (3) [27].

$$\min_{\gamma_s^d, \gamma_s^p} \left(\left(\gamma_{l_3}^d \gamma_s^d \right)^{1/2} + \left(\gamma_{l_3}^p \gamma_s^p \right)^{1/2} - \frac{\gamma_{l_3} (1 + \cos \theta_3)}{2} \right) \quad (2)$$

$$s.t. \begin{cases} \left(\gamma_{l_1}^d \gamma_s^d \right)^{1/2} + \left(\gamma_{l_1}^p \gamma_s^p \right)^{1/2} - \frac{\gamma_{l_1} (1 + \cos \theta_1)}{2} = 0 \\ \left(\gamma_{l_2}^d \gamma_s^d \right)^{1/2} + \left(\gamma_{l_2}^p \gamma_s^p \right)^{1/2} - \frac{\gamma_{l_2} (1 + \cos \theta_2)}{2} = 0 \\ E[\theta_1] - \sigma_{\theta_1} \leq \theta_1 \leq E[\theta_1] + \sigma_{\theta_1} \\ E[\theta_2] - \sigma_{\theta_2} \leq \theta_2 \leq E[\theta_2] + \sigma_{\theta_2} \\ E[\theta_3] - \sigma_{\theta_3} \leq \theta_3 \leq E[\theta_3] + \sigma_{\theta_3} \end{cases} \quad (3)$$

In the problem, a gaussian distribution of the contact angles was assumed in order to statistically characterize them through the mean value ($E[\theta_1]$, $E[\theta_2]$, $E[\theta_3]$) and the standard deviation (σ_{θ_1} , σ_{θ_2} , σ_{θ_3}). The stated optimization problem allowed us to determine the θ_1 , θ_2 , θ_3 contact angles that identify the SFE components, which minimize the

third equation, considering the measured variability and the constitutive equations of the considered liquids.

A cutting-and-tape adhesion test was performed following the ASTM D3359-09 standard [28]. The test has been performed by incising the coating with a standardized tool with a PA-2056 blade, creating a grid on the material. Subsequently, regulated tape has been applied and then abruptly removed from the surface to induce the possible detachment of the material from the surface. Adhesion has then been assessed by analyzing the image with a digital microscope (Dino-Lite Edge3.0, AnMo Electronics Corporation, Taipei, Taiwan) according to a chart provided by the standard.

Freezing delay time (FDT) was evaluated using an experimental setup developed at CIRA (Figure 1), consisting of an environmental chamber capable of precisely controlling temperature (down to $-12\text{ }^{\circ}\text{C}$) and relative humidity (below 24%), managed via Arduino-based software, 2.1.4 version. Real-time video and image acquisition was performed using a Dino-Lite Edge3.0 digital microscope, enabling the determination of both the freezing point of $5\text{ }\mu\text{L}$ water droplets and the FDT, defined as the time elapsed between droplet deposition and the onset of freezing on the tested coatings, in comparison to a reference surface.

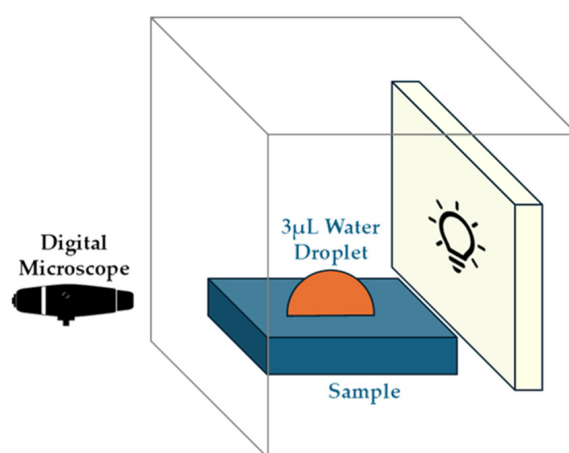


Figure 1. Schematic representation of the experimental setup for FDT evaluation.

3. Results

3.1. Design of Formulation

Icephobicity is typically defined as the ability of a surface to delay freezing time, decrease the nucleation temperature and/or facilitate the ice detachment, by reducing the adhesion strength between the ice and the surface. The interactions between the ice and the coating are the result of a combination of electrostatic forces, hydrogen bonding, van der Waals forces and mechanical interlocking [29], assessed by means of the work of adhesion, W_a , using the Young–Dupré equation:

$$W_a = \gamma_{LV}(1 + \cos\theta) \quad (4)$$

where γ_{LV} is the surface tension in air and θ is the water contact angle (WCA). Therefore, interfacial interactions can be effectively minimized by employing coatings characterized by a low SFE and high WCA. Superhydrophobic coatings, which exhibit these properties, can offer significant advantages within a specific environmental range, particularly under glaze ice conditions, i.e., temperatures between $-12\text{ }^{\circ}\text{C}$ and $0\text{ }^{\circ}\text{C}$ [13,16]. However, under more demanding environmental conditions, i.e., at lower temperatures, such coatings may not be able to allow water droplets to run off before freezing or unintentionally increase ice adhesion due to the interlocking of ice within the surface's high roughness. To address this

issue, the objective of this study is to design and develop coatings characterized by a low SFE and appropriately tailored surface roughness, optimized for specific environmental and operational conditions.

To this end, two distinct low-SFE matrices were selected and investigated. The first matrix, a bifunctional silicone–epoxy resin, was designed to be crosslinked with amino-silane hardeners, enabling the functionalization of the silicone–epoxy network with Si–OR groups. These groups facilitate further bonding with selected silica fillers via the sol–gel process, resulting in a co-continuous organic–inorganic three-dimensional network whose roughness depends on the amount of filler used. In line with this objective, mono-functional amino-silanes, i.e., DY, were also used as hardeners, primarily to enable functionalization rather than to crosslink the epoxy network. In contrast, the second matrix—a hydroxyl-functionalized polytetrafluoroethylene (PTFE) resin—utilizes its OH groups for both crosslinking and anchoring the silica fillers.

Many formulations have been prepared and characterized. However, only the most relevant results will be discussed.

3.2. Characterization Results

3.2.1. DMA

The DMA main results in terms of T_g , E'_g and E'_r are displayed in Table 4. A comparison between the EF and Z samples reveals that the first family exhibits a higher T_g . This is likely due to the higher rigidity given by the epoxy–silicone network of the EF samples, whereas the linear chain in Z results in a lower energy barrier for the glass transition. More difficult is the comparison in terms of the glassy modulus, which reflects the network architecture and the intermolecular forces among its chains. In this regard, the Z samples are stiffer compared to the EF ones, with a glassy modulus that can be as high as 1 GPa.

Table 4. T_g , E'_g and E'_r data measured through DMA.

Sample ID	T_g (°C)	Glassy Modulus E'_g (MPa)	Rubbery Modulus E'_r (MPa)
EF-AM 2.4	45.3	891	9.43
EF-TC 20	69.4	655	8.50
Z-DH 0.8	26.9	1261	5.59
Z-ID 1.0	34.8	1047	1.29
Z-D33 2.2	32.8	201	5.13

Regarding the two EF samples, the one cured with TC exhibits a higher glass transition temperature (T_g). This is likely due to the specific composition of the TC hardener, which is optimized for ambient temperature curing, as indicated in the corresponding technical data sheet.

Regarding the Z samples, the DH-based formulation exhibits the lowest T_g . The linear and more flexible structure of the DH hardener could result in a more flexible and less densely crosslinked network compared to that obtained with ID. Contrary to expectations, the sample cured with the trifunctional D33 neither showed the highest T_g nor exhibited the highest glassy modulus. This unexpected result may be explained by an incomplete curing reaction, possibly due to steric hindrance or suboptimal curing conditions, which limited the extent of crosslinking.

3.2.2. TGA

The Thermogravimetric Analysis (TGA) of EF-containing samples revealed a multistep thermal degradation profile (Figure 2). The decomposition behavior was strongly dependent on the curing agent, indicating differences in both the thermal stability and degradation pathways of the crosslinked networks. Among the investigated silane curing agents, the systems cured with AM and DY exhibited the highest thermal stability below 300 °C. At higher temperatures, however, the formulation cured with TC showed a superior thermo-oxidative stability, yielding the highest residual mass at 750 °C (40.6 wt%), compared with 28.6 and 29.6 wt% for the corresponding formulations cured with AM and DY, respectively (Figure 2 and Table 5). The latter formulations enabled an estimation of the SiO_x mass contribution attributable to EF, which was determined to be 24.0 and 24.5 wt% for the AM- and DY-cured systems, respectively, considering $\text{SiO}_{3/2}$ contributions from the hardeners of 4.61 and 5.08 wt%. Assuming the same EF-derived SiO_x contribution for the TC-cured sample, the $\text{SiO}_{3/2}$ content associated with the TC hardener was estimated to be 16.6 wt%. This comparatively high value accounts for the enhanced thermo-oxidative stability observed for the EF-TC 20 formulation.

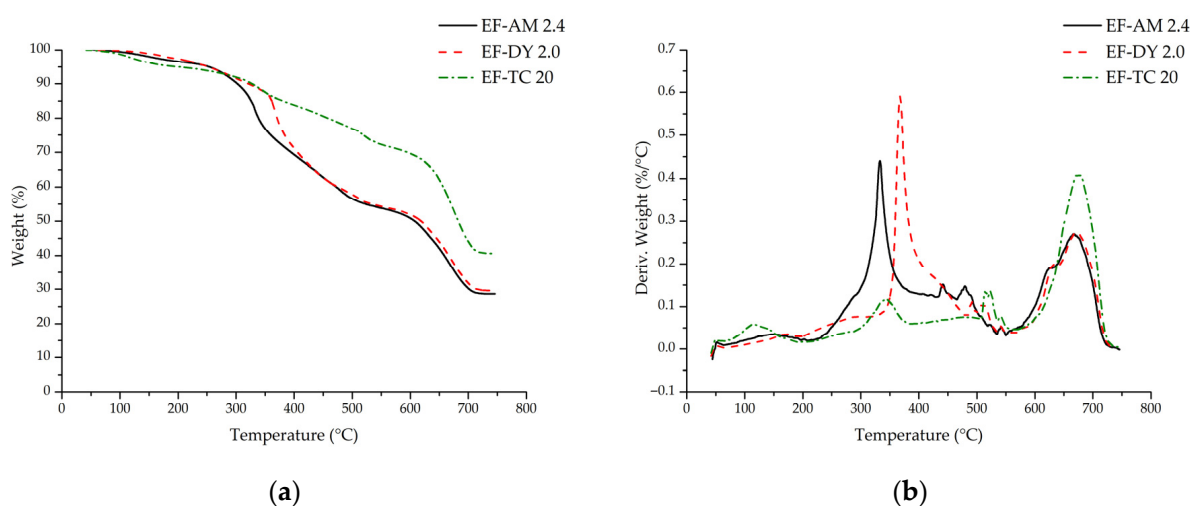


Figure 2. TGAs of the EF samples; weight percentage (a) and derivative weight (b).

Table 5. Assessment of SiO_x moiety mass from EF. $m_{\text{SiO}_{3/2}}$ is the mass of $\text{SiO}_{3/2}$ moieties, $\text{H-SiO}_{3/2}$ is the weight percentage of $\text{SiO}_{3/2}$ moieties derived by the hardener, Res.mass is the weight percentage of residual mass at 750 °C, and EF- SiO_x is the weight percentage of SiO_x moieties from EF. Values in italics were derived assuming an EF- SiO_x contribution of 24 wt% for EF-TC-20.

ID Sample	$m_{\text{SiO}_{3/2}}$ [g]	H- $\text{SiO}_{3/2}$ [wt%]	Res.Mass @ 750 °C [wt%]	EF- SiO_x [wt%]
EF-AM 2.4	0.57	4.61	28.6	24.0
EF-DY-2.0	0.61	5.08	29.6	24.5
EF-TC-20	1.99	<i>16.60</i>	40.60	24

In contrast, the Z samples did not exhibit significant differences among themselves, as their thermal responses were highly similar owing to their comparable chemical compositions; only the Z-D33 2.2 formulation showed a slightly higher thermal stability at temperatures above 300 °C. As expected, the residual mass at 750 °C was negligible, reflecting the absence of inorganic components capable of withstanding oxidative degradation (Figure 3).

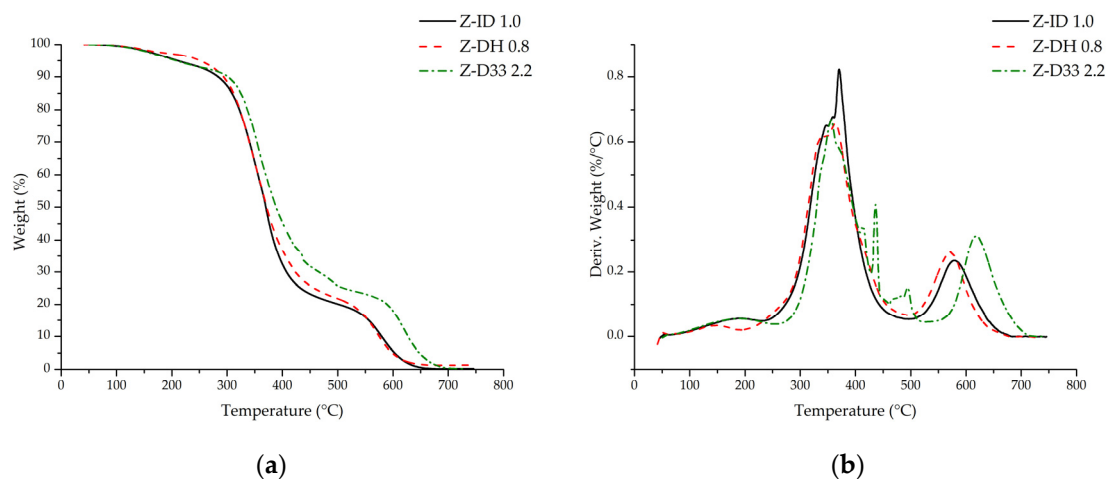


Figure 3. TGAs in air for the Z samples; weight percentage (a) and derivative weight (b).

3.2.3. Fourier Transform Infrared Spectroscopy (FTIR)

Representative IR spectra for each synthesized system are presented in Figures 4–9, with a focus on the peaks related to the characteristic groups involved in the crosslinking reactions. The intensity of these peaks has been monitored and quantified to assess the successful reaction.

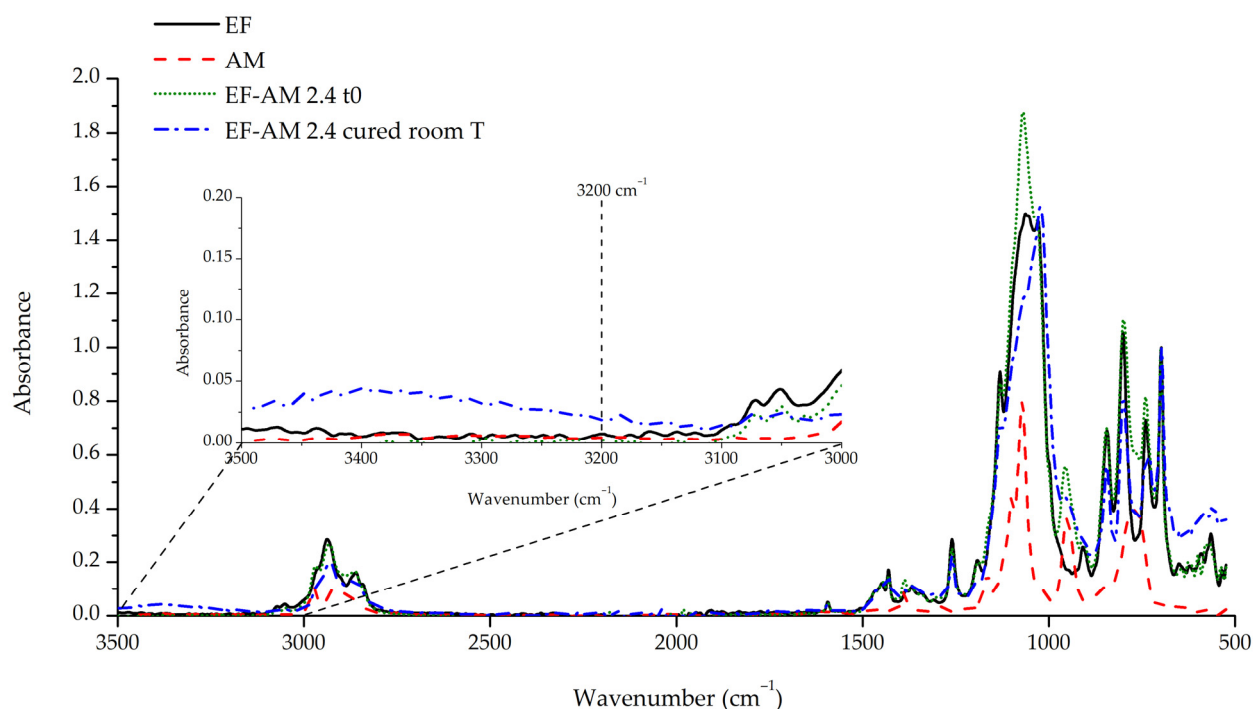


Figure 4. FTIR analysis of EF-AM 2.4 with a zoom on the characteristic -OH stretching band.

In particular, for samples containing SILIKOPON EF (Figures 4–6), the -Si-O-Si- stretching vibration peak, between 697 and 699 cm⁻¹, has been considered as the internal standard for normalization, while the progressing of the crosslinking reaction has been monitored with the development of the stretching vibration of the -OH group-associated band between 3200 cm⁻¹ and 3500 cm⁻¹, indicative of the appearance of the hydroxyl group as a result of oxirane ring opening. This choice was forced by the fact that the direct monitoring of the characteristic epoxy peak at approximately 910 cm⁻¹ was precluded by the formation of unidentified products, which altered the cured samples' spectra, not allowing us to observe

the peak's evolution. On the other hand, the C–H stretching vibration of the oxirane ring at $\sim 3050\text{ cm}^{-1}$ [30] could not be clearly detected due to its inherently low intensity.

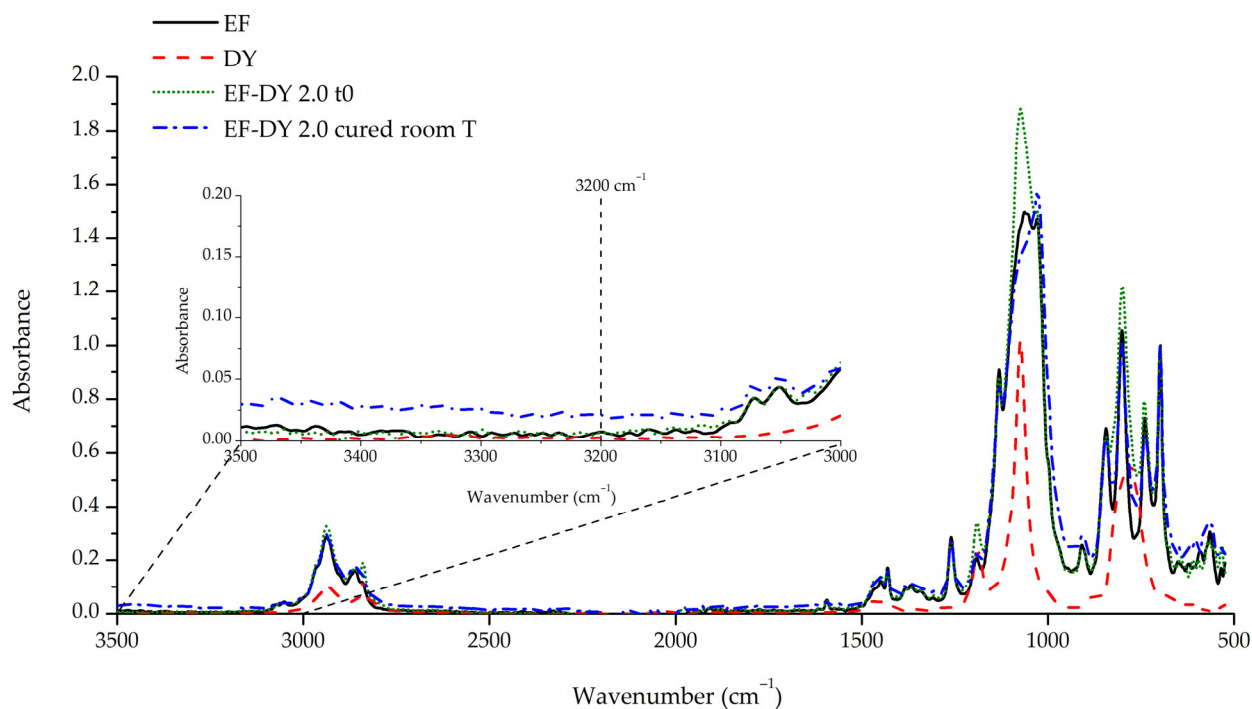


Figure 5. FTIR analysis of EF-DY 2.0 with a zoom on the characteristic -OH stretching band.

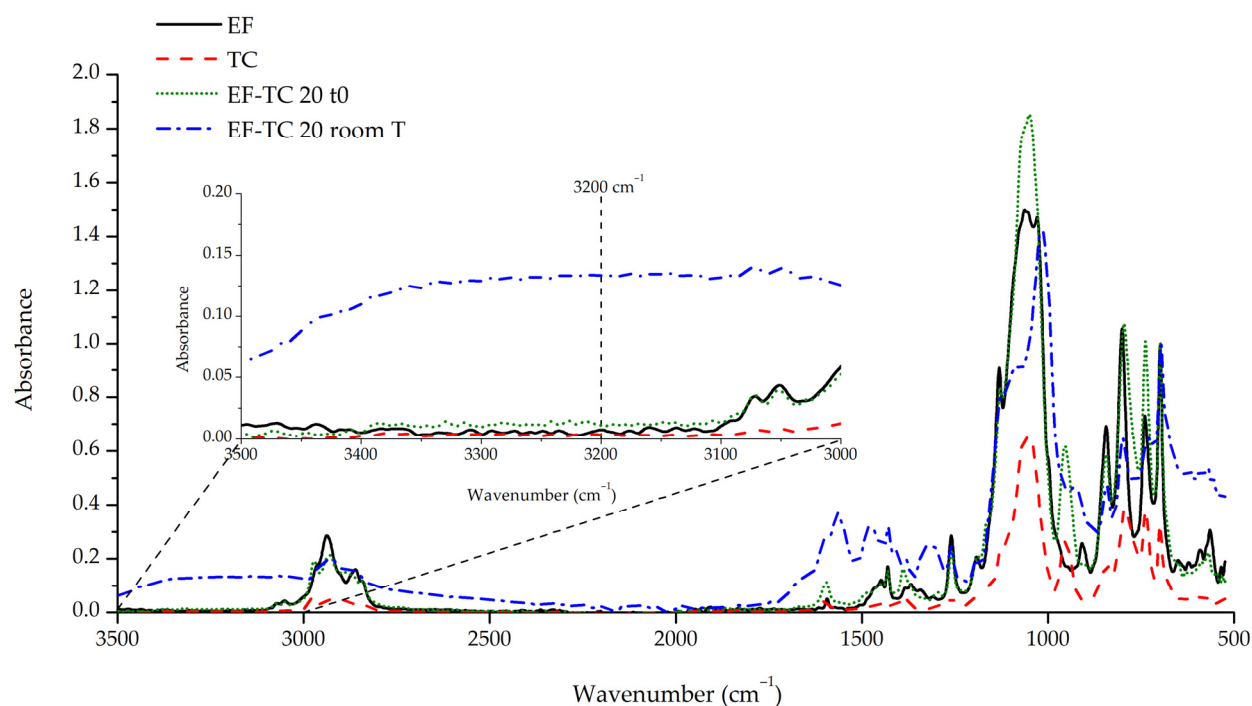


Figure 6. FTIR analysis of EF-TC 20 with a zoom on the characteristic -OH stretching band.

The formation of the -OH group characteristic band in the EF-AM 2.4 and EF-TC 20 formulations is a proof of the successful crosslinking reaction [31]. On the other hand, EF-DY 2.0 (Figure 5) shows the worst increment of the -OH band in the cured sample and suggests an incomplete curing process. This may be attributed to the fact that DY is a secondary amine, which exhibits a slower reactivity compared to the primary amine

functionality of AM. Differences in the degree of curing are also evident from the variations in the shape of the multiple peaks observed in the $2800\text{--}2900\text{ cm}^{-1}$ region, corresponding to the asymmetric and symmetric C-H stretching vibrations of -CH_2 groups at $\sim 2920\text{ cm}^{-1}$ and $\sim 2850\text{ cm}^{-1}$, respectively. The incomplete curing of the EF-DY 2.0 formulation is supported by the similarity between the spectra of the cured sample and that of the unreacted EF. In contrast, the spectra of the cured formulations differ significantly from those of EF in the $2800\text{--}2900\text{ cm}^{-1}$ region, indicating that EF has successfully reacted in these systems (Figures 4–6).

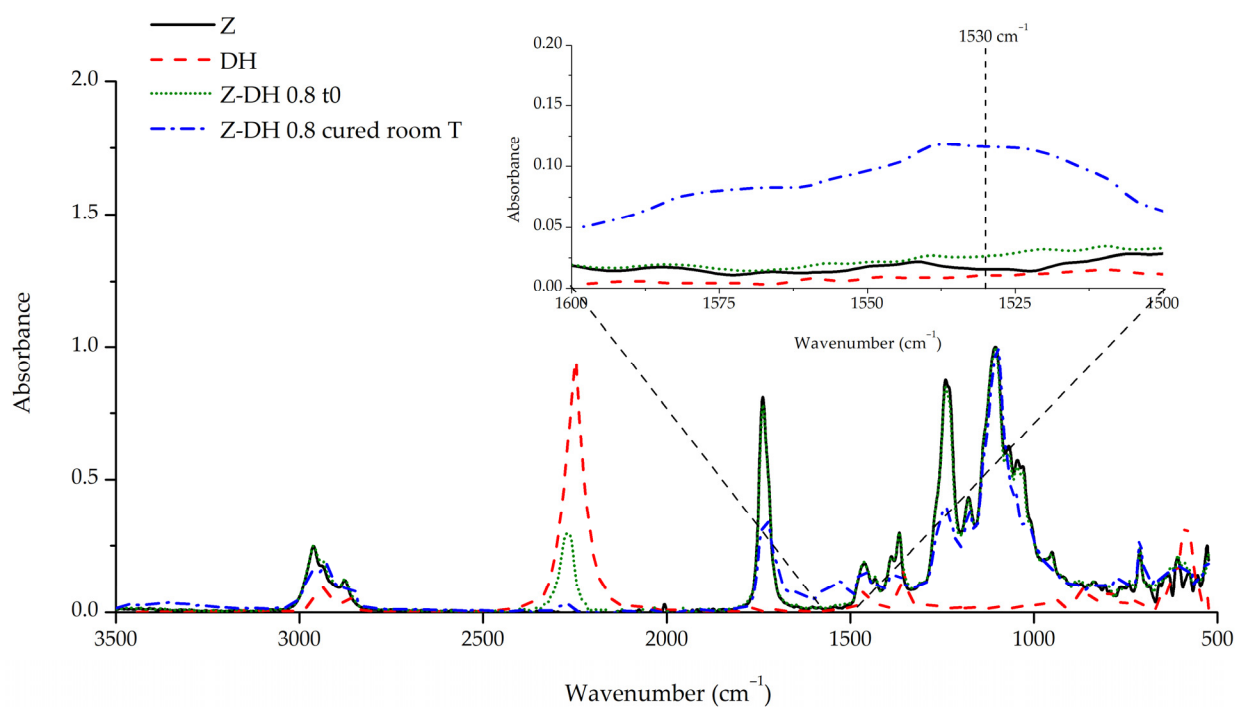


Figure 7. FTIR analysis of Z-DH 0.8 with a zoom on the characteristic -NH bending peak.

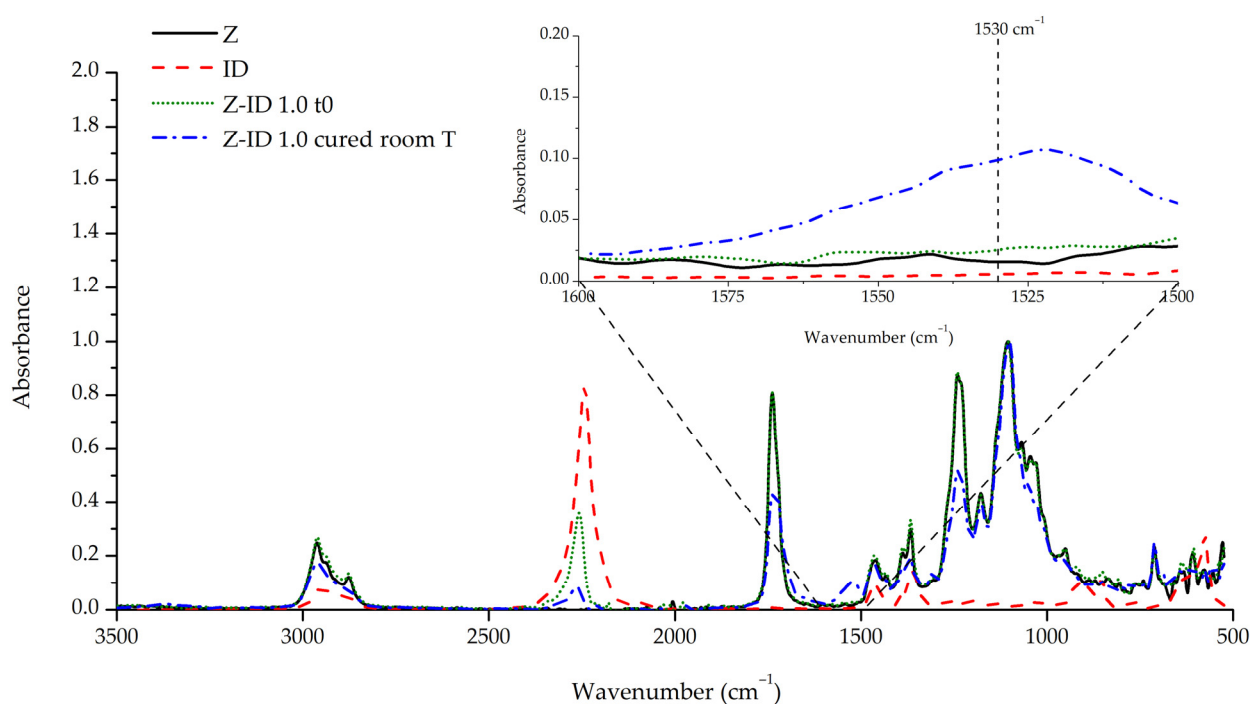


Figure 8. FTIR analysis of Z-ID 1.0 with a zoom on the characteristic -NH bending peak.

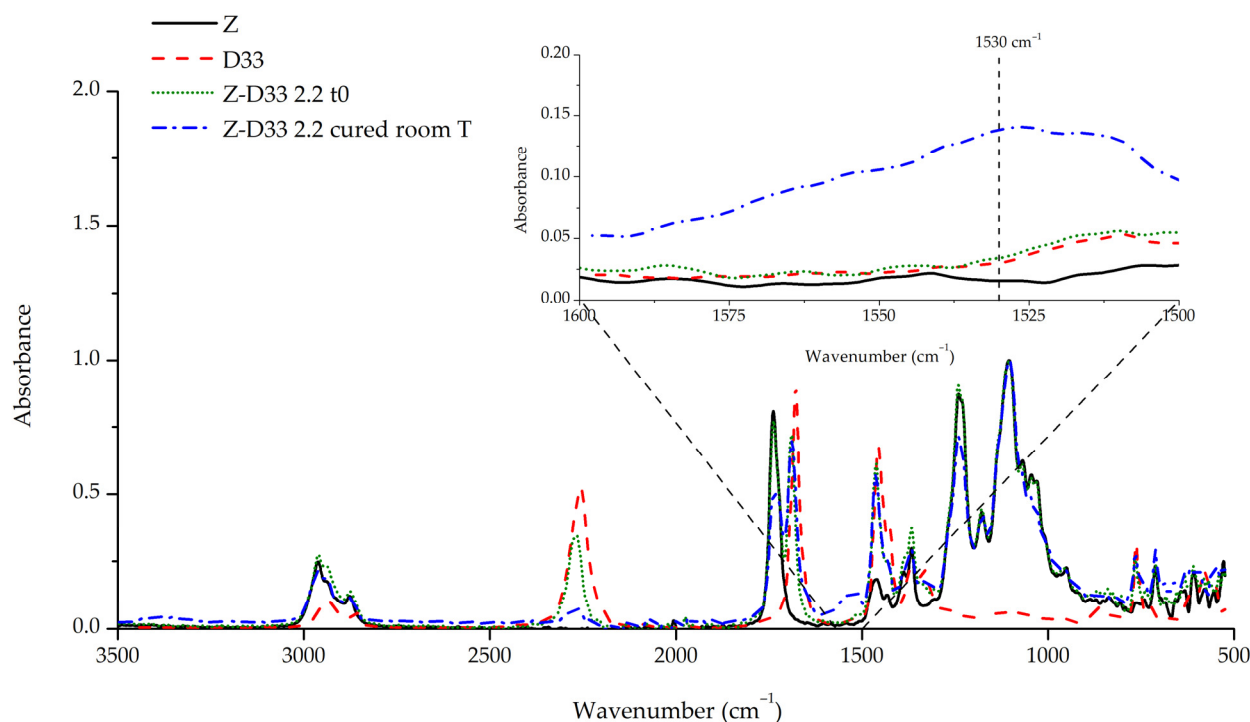


Figure 9. FTIR analysis of Z-D33 2.2 with a zoom on the characteristic -NH bending peak.

In Z-containing formulations, the CF_2 stretching vibration at approximately 1105 cm^{-1} was selected as the internal standard for normalization, whereas the formation of urethane linkages was monitored by observing the -NH bending vibration of the carbamate group at 1530 cm^{-1} in order to assess the occurrence of the crosslinking reaction (Figures 7–9).

The development of the -NH group-associated peak confirms the correct happening of the network formation. No clear differences in the peak intensity can be noticed between the formulations.

3.2.4. CA and SFE Measurements Results

Table 6 shows the average values of the CA measured at room temperature for a sample of each formulation, with the corresponding standard deviation value. The improvement in wettability can be seen in all samples, each of which achieves a slight hydrophobicity, as the CA is around 90° . The representative images of contact angle measurements can be found in Figure 10.

Table 6. Samples' CA [$^\circ$] measurements with water, diiodomethane and formamide, with corresponding standard deviation.

	H_2O	CH_2I_2	HCONH_2
Reference	64 ± 11	63 ± 3	60 ± 10
EF-AM 2.4	84 ± 4	53 ± 2	80 ± 3
EF-DY 2.0	88 ± 2	60 ± 7	77 ± 3
EF-TC 20	96 ± 7	72 ± 9	86 ± 4
Z-DH 0.8	94 ± 11	52 ± 3	77 ± 5
Z-ID 1.0	87 ± 3	51 ± 5	72 ± 6
Z-D33 2.2	85 ± 3	51 ± 3	64 ± 6

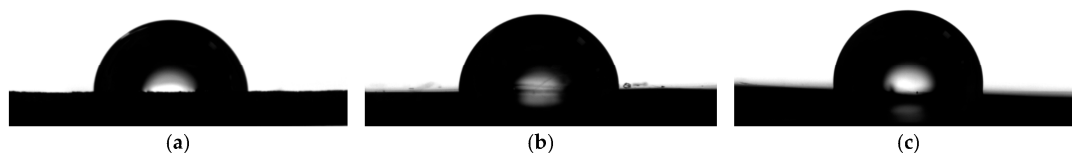


Figure 10. Water CA measurements for reference (a), EF-AM 2.4 (b), and EF-TC 20 (c) samples.

Among the samples tested, EF-TC 20 achieved the best improvement in wettability with all employed liquids.

Figure 11 illustrates the calculated SFE for the selected samples compared to the reference. An inverse relationship between CA and SFE values is observed, as expected.

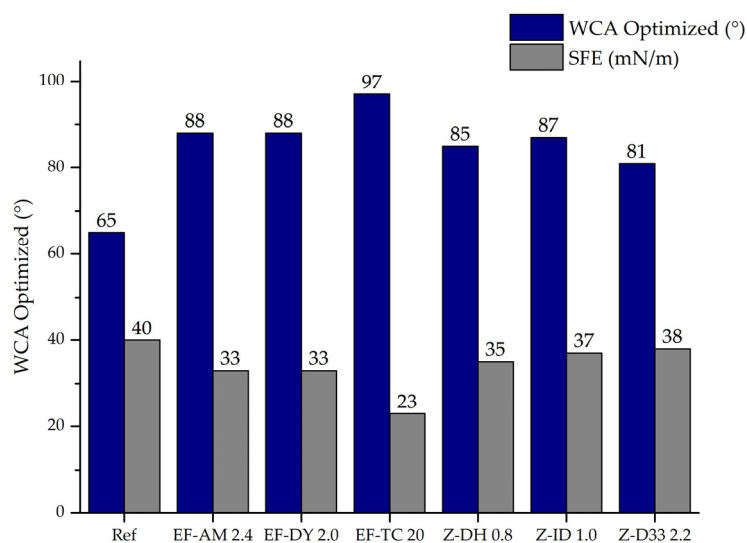


Figure 11. CA and SFE values for reference and selected formulations.

As already seen with the CA measurements, EF-TC 20 is one of the most promising formulations, as the measured SFE is the lowest among all formulations.

On the other hand, Z-containing formulations exhibit the poorest performances in terms of both CA and SFE. This result deviates from the expected behavior typically associated with fluoropolymer-based systems; however, it is consistent with the surface energy value of 30.8 mN/m reported by Li et al. [32]. The slight discrepancy may be attributed to the use of a different isocyanate curing agent in the present formulation. Overall, all developed formulations give a higher CA and decrease SFE, improving the system hydrophobic behavior.

3.2.5. Cutting-and-Tape Adhesion Test Results

The most promising samples, EF-AM 2.4 and EF-TC 20, gave an excellent response in terms of adhesion strength, as no evident detachment was detected, achieving an evaluation of 5B, which is the highest score in the standard scale.

A demonstration of the excellent adhesion properties of the coatings can be found in Figure 12, where the larger clear lines are the ones constituting the generated grid and the irregular ones are the result of the sandpaper treatment prior to coating application, visible thanks to the transparency of the coatings. In the figure, the grids are intact and present no sign of detachment, especially at their intersections, which are supposed to be the weakest points of the coating.

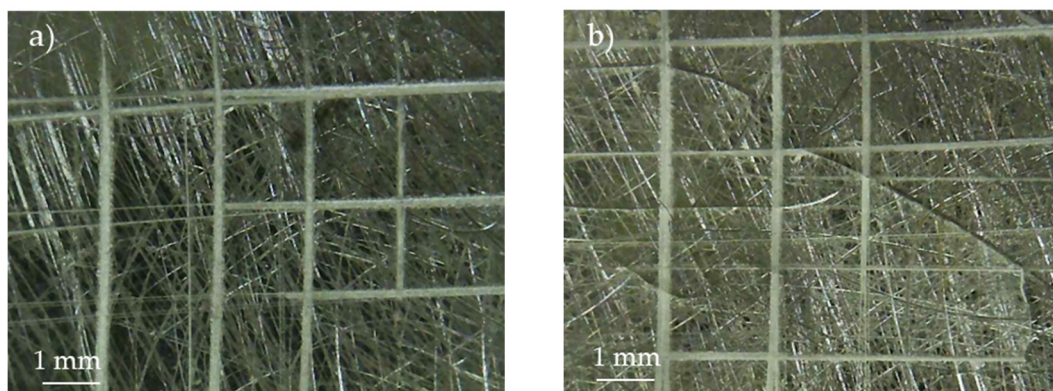


Figure 12. Surface grid generated by the adhesion test with no sign of detachment for EF-AM 2.4 (a) and EF-TC 20 (b).

3.2.6. Freezing Temperature Test Results

The results demonstrated a satisfactory average freezing temperature of -8.5°C , which is much lower compared to the icing temperature of the reference, which is as low as around -2°C . Table 7 presents the individual values for each sample from which it was possible to obtain a numerical value. Notably, the freezing temperature of some droplets was below the operational limit of the experimental setup, i.e., -12°C , preventing their observation, which means that their freezing has been considerably delayed in terms of temperature. These results are more promising than those reported for similar epoxy–fluoropolymer (EF) systems [33]. In that study, comparable formulations exposed to freezing drizzle at 8°C for 5 min exhibited irregular and discontinuous wetting due to their hydrophobic nature, which in turn led to non-uniform ice accretion. Furthermore, samples EF-DY 7.6 and EF-DY 9.4 could not be tested due to inadequate adhesion to the metal substrate following curing.

Table 7. Lowest freezing temperature, where measured, for each sample.

Sample ID	Drop Freezing Temperature [$^{\circ}\text{C}$]	Sample ID	Drop Freezing Temperature [$^{\circ}\text{C}$]
Reference	-1.81		
EF-AM 2.0	/	Z-DH 0.8	-10
EF-AM 2.2	-6.56	Z-DH 0.9	-10.2
EF-AM 2.4	/	Z-DH 1.1	-11.1
EF-AM 2.6	-8.5		
EF-DY 2.0	-9.75	Z-ID 1.0	-6.7
EF-DY 3.8	/	Z-ID 1.2	-6.8
EF-DY 5.6	-8.44	Z-ID 1.5	-10.4
EF-TC 5	/	Z-D33 2.0	-8.56
EF-TC 10	-8.75	Z-D33 2.2	-9.25
EF-TC 15	/	Z-D33 2.5	-9.13
EF-TC 20	-11.6		
EF-TC 30	/		

This significant slowing of ice nucleation may be attributed to the potential thermal insulation provided by the coating, with only a minor contribution from the reduced surface contact arising from the enhanced hydrophobicity of the coating.

3.2.7. Freezing Delay Time (FDT) Test

Samples exhibiting a significant freezing point depression were selected for evaluation in the freezing delay time test. The test temperature was set to -0.5 ± 0.1 °C, based on the reference sample's freezing point of -1.81 °C. For each selected sample, the time required for ice formation was monitored. At -0.5 °C, the reference sample froze in less than 10 min (Figure 13). In contrast, none of the droplets deposited on the selected formulations underwent freezing during the entire residence time in the test chamber (Figure 14), with the exception of sample Z-D33 2.2, where freezing occurred after 48 min (Table 8).

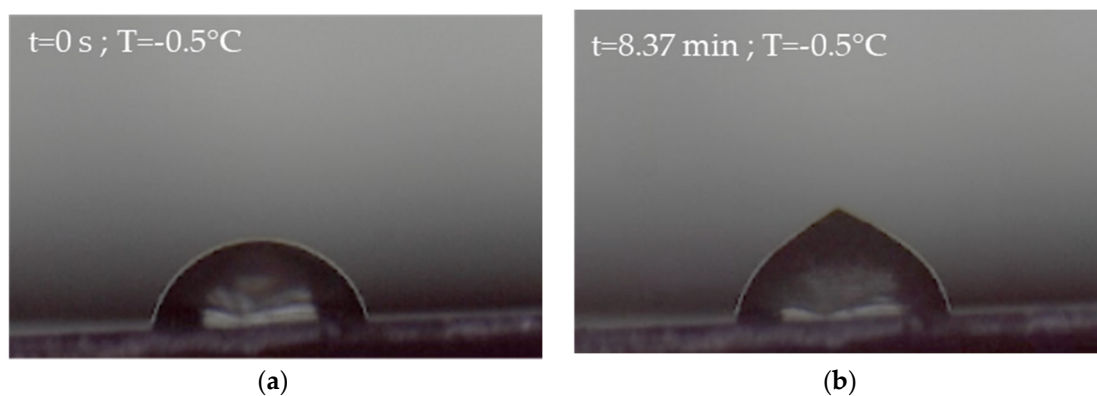


Figure 13. Water droplet on reference surface at the beginning (a) and the end (b) of FDT test.

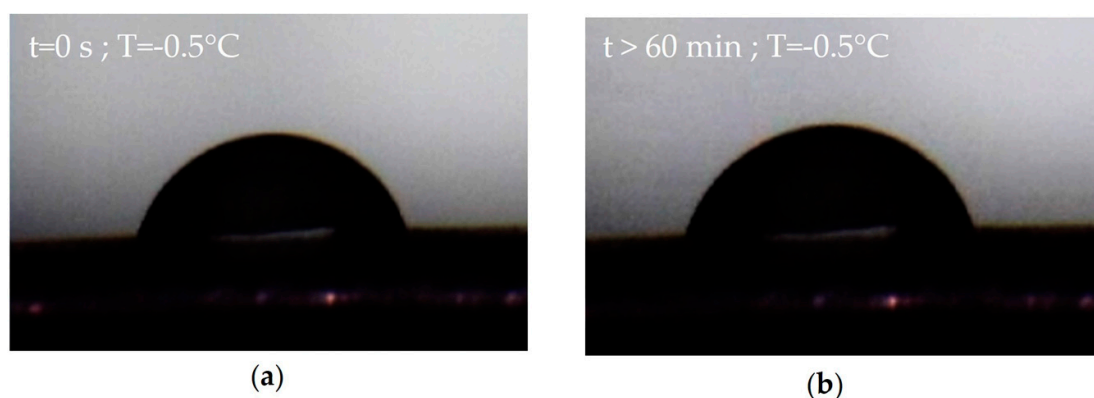


Figure 14. Water droplet on EF-TC 20 surface at the beginning (a) and the end (b) of FDT test.

Table 8. Freezing time data for each selected sample.

Sample ID	Freezing Time [min]
Reference	8.37
EF-AM 2.4	>60
EF-TC 20	>60
Z-DH 0.8	>60
Z-ID 1.0	>60
Z-D33 2.2	48.1

This result proves the successful role of the developed coatings in delaying ice formation, increasing by at least 7 times the performances, except for Z-D33 2.2, which delayed ice nucleation by 40 min compared to the reference.

4. Discussion

The present study investigated two families of low-surface-energy polymeric matrices as base formulations for the development of passive icephobic coatings curable at room

temperature, intended for aeronautical applications. The discussion of the results highlights the interplay between chemical structure, curing behavior, thermo-mechanical properties, surface characteristics, and icephobic performance, allowing key design guidelines to be identified for future coating optimization.

The DMA and FT-IR results collectively demonstrate that the curing agent plays a decisive role in determining the final network architecture and, consequently, the thermo-mechanical behavior of the coatings. EF-based formulations generally exhibited higher glass transition temperatures compared to Z-based systems, reflecting the intrinsic rigidity of the epoxy–silicone backbone relative to the more flexible PTFE-derived chains. Among EF systems, the TC-cured formulation showed the highest T_g . This behavior is attributed to the optimized reactivity of the TC hardener, which promotes a more homogeneous and tightly crosslinked network at room temperature. In contrast, EF-DY formulations exhibited evidence of incomplete curing, as confirmed by the FT-IR analysis, and supported by their inferior mechanical consistency and adhesion performance. The lower reactivity of the secondary amines in DY limits epoxy ring opening and hinders the formation of a fully crosslinked network. As DY was primarily selected to promote functionalization rather than network formation, it may be better suited for future developments involving additional crosslinking through silica-based moieties. For Z-based formulations, the relatively narrow range of T_g values and similar DMA responses suggest that the different isocyanate hardeners produced networks with comparable crosslink densities under the adopted curing conditions. However, the unexpectedly low modulus and T_g of the Z-D33 system indicate that steric hindrance and incomplete network formation can outweigh the theoretical advantages of trifunctional hardeners, especially under room-temperature curing constraints.

The Thermogravimetric Analysis revealed a clear distinction between EF- and Z-based systems. EF coatings showed a superior thermo-oxidative stability, particularly when cured with TC, which resulted in a significantly higher residual mass at 750 °C. The large contribution of $\text{SiO}_3/2$ moieties confirms the formation of a robust inorganic–organic hybrid network, which is highly desirable for further functionalization with silica nanoparticles to enhance the icephobic performances. In contrast, Z-based coatings exhibited a negligible residual mass, consistent with their purely organic nature.

Contact angle and surface free energy measurements demonstrated that all coatings improved surface hydrophobicity relative to the bare steel reference. EF-TC 20 emerged as the most promising formulation, combining the highest water contact angle and the lowest SFE. This behavior is consistent with the presence of silicone segments and a well-developed crosslinked network that effectively lowers the surface energy. Interestingly, Z-based formulations exhibited a poorer hydrophobic performance than expected for fluoropolymer-derived systems. This result aligns with the literature data for hydroxyl-functionalized PTFE matrices [34] and can be attributed to the presence of polar urethane groups and the specific curing chemistry employed.

The most significant outcome of this work is the pronounced delay in ice nucleation observed for all coated samples compared to the reference surface. The freezing temperature depression of up to ~ 10 °C and the complete suppression of freezing over one hour for most formulations represent a substantial improvement over uncoated steel and compare favorably with previously reported chemical modified epoxy–silicone hybrid resins, i.e., about 40 min at -15 °C [35], and PTFE-based coatings, i.e., 1295.3 s at -10 °C [36]. When compared with other advanced anti-icing technologies, the performance achieved in the present work remains highly competitive. Slippery liquid-infused porous surfaces (SLIPS), for instance, have demonstrated significant freezing delay times at subzero temperatures. Shen et al., for instance, reported that GO-based SLIPS systems exhibit freezing delay times

of up to 970 s at $-6\text{ }^{\circ}\text{C}$ and 364 s at $-10\text{ }^{\circ}\text{C}$ [37], whereas Yu et al. described amphiphilic organogel-based SLIPS capable of delaying freezing by approximately 1000 s at $-20\text{ }^{\circ}\text{C}$ [38]. Similarly, superhydrophobic coatings based on room-temperature vulcanized silicone rubber combined with silane-modified micro-/nano- SiO_2 particles have shown promising results. Peng et al. reported that such coatings could extend the droplet freezing time by 193% at $-10\text{ }^{\circ}\text{C}$, increasing it from 15 to 44 min [39]. In addition, nanoporous microstructured surfaces consisting of carbon nanotube (CNT) forests and CNT micropillars coated with poly (1H,1H,2H,2H-perfluorodecylacrylate) (pPFDA) demonstrated a freezing delay up to 1351 ± 50 s, while the reference value (27s) corresponds to the freezing time measured on bare silicon [40]. Notably, the anti-icing performance did not directly correlate with surface hydrophobicity alone. Instead, the results suggest that thermal insulation provided by the polymeric coating may play a dominant role in delaying ice nucleation, while the reduced solid–liquid contact due to the poorly enhanced hydrophobicity contributes only partially. The freezing delay time (FDT) tests further corroborate this interpretation. At $-0.5\text{ }^{\circ}\text{C}$, five out of six of the tested coatings completely inhibited ice formation for more than 60 min, whereas the reference surface froze in less than 10 min. The inferior performance of Z-D33 2.2 is consistent with its weaker mechanical integrity and less optimal network structure, reinforcing the importance of curing efficiency and bulk properties in anti-icing behavior. However, it should be noted that the observed performance in terms of delayed ice nucleation and freezing delay time is likely influenced by the relatively high thickness of the coatings applied in this study. With thicknesses ranging from 300 to 1000 μm , the coatings may enhance the system's thermal insulation, thereby contributing to the measured freezing delay. Future work will be dedicated to a systematic investigation of the effect of the coating thickness on the freezing delay time, with the aim of separating thickness-related thermal effects from intrinsic material properties and further optimizing the overall anti-icing performance.

From an application-oriented perspective, the results demonstrate that effective anti-icing behavior, capable of delaying ice formation under specific environmental conditions, can be achieved without relying on extreme superhydrophobicity. EF-AM and EF-TC formulations combine adequate mechanical robustness, excellent adhesion to metallic substrates, low surface energy, and outstanding freezing delay performance, making them strong candidates as base matrices for further functionalization. These formulations are particularly well suited for integration with silica-based fillers to improve their hydrophobic/icephobic properties, where surface roughness and chemistry can be fine-tuned without compromising mechanical integrity or adhesion. Moreover, their ability to significantly delay ice formation under static conditions highlights their potential as passive elements in hybrid Ice Protection Systems, enabling a reduced energy consumption and lower CO_2 emissions when coupled with active IPS technologies.

5. Conclusions

In this work, base formulations for anti-icing coatings targeting aeronautical applications were successfully designed, synthesized, and systematically evaluated. Two low-surface-energy polymeric matrices, i.e., a silicone–epoxy hybrid and a hydroxyl-functionalized PTFE resin, were investigated using different curing agents to identify room temperature-curable formulations with suitable thermo-mechanical and functional properties.

Thermal, mechanical, and spectroscopic analyses confirmed the formation of crosslinked networks in most formulations, with EF-based systems exhibiting higher glass transition temperatures, a superior thermo-oxidative stability, and a better overall mechanical consistency compared to Z-based systems. All coatings demonstrated an improved surface hydrophobicity and reduced surface free energy relative to the uncoated steel substrate.

However, the anti-icing performance was found to be only weakly correlated with the water contact angle. Instead, the freezing temperature and freezing delay time measurements revealed that the dominant mechanism governing ice nucleation delay may be the thermal insulation provided by the polymeric coating, with hydrophobicity playing a secondary role. Several formulations achieved a freezing point depression of up to approximately 10 °C and suppressed ice formation for more than 60 min under static icing conditions. Among the investigated systems, EF-AM and EF-TC emerged as the most promising candidates, combining strong adhesion, thermal stability, and superior anti-icing performance.

Overall, these results indicate that effective anti-icing behavior may be achieved through polymeric thermal insulation, yielding already low freezing temperatures and extended freezing delay times without relying on extreme superhydrophobicity. However, the relative contribution of thermal insulation to the reduction in freezing temperature and the enhancement of freezing delay time cannot be definitively established at this stage and remains a working hypothesis. Further dedicated investigations are required to clarify the underlying mechanisms. This study therefore provides a solid foundation for further performance enhancement through material design. Future work will focus on the incorporation of silica fillers into EF-AM and EF-TC matrices by exploiting their silica functionalities to enable chemical anchoring. This approach is expected to further reduce thermal conductivity and, consequently, to extend the freezing delay time, strengthening the potential of these coatings for integration into durable hybrid Ice Protection Systems for next-generation, energy-efficient aircraft.

Author Contributions: Conceptualization, F.P.; methodology, F.P., G.F. and V.A.; hardware and software, G.B.; validation, F.P., A.D., G.B. and G.C.; investigation, F.P., A.D., G.B. and G.C.; data curation, F.P., A.D., G.C., V.A. and G.F.; writing—original draft preparation, F.P., A.D. and G.C.; writing—review and editing, F.P., G.C., V.A. and G.F.; visualization, F.P., A.D. and G.C.; supervision, F.P. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by The Italian Ministry for University and Research (MUR) through the National Aerospace Research Program (PRORA) D.I. n° 662/2020 as TECH-ICE project.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding author.

Acknowledgments: The authors gratefully acknowledge M. Costantini of CIRA for his valuable technical support in the development of the experimental setup.

Conflicts of Interest: The authors declare no conflicts of interest.

Abbreviations

The following abbreviations are used in this manuscript:

CIRA	Italian Aerospace Research Centre
IPS	Ice Protection System
PTFE	Polytetrafluoroethylene
FT-IR	Fourier Transform Infrared Spectroscopy
DMA	Dynamic Mechanical Analysis
TGA	Thermogravimetric Analysis
CA	Contact Angle
SFE	Surface Free Energy
FDT	Freezing Delay Time
EF	SILIKOPON EF

Z	ZEFFLE GK
AM	DYNASILAN AMEO
DY	DYNASYLAN 1124
TC	TEGO® Cure 100
ID	Isophorone Diisocyanate
DH	1,6-Diisocyanate Hexane
D33	Desmodur® Ultra N 3300
T _g	Glass Transition Temperature
E' _g	Glassy Modulus
E' _r	Rubbery Modulus

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