



## Ice accretion by spraying supercooled droplets is not dependent on wettability and surface free energy of substrates



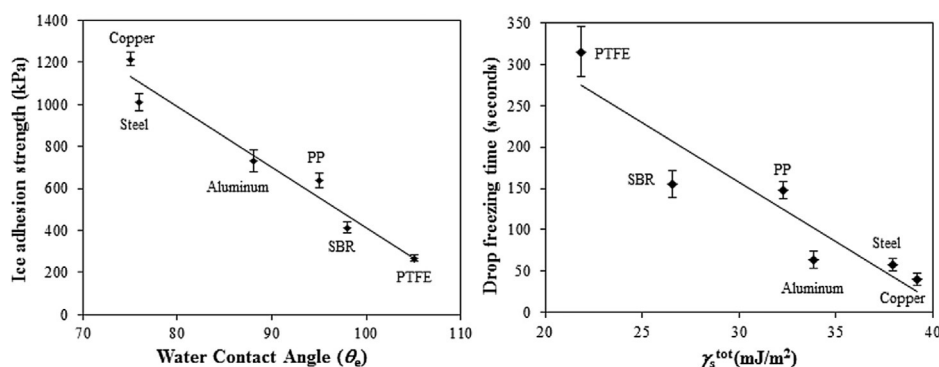
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### HIGHLIGHTS

- Ice accretion, ice adhesion strength and drop freezing time tests were applied.
- Ice accretion results are found to be independent from the surface properties.
- Ice adhesion strengths vary linearly with water contact angle of the surfaces.
- Drop freezing time results vary linearly with surface free energy of the surfaces.

### GRAPHICAL ABSTRACT



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### ABSTRACT

Three different characterization tests namely, ice adhesion strength, drop freezing time and a newly developed ice accretion method were applied to surfaces which were commonly used in anti-icing research such as aluminum, stainless steel, copper, styrene butadiene rubber, polypropylene and polytetrafluoroethylene in order to investigate the effect of their water wettability and surface free energies on their anti-icing performances. It was found that ice adhesion strengths increased and drop freezing times decreased with the increase of the total surface free energies and decrease of the water contact angles on these surfaces similar to previously published reports however, no direct relationship was found between the ice accretion results and solid wettability, surface free energies and the reasons were discussed in terms of heterogeneous nucleation mechanisms.

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### 1. Introduction

Ice accretion on surfaces is a very important problem for human life because this natural event causes the breakdown of many critical systems such as aircraft [1,2], ships [3], offshore platforms [4], wind turbines [5,6], power lines [7] and photovoltaic devices

[8]. There is an intensive anti-icing surface research activity in the world in the last two decades. Supercooling (also known as undercooling) is a process where a liquid and gas cools down below its freezing point without solidification [9] and the main reason of ice formation is the direct contact and adhesion of supercooled water droplets onto a solid surface in sub-zero temperature conditions. The largest natural inventory of supercooled water occurs in the form of small droplets in the clouds and when an aircraft flies through these clouds, supercooled water droplets may easily cause ice accretion on the plane especially on the wings and tails and

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this event can lead to the breakdown of the flying performance and sometimes to aircraft crashing [1]. Thermal or pneumatic energy can be supplied from beneath or outside the surface to decrease the ice accretion for “active” anti-icing systems, however many of these methods are expensive, time and energy consuming and there is the risk of faulty applications which damage the infrastructure [6,10–12]. In the “passive” anti-icing systems where no external energy is supplied, specific anti-icing surfaces were used to reduce the ice adhesion strength [13–19], to delay the freezing time of water droplets [20–33] and to reduce the amount of accreted ice [17,34–45].

The investigation of the effect of surface properties such as water contact angle (WCA), contact angle hysteresis (CAH) and surface roughness onto ice adhesion strength is a well-known research topic. Many types of superhydrophobic surfaces having water contact angles greater than  $150^\circ$  where a water droplet can easily roll off with a small sliding angle (less than  $5^\circ$ ) were fabricated to be used in the anti-icing research in recent years [2,10,29–33,37–46]. Many researchers believed that high water repellent superhydrophobic surfaces would have been successful for producing anti-icing materials by repelling the contacting water droplets and eliminate them before they can freeze however, currently there is no known material that can completely prevent ice from accumulating on a surface. Some researchers hypothesized that superhydrophobic surfaces would have been successful to reduce the ice adhesion strength after icing occurs [15,47–53]. In contrast, many researchers reported that the use of superhydrophobic surfaces were not successful to reduce the ice adhesion strength [18,33,54–58]. Varanasi et al. have reported that frost nucleation occurs on all areas of the textures of a superhydrophobic surface leading to the loss of its superhydrophobic properties and could increase the ice adhesion strength [58]. Kulinich et al. have reported that when ice accretion occurs on a superhydrophobic surface, it causes a gradual damage of the surface microstructure during icing and deicing. In addition, the ice adhesion strength on superhydrophobic surfaces was significantly high especially in a humid atmosphere [56]. Nosonovsky and Hejazi showed that superhydrophobic surfaces which do not possess sufficiently large voids at the interface can have strong ice adhesion [57]. Fang and Amirfazli pointed out that the majority of the published papers discussing the success of superhydrophobic surfaces in antifreezing has been clouded by confluence of different factors and the lack of sufficient characterization methods [46].

In summary, even some superhydrophobic surfaces may exhibit lower ice adhesion strength in some cases, the anti-icing efficiency of a superhydrophobic surface is significantly low in a humid atmosphere where water condensation occurs both on top and between surface asperities leading to high values of ice adhesion strength and the use of superhydrophobic surfaces for anti-icing applications is under question [18,23,33,46,54–59].

On the other hand, the effect of surfaces which are not superhydrophobic, but having varying hydrophobicities on their ice adhesion strengths was also investigated by many researchers [13,14,60–62]. Some researchers reported the effect of surface wettability on drop freezing time delay [20–33]. For example, Hao et al. showed that the freezing processes on surfaces are controlled by nucleation and heat transfer and the freezing delay time of the smooth surfaces with roughness smaller than the size of the critical ice nuclei was found to be much longer than superhydrophobic surfaces with hierarchical structures. The time of freezing after freezing starts depends on the actual liquid–solid contact area and increased with the increase of the contact angle [31]. Poulikakos and coworkers reported that hydrophilic surfaces with nanometer-scale roughness showed unexpectedly long freezing delays, at least one order of magnitude longer than typical superhydrophobic surfaces with larger roughness and low wettability [23,33].

On the other hand, the determination of the ice accretion on surfaces is another important practical parameter to evaluate the anti-icing performance of a surface [6,10–12,17,34–44,63,64]. The minimization (or complete prevention if possible) of the ice accretion is a discriminative test for the success of an anti-icing coating together with the ice adhesion tests. However, only few papers reported ice accretion results on surfaces in mass/area units [17,34–36,39–41,44,63]. In general, photographs were given in most of these papers to compare the relative ice or snow accumulation on different surfaces [10,37,38,42,43,64]. It is possible that the high cost of the laboratory set-ups used for ice accretion tests prevents some researchers to carry out these experiments although ice accretions tests were applied long ago. Tattelman developed a quantitative method to measure ice accretion on surfaces in mass/area units in a climatic chamber in 1982 [34]. Cao et al. reported low ice accretion on a superhydrophobic surface under outdoor conditions under freezing rain [37]. Yang et al. prepared fluoropolymer surfaces and reported that coatings having a smooth surface can significantly reduce ice adhesion strength but did not reduce the ice accretion at  $-8^\circ\text{C}$  [17]. Yin et al. prepared representative superhydrophilic, hydrophilic, hydrophobic and superhydrophobic surfaces and applied ice accretion tests by spraying supercooled water to samples at different horizontal inclination angles, but a correlation between surface wettability and ice accretion was not observed in this work [39]. Yuan and coworkers prepared superhydrophobic polyvinylidene fluoride coatings and applied ice accretion tests by spraying supercooled water onto samples in a climatic chamber and reported that superhydrophobic coatings with low sliding angle exhibited better anti-icing when compared with smooth low density polyethylene and uncoated wind turbine blade [40,41]. Jiang et al. found that ice accretion on styrene-butyl acrylate copolymer latex coatings which copolymerized with different functional monomers were slightly lower than bare stainless steel [35]. Recently, we developed a novel ice accretion test method to investigate the increase of the mass per unit area of formed ice gravimetrically by spraying supercooled water using a spray gun onto various hydrophilic and hydrophobic solvent impregnated surfaces [63].

Although many papers were published on anti-icing surfaces in the last decade, there was no publication in the literature to report the results of ice adhesion strength, ice formation time and ice accretion mass per unit area of the same practical surfaces simultaneously and compare all the results in terms of water contact angles and surface free energies of these test surfaces to the best of our knowledge. In this work, we used some practical surfaces which were frequently used in anti-icing research such as aluminum, stainless steel, copper, styrene butadiene rubber, polypropylene and PTFE and applied three different characterization tests namely, ice adhesion strength, drop freezing time and ice accretion method simultaneously. It was found that the ice adhesion strengths increased and drop freezing times decreased with the increase of the total surface free energies and decrease of the water contact angles however, no direct relationship was found between the ice accretion results and the wettability and surface free energies of the substrates. The reasons are discussed in terms of frost film formation via heterogeneous nucleation of the supercooled drops on substrates.

## 2. Experimental

### 2.1. Materials

Commercial samples of aluminum (Al), stainless steel (SS), copper, polypropylene (PP) and polytetrafluoroethylene (PTFE) produced in Turkey were used for the experiments. Al, SS, PP and

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