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ADHESION OF LABORATORY GROWN SEA SPRAY ICE

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ABSTRACT

Sea spray icing is a major safety concern for operations in polar regions. During a sea spray event several centimeters of sea spray ice can grow within an hour and can cause the inability to navigate and submerging of small vessels. The knowledge about the growth process and the properties of the ice growing in a sea spray event is limited. The adhesion strength of the ice is of particular concern, because it may limit the amount of sea spray ice that can grow before it is dropping due to its own weight. But data about the adhesion strength of spray ice is very limited. On the one hand it is difficult to measure; on the other hand, the focus during a natural spray event is the deicing to ensure operation of the structures. In the MICROSPRAY project at NTNU we perform for the first time systematic in-situ ice adhesion measurements on spray ice. The spray ice has been growing in a cold laboratory with air temperatures from -7 to -15 °C and wind speeds from -10 to -15 m/s. Thus far, data about the adhesion strength of spray ice is very limited. On the one hand it is difficult to measure; on the other hand, the focus during a natural spray event is the deicing to ensure operation of the structures. In this work we present the setup used for the spray experiments and a first overview of the adhesion strength of sea spray ice based on in-situ testing.

KEY WORDS: Sea spray ice, marine icing, ice adhesion strength, brine channels, salinity

INTRODUCTION

The phenomenon of sea spray droplets freezing on objects such as ships, offshore structures and other coastal infrastructure is called sea spray icing. As the droplets freeze, they form a layer of ice, this layer can accumulate to a significant thickness and cause a range of problems. The problems include reduced maneuverability, structural damage and even capsizing of small vessels (Dehghani-Sanij et al., 2017; Samuelsen and Graversen, 2019; Samuelsen, 2018; Sawada, 1968; Shestakova, 2021) . The droplets in a sea spray event can be created by wave structure interactions, for example a vessel slamming into waves resulting in large droplets that are transported by the wind, before impinging on marine structures, this kind of spray is referred to as wave spray (Kulayakhtin, 2014; Dehghani et al., 2016; Dehghani-sanij et al., 2015). Another origin for the droplets can be wind spray, it is generated by the action of wind on the surface of the water. When the wind blows over the the ocean surface it creates small ripples and waves, and can raise some droplets from the wave crests (Dehghani-Sanij et al., 2018; Kulayakhtin, 2014; Jones and Andreas, 2012). Wind spray is typically smaller and less energetic than wave spray and is more strongly influenced by wind speed and direction. During a spray event both wave and wind spray can occur simultaneously, however for icing on ships wave spray plays the major role, while wind spray becomes more important for stationary structures (Jones and Andreas, 2012). In temperatures below the freezing point these droplets can freeze and form an ice layer upon hitting a surface. The salt in the water is not incorporated in the growing matrix but expelled to either run of or be entrapped in so called brine pockets, pores within the salt free ice matrix that are filled with liquid brine of increased salinity (Ozeki et al., 2012).

Adhesion of saline ice

The adhesion of ice describes how strongly the ice is attached to a given surface.

Often the thermodynamic work of adhesion is used. It is derived from wetting a surface with a droplet of water and focuses on the contact angle between surface and droplet as well as the surface energies. The derivation which is done step by step in Makkonen (2012) leads to

$$W_{\mathrm{a}} \approx \gamma_{\mathrm{w}}(1 + \cos\Theta). \tag{1}$$

This means that the thermodynamic work of ice adhesion can be approximated by the surface tension of water and the contact angle of water on the solid. This further explains the reduction of adhesion by hydrophobic and super-hydrophobic surfaces.

However, the work that is spent to separate the interface W_{a} is not strongly correlated to W_{a}, due to the deformation of both ice and substrate during the adhesion failure. Therefor, the adhesion strength (τ)is defined as the maximum value $\mathrm{F}_{\mathrm{max}}$ of F(x) used to separate a interface between substrate and ice of the area A (Rønneberg et al., 2020)

$$\tau = \frac{\mathrm{F}_{\mathrm{max}}}{\mathrm{A}}. \tag{2}$$

τ may be affected by a variety of factors, as the nature and purity of the accreted ice, nature and texture of the surface and icing conditions, including temperature, heat conduction on the interface and mass transport (Laroche et al., 2020). While most ice adhesion research has focused on improvement of the surface properties to reduce the adhesion of freshwater ice, experiments with saline ice show that the adhesion is naturally lower (Makkonen, 2012; Ozeki et al., 2012). This may partly be related to the brine pockets reducing the effective surface area. But, the adhesion is even lower as expected purely by the porosity. To explain the effect Makkonen the introduced the concept of a brine layer forming on the interface, which would further lower the adhesion (Makkonen, 2012).

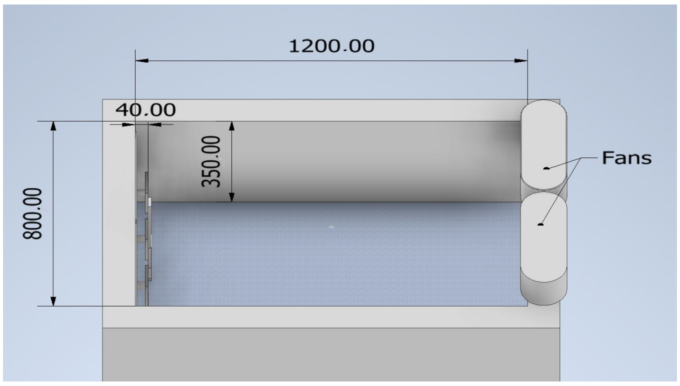
EXPERIMENTS AND METHODS

The experiments were performed on spray ice samples grown in a cold laboratory at NTNU, in temperatures from -7 °C to -15 °C. To produce the spray two strong fans (10-15m/s wind speed) have been directed on the surface of a tank (120 cm x 80 cm) filled with saline water (3 w% NaCl), a schematic of this tank can be found in figure 1. The spray travels trough the cold air and impinges on the back wall of the tank, as well as on sample plates placed in front of the back wall. It freezes on these surfaces and forms a layer of spray ice. After the spray experiment the spray ice-adhesion on the plates is measured using an in-situ setup, which is described below.

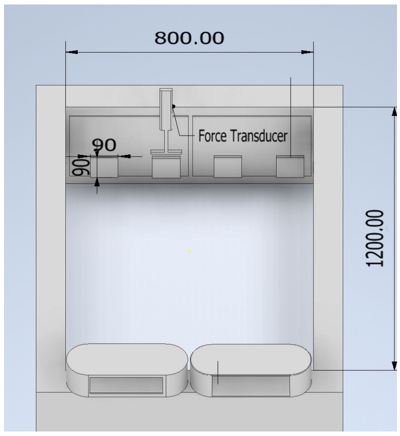
Spray ice experiments

Using the tank with saline water and fans we are able to generate spray in the laboratory. The tank is situated in a cold laboratory to be able to control and monitor the air temperature. To grow spray ice, spray has been generated at air-temperatures of -7 °C, -10 °C and -15 °C and the water temperature at the melting point. One cm thick square shaped sample plates of aluminium with an edge length of 3 cm, 6 cm, 9 cm and 12 cm where used to grow the ice on. These are mounted on aluminium cylinders of 5 cm length to separate the ice growing on the back wall of the tank and the ice growing on the plates.

The spray in this experimental setup resembles wind spray, it is generated by the fans blowing on the water surface, not by wave interactions. The generated droplets have a short time of flight



a



b

Figure 1: Schematics of the experimental setup **a)** viewed from the side and **b)** viewed in spray direction. Annotations in mm.

before impinging on the plates because of the dimension of the tank. Thus, the water in our experiments needs to be close to the freezing point before the start of an experiment. This results in a change of spray rates during the experiment as an slush ice cover builds up on the far end of the tank and grows towards the generation zone.

in-situ Adhesion test

The test setup is constructed for a vertical shear test and consist out of a „GLIDEFORCE LACTP-12V-20“ light duty linear actuator and a load-cell which are mounted above a plate in the tank used for spray icing experiments compare figure 2. During the test the linear actuator moves with a set speed of ca. 14 mm/s, while the load-cell records the force which is applied. The setup was created for the experiments because the low adhesion of the spray ice samples prevented to move the samples to the existing adhesion setup at NTNU.

a

b

Figure 2: **a)** Adhesion test setup for in-situ spray ice adhesion testing. Mounted on top of a saline ice water tank. Photo: Eirik Samuelson; **b)** 6 cm sample plates during a spray experiment. Ice is forming on all 4 plates, but it is thickest on second plate from the camera position. Underneath the plates slush ice has formed.

In the adhesion test the force transducer pushes at the ice, that has been growing on the sample plates, until the ice detaches. The adhesion strength (σ) is the quotient of the maximum force (F_{max}) during the experiment and the (front) surface area (A) of the sample plate.

Testing the adhesion setup

Adhesion test results depend on the chosen test method, which makes it difficult to compare results from different test setups without comparing ice of the same type and conditions (Laroche et al.,

2020; Rønneberg et al., 2020).
There is no data-set on the adhesion of sea spray ice, so we used bulk water ice samples of distilled water, tap water and saline water (S=32 w‰), that was also used for the spray experiments, to test the setup. To grow the samples water of the respective salinity was filled in cooled plastic cylinders of 2 cm radius which were positioned on cooled aluminium surfaces. Afterwards the samples stayed untouched in the laboratory at -15 °C for 6 hours, before being tested. For all three salinities 2 samples were prepared. Later, 3 more samples were added for the saline water, two additional samples in the same cylinders and one sample in a larger cylinder with 4.05 cm radius. The tests with the bulk water ice samples where conducted at -15 °C.

RESULTS

The test setup

The results of the adhesion strength test with bulk water ice are presented in table 1. The measured adhesion strength ranges from 210 kPa for desalinated ice too 7 kPa for saline ice. The sample size is too low to have a statistical significance, but allows to evaluate the test setup.

The test of the samples with different salinities shows that adhesion strength decreases with increasing salinity as has been shown before (Makkonen, 2012; Ozeki et al., 2012). It wasn't possible to measure the adhesion of the saline samples with the same contact area as used for the other samples, because the ice detached before testing was possible. Thus, we increased the contact area by using a larger container to grow the bulk ice in and it was possible to measure the adhesion strength for an ice sample with high salinity. The resulting adhesion strength is significantly lower than the adhesion of ice samples with lower salinity. This however is the result of one successful test so far.

Table 1: Overview over the bulk ice adhesion tests. The salinity (Sal.) of the samples was determined once per sample type, max Load describes the maximum load recorded by the force transducer, the displacement (Disp) describes the extension of the force transducer when the max load occurred. The ice adhesion strength τ_{max} is calculated from equation 2 with the contact area.

Sample	max Load [N]	Area [cm^2]	τ_{max} [kPa]
Aqdest1	157.8	12.57	126
Aqdest2	263.4	12.57	210
Tap1	172.8	12.57	138
Tap2	142.1	12.57	113
Saline5	37.1	51.53	7

Sea spray ice

Sea spray ice is growing on the plates during the spray experiments. The ice thickness of the resulting sample is not equal over the height of the sample, but thinner on the top and getting thicker to the bottom of the sample. Furthermore, the icing is not equal between the 4 plates that are in the tank simultaneously as can be seen in figure 2 b. This is related to different amounts of spray reaching the plates in the experimental setup.
Ice doesn't only grow on the forward pointing surface of the plates, but also grows around the edges of the sample plate, the extend to which the sides of the plate is iced vary. Some samples form icicles during the spray experiments. These icicles align with visible brine channels on the former aluminium-ice-interface (figure 3). The formation of large icicles was not observed on the samples plates with 3 cm and 6 cm edge-length.

The microstructure and brine distribution of the samples is discussed in our other POAC-contribution Sea spray microstructure (Maus et al.2023).

Figure 3: A sample after the adhesion test. Icicles formed on the lower end of the sample, brine channels are visible with eye and camera on the former interface. Small ice walls on the side of the sample show that icing around the edges occurred.

Adhesionstrength

The results of successful adhesion tests, e.g. where the ice sample wasn't falling of before the adhesion measurement could start, are presented in figure 4. The adhesion strengths are grouped by the size of the ice samples, the standard derivation between samples of the same size is plotted in the error-bars. All measured adhesion strengths were below 60 kPa. For samples sizes larger than 6 cm all measured adhesion strengths were below 20 kPa.

There were two different reasons for unsuccessful adhesion tests. The ice was to thin to have a sufficient contact area between force transducer and ice, causing the transducer to slide on the ice surface instead of detaching the ice. This was observed repeatably at sample position 4 (the furthest plate in figure 2 b), which was reached by smaller amounts of spray. The second reason was that ice detached from the plate before the adhesion test started. An detachment without external force occurred at high air temperatures for example on 3 cm and 12 cm samples at -7 °C.

Figure 5 shows the adhesion of the samples plotted over the brine volume fraction of the adhesion strength for samples grown at -15 °C and -10 °C. It does not show a correlation between brine volume fraction and adhesion.

DISCUSSION

Sea spray ice from cold laboratory experiments

The experimental setup is suited to grow spray ice in a laboratory allowing for a large data set of ice from different growth conditions and the testing of the spray ice's adhesion, but it has some limitations regarding possible icing conditions. For example is the initial water temperature limited

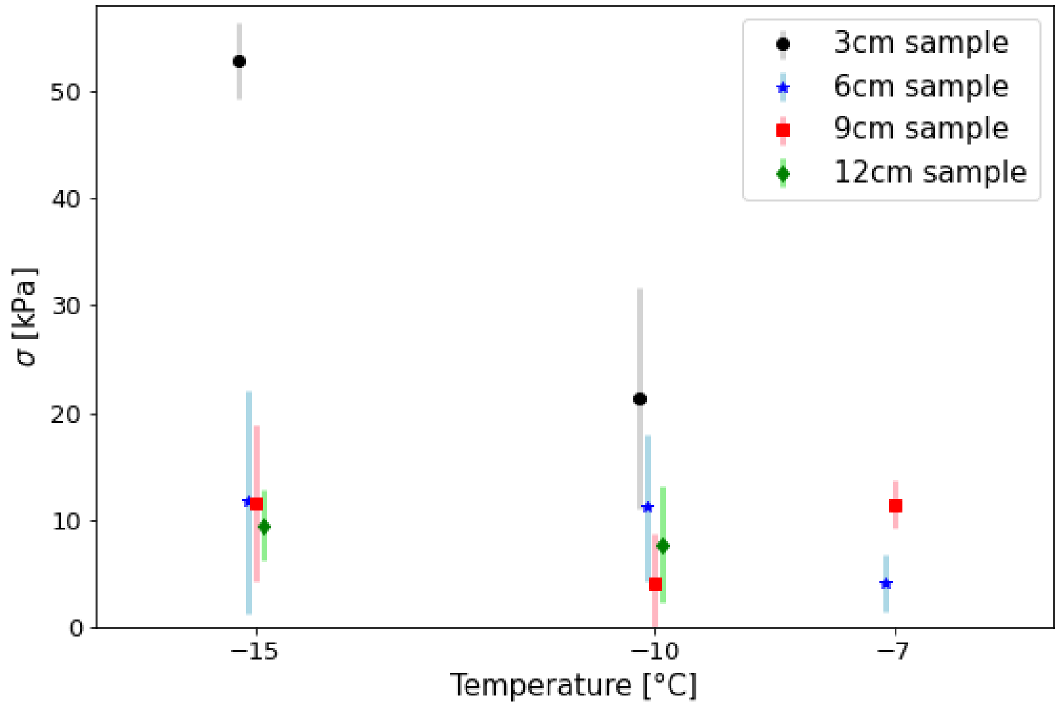


Figure 4: Overview of adhesion strengths of spray ice samples of different sizes.

to temperatures close to the melting point. If the water is too warm is it not only possible that the droplets are not freezing on the plates before they run off, but waves can build up in the tank and remove already growing ice from the plates. With temperatures close to the melting point some ice crystals in the water prevent the buildup of large waves. This makes it unsuited to test icing for reported conditions with water temperatures above the melting-point. The limitation to wind spray makes it easier to compare the results of differently sized samples with each other as wave structure interactions do not occur.

The visible brine channels and liquid residue at the interface show the brine rejection of the saline ice in macroscopic scale. The channels on the interface reduce the effective area of the interface between ice and aluminium.Brine channels of the form as in figure 3 are absent in 3 cm samples. Possible explanations are (i) that brine channels require a critical size to form, (ii) a higher heat transport from smaller samples due to higher specific surface area and additional heat flux from the cylinder used for mounting and (iii) the perimeter to area ratio of small samples (The absence of visible brine channels in 3 by 3 cm samples indicates that there is a critical size for the brine channels to form. The brine might not have enough space for pores to interconnect and form channels through which a lot of brine drains out of the sample increasing the size of these channels and allowing more brine to drain out. But, the brine pockets should be smaller for them not to form an interconnected network. Therefore, the absence could be explained with a higher temperature transport away from the surface leading to faster freezing and less time to eject brine in the process. This higher temperature transport could be explained by the plate being approximately the same size as the cylinder used to mount the sample.

Adhesion strength of sea spray ice

Measuring the spray ice adhesion is possible due to the in-situ test setup, allowing for the grown ice to be tested without moving or handling it. That all handling of the samples leads to detachment of the ice, shows that the adhesion is generally low.

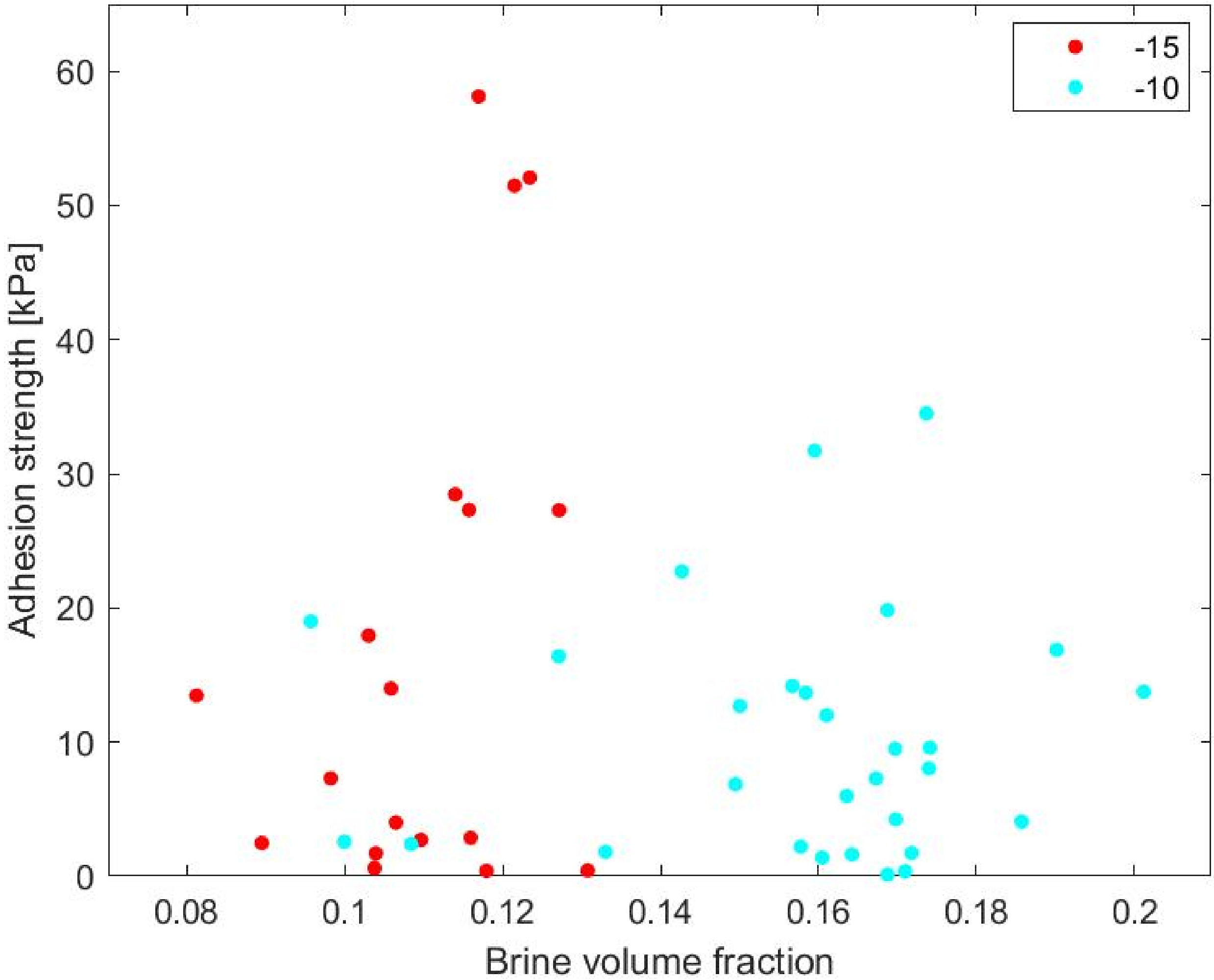


Figure 5: Adhesion strength of samples grown at -15 °C (red) and -10 °C (blue) plotted over the brine volume fraction of the samples.

Other authours have observed that the adhesion strength drops by one or two orders of magnitude when salt is added. Such a reduction already happens for solute concentrations of a few ‰ NaCl (Zhang et al., 2022; Sackinger et al., 1987; Makkonen, 2012).

The porosity of saline samples both due to air bubbles and brine pockets has been used to explain the lower adhesion, but the porosity alone could not explain the reduced adhesion observed in saline ice (Makkonen, 2012). In our spray ice samples no correlation between adhesion and brine volume fraction of the growing ice has been found (figure 5), which is further supporting that the porosity is not exclusively responsible for reducing the adhesion strength.

Makkonen (2012) proposed that a saline layer on the interface is responsible for the low adhesion. We observed a liquid residue remaining at the former ice sample interface. This is an indication that the low adhesion could indeed be explained by a saline layer at the interface. However, the dimension of this layer has not been experimentally determined. A similar layer at the interface has been found by Zhang et al. (2022) in adhesion experiments with seawater on airplane tyres. They used the existence of the layer and expected variations in the thickness of the layer to explain the different behaviors of saline water at different temperatures, based on Makkonens work. But, as in our experiments the thickness of the layer has not be determined experimentally (Zhang et al., 2022).

The adhesion of the smallest samples is significantly higher (at values of 50-12 kPa) than the values of larger samples. This could partly be explained by the absence of brine channels resulting in a smaller reduction of the surface area. Additionally, ice that grows on the sides of the sample plate instead of the front surface makes out a larger percentage of the total area, leading to an higher overestimation of the adhesion for smaller samples. Only considering the front surface in the calculation of the adhesion strength, while some ice grows on the sides of the samples, leads to an overestimation of the adhesion strength.

The adhesion strength for all larger samples is between 2 and 20 kPa, and with that in a low adhesion regime. The adhesion of the high saline bulk sample is 7 kPa and with that in the same range as sea spray samples. A decrease in adhesion strength is observed with increasing temperature. This can be explained with the ice being closer to the melting point, having a comparable higher amount of liquid brine,increasing both the brine pocket size and thickness of the liquid layer

Adhesion measurements often show a high variance, even for bulk water ice samples due to outliners in experiments (Rønneberg et al., 2020). The growth of sea spray adds additional factors compared to the freezing of bulk water ice, that can influence the growth of the ice and its adhesion. For example will the water level inside the tank change slightly during experiments resulting in variations in the spray flux reaching the surface during the experiments.

CONCLUSIONS

To gain a better understanding of the ice formation and adhesion of sea spray ice we performed spray experiments and adhesion tests in cold laboratories at NTNU. The ice was growing on aluminum samples to establish a base line for the adhesion strength of sea spray ice, which can be extended by using coatings or other substrate material.
The experiments show a low adhesion of saline spray ice on aluminium. The low adhesion can be explained by brine channels and a brine layer on the interface, but additional characterisation of the brine layer will be necessary to determine how it is effected by the growth conditions. Experiments with a higher spatial resolution and time resolution, could allow to study the interface and the brine layer.

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