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Investigating surface coating effects on the ice adhesion behavior of woodchips

A Nordic Icing Centre of Expertise report

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Investigating surface coating effects on the ice adhesion behavior of woodchips, A Nordic Icing Centre of Expertise report

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Abstract

This project investigates the effect of surface coating on ice adhesion of frozen woodchips. The study was initiated by challenges faced by forestry workers in winter, specifically the storage and transportation of woodchips from the forest to the plant.

The first part of the report presents a review of such available methods of anti-icing and de-icing as chemical spray, container lining, thermal coating, industrial vibrators and heating system.

The second part of the report deals with experimentation. This part is divided into two different testing protocols at -10°C. In the first protocol, 5 boxes are made with different coatings to evaluate the detachment time from the shear of the bottom plate at room temperature. The coatings include FS-SLIPS, PE Quicksilver lining, FS-PE rough, FS-PE smooth, steel covered with rapeseed oil and steel with sprayed silicone oil. In the second protocol, 9 surfaces are investigated with centrifuge ice adhesion tests (CAT) at -10°C, where the coatings include the same materials as the boxes as well as uncoated steel and steel covered with antifreeze and PTFE tape.

Both protocols result in similar trends regarding the FS-SLIPS + silicone oil. It has fast ice removal and low ice adhesion which make it the most promising results.
This report gives a comprehensive overview of the existing methods on ice-adhesion. Every solution tested gives a lower ice-adhesion than stainless steel.

Language: English
Key Words: Woodchips, ice adhesion, surfaces, thermal coatings, winter, transport, storage

Table of abbreviations and symbols

Abbreviation	Meaning	Page
AMIL	Anti-icing Material International Laboratory	4
CAH	Contact Angle Hysteresis	9
CAT	Centrifugal Ice Adhesion Test	4
CH_2O	Formaldehyde	6
FDA	Food and Drug Administration	7
DMSD	Dimethylsilanediol	6
FS-PE	Flame sprayed polyethylene	4
FS-SLIPS	Flame sprayed slippery liquid-infused porous surface	4
IA_Av	Ice Adhesion average	22
IA_STD	Ice Adhesion standard deviation	22
LD-PE	Low density polyethylene	14
MS%	Moisture content in percent	18
NoICE	Nordic Icing Center of Expertise	1
PE	Polyethylene	2
PDMS	Polydimethylsiloxane, mentioned as silicon oil	6
PTFE	Polytetrafluoroethylene, mentioned also as Teflon	13
SiO_2	Silicon dioxide	6
TAU	Tampere University	2
TS	Thermally sprayed	9
UHMWPE	Ultra-High Molecular weight polyethylene	4
UQAC	Université du Quebec à Chicoutimi	4

1 Introduction

Woodchips are pieces of wood between a size of 2 to 5 cm, made by chipping branches, stumps, roots, wood waste, etc., with a woodchipper. There are many types of woodchippers that include disc, drum and other mechanisms. Woodchips may be used as a source of bioenergy, in gardening and in mushroom cultivation as well. In most cases, transportation of woodchips to the customer is necessary when the chipping is made on the lumbering site.

At subzero temperature, woodchips have the tendency to aggregate due to their moisture content. The free water and bound water present in the wood will freeze and create a bond between chips resulting in bigger lumps. The size of woodchips is a factor of icing as small woodchips freeze easier.

Forest workers face ice troubles in winter. The NoICE project aims to solve selected issues related to this, such as the problem caused by the woodchips freezing and attaching to the trailer walls. As the NoICE project goal explains:

> *The cold climate causes various problems for Nordic companies and organizations. Wintertime icing causes some systems to function abnormally or not at all. This in turn might cause increased operating and maintenance expenses. So far there is no overall structure working to solve problems related to wintertime icing in the concerned region. As a result of this problem solving is not systematically practiced and recent progress within related research has not been fully implemented in industry. NoICE will gather existing and develop new research related to problems caused by wintertime icing and implement it on key-industries. This knowledge will then be shared with project stake holders e.g., within the business networks.* (NoICE, 2021)

The company Merinova has been hired to conduct a survey among companies and their troubles with ice and icing conditions. Many sectors have been investigated but the most commonly occurring was the forestry. Companies in the forestry sector are facing multiple troubles from harvesting, chipping, transport until storage. (Wasberg, 2019). Troubles with frozen and attached woodchips in transport containers in winter are the main concern. Troubles with ice formation on forest machinery windscreens and on undercarriage structures of transport trunks increased the weight on the trailers and therefore reduce the loading capacity. Companies expressed the need to reduce ice adhesion to be able to work with full load, decreasing the number of loads per transports and decreasing the economic losses.

There have been previously many attempts to study ice removal related to transportation and logistics. For instance, Ryerson, 2013, conducted research to avoid and/or remove ice adhesion on marine vessels, airplane wings and other vehicles (Ryerson, 2013). In addition, nowadays, companies are spraying fuel oil on walls as “coating”. Due to its physical properties such as a low freezing point, diesel is sprayed in containers to prevent woodchips from freezing on the walls. Unfortunately, diesel is not sustainable. (Springer & Schuchard, 2015). Different anti-icing and de-icing systems will therefore be evaluated in this project.

Below is a list of existing techniques on which we will focus and their usability on containers. As all the previous studies only focused on ice, this review will have a focus on frozen woodchips and evaluate the adaptability of the following techniques:

- Chemical
- Container design improvements
- Surface engineering and chemistry
- Mechanical methods
- Thermal system

Some anti-icing and de-icing techniques that are available on the market and are used on boats and airplanes are not included in this report. Techniques like hot water, hot air, laser or infrared heating (Ryerson, 2013) are not applicable to frozen woodchips or are counterproductive like hot water or steam as we try to maintain woodchips moisture as low as possible.

In this report, we show how maintaining a liquid layer between the wall and the woodchips could help avoid the adhesion. The first part is centered on a review of the existing methods adaptable to container and frozen woodchips. The second part of this report is the experimental part. The tests were conducted at the ICE Laboratory at Tampere University (TAU). The tests were focused on chemical application, polyethylene (PE) lining and surface engineering (icephobic coating) approaches.

1.1 NoICE project

NoICE is a project that will gather and develop wintertime icing expertise to benefit stakeholders in the Botnia-Atlantica region. The first goal of the Nordic Icing Centre of Expertise intends to create a permanent competence center, which will focus on minimizing problems related to atmospheric icing.

The second goal of the center of expertise will be set to gather and share knowledge on icing observation, challenges and solutions. By putting in contact private companies that are concerned with icing problems and a research center that can offer solutions and expertise on icing situation, the Nordic Icing Center of Expert will create a broad communication network aiming to address the existing and potential issues.

The project partners are: Luleå University of Technology (Sweden), Umeå University (Sweden), University of Vaasa (Finland), Tampere University (Finland) and Novia University of Applied Sciences (Finland). The project is funded by Interreg Botnia-Atlantica, Region Västerbotten (Sweden) and Österbottens förbund (Finland), for the period between September 2018 and February 2022.

1.2 Limitations

Ryerson (2013) listed many systems to avoid icing on coast guard assets. Methods that use geometry, nature of surface, protective covers and de-icing products are successful in either avoiding the buildup of ice or removing the accreted ice. Unfortunately, not all the methods are usable for woodchips containers. In his review, the technologies are listed according to their design application and are reviewed in connection with their possible application to the marine environment. The review will only focus on applicable technology to woodchips containers. Due to the geometry of the container and the position of the woodchips, any treatments involving de-icing chemicals and hot water cannot be used without contaminating a larger portion of the woodchips and increasing their moisture content which will cause to decrease the boiler efficiency. Only chemicals which are sprayed on the walls of the containers prior to the loading of the material can be used.

De-icing chemicals like chloride salt sprayed in the containers would have a good result to create a liquid layer between the woodchips and the metal due to their freezing-point depression (around -7 to -10°C). Unfortunately, chloride salt causes corrosion to metal and damage to plant at high concentration. (Ryerson, 2013)

The use of cover can be effective to reduce the moisture content and enhance the drying process of the woodchips. Although this can help when placed on top of a wood pile during snow melting and raining season and removed once the weather reaches a higher temperature and drier environment, a constant cover on a container might cause aeration trouble and decrease the necessary air flow through woodchips thus being counterproductive and limiting the drying process. Another solution would be using the cover inside the container as a wall cover, which will bring the same result as using anti-adhesion coating without the risk of breaking the cover in the process of loading and unloading the woodchips.

2 Theory

2.1 Literature review

In wintertime, woodchips transportation may entail technical difficulties due to the adhesion of woodchips to the container. To be able to compare the ice adhesion of different surfaces, similar methods should be used. The results obtained from shear tests and traction tests can vary greatly.

Work and Lian (2018) made a critical review of the available methods to analyze and quantify the ice adhesion on solid substrates (Work & Lian, April 2018). The methods mentioned include centrifugal adhesion test (CAT), developed in 1946 by Loughborough (Loughborough & Haas, March 1946) and modernized by the Anti-icing Material Laboratory (AMIL) by Laforte and Beisswenger in 2005. The AMIL of the Quebec University in Chicoutimi (UQAC) has developed a CAT machine. The ice adhesion is determined by the rotational speed necessary to detach the ice accreted on the sample. This sample is attached on a balanced rotational beam, weighted and installed in the machine where a rotational acceleration rate of 300 rpm/s is applied. When the ice detaches and hits the centrifuge cover, two piezoelectric cells attached to the side of the centrifuge vat will pick up the vibration and signal to the computer to stop the recording. The shear force necessary to remove the ice is then calculated by the centrifugal force divided by the iced area. The research team performed several tests on aluminum to evaluate the performance and validity of the instrument. (Laforte & Beisswenger, 2005).

Koivuluoto et al (2020) studied the effect of thermally sprayed (TS) icephobic coating on ice adhesion by accreting ice in a wind tunnel. The sample plates were cooled down at -10°C prior to be glazed with purified water (ELGA Veolia, 2021) in the wind tunnel. Their paper contains the studies of several surfaces divided in different groups, such as flame-sprayed (FS) coatings, bulk polymers, bulk metals, commercial paints and references. The ice adhesion strength is determined by CAT. The CAT determined classification is extremely low when the adhesion is below 10 kPa, low below 50 kPa, medium-low below 100 kPa, medium below 150 kPa and high when higher than 150 kPa. The Slippery Liquid-Infused Porous Surface (SLIPS) + silicone oil shows the lowest ice adhesion below 50 kPa. Flamed-sprayed polyethylene (FS-PE) and bulk material ultra-high molecular weight polyethylene (UHMWPE) show medium-low ice adhesion. This study also shows a high correlation between hydrophobicity and icephobicity, as low and medium-low adhesion surfaces are hydrophobic and high ice adhesion metals are hydrophilic. (Koivuluoto, et al., 2020).

Another method used to test adhesion of ice and woodchips on substrate is shear test. There are not many studies regarding the shear test method of ice adhesion of woodchips on container walls. A relevant publication about frozen woodchips adhesion on wall of containers is from J.E. Mattsson (1996) where the author studied the strength of frozen woodchips attachment. The author used shear test to determine the force required to break the ice layer between woodchips and wall. This was tested by freezing woodchips in 1200mm x 187mm tube on vertical wall at -25°C on one side and +25°C on the other side simulating the heat from biochemical decomposition. A force was applied parallel to the wall and pointing downward. Three surfaces were tested: smooth surface concrete, urethane rubber and coated plywood. The moisture content was also measured to determine the effect on ice adhesion. The results showed a correlation between the moisture content and the adhesions, and the higher moisture content, the higher adhesion strength. Yet no differences between the wall material studied were noticed (Mattsson, 1996).

Regarding the woodchips adhesion, Föhr et al (2015) studied the difference between metal and composite trailers filled with woodchips and stored at -30°C for 12 hours and 24 hours. They used a sensor to determine the progression in the temperature drop at different positions in the container. Three tests were made using a metal container, a metal container with a special coolant (EC1) made in Sweden, and a composite trailer. The woodchips moisture was between 47 and 49%. Unloading the containers revealed a 50-60 cm layer of frozen attached woodchips on the bottom of the metal containers even with sprayed on coolant. The composite trailer did not show any woodchips adhesion on the surface after unloading (Föhr, et al., 2015).

2.2 Industrial supply chain of forest chips

Kärhä (2011) studied the supply chains and production machinery of forest chips in Finland. The study identified the following chains for wood chipping: *terrain chipping* when processing the wood at the harvesting site; *roadside chipping* when processing the wood at the roadside with a chipper and transport with a separate truck or an integrated chipper-chip truck; *terminal chipping* when processing the wood at a terminal before transport to the plant; and *chipping at the plant* where the raw material is transported to the plant where it is then processed. All these methods have their strengths and weaknesses; winter conditions appear as a weakness for the terrain chipping but as a strength for the terminal chipping. That is why it is more recommended for winter chipping.

This study shows an increase in woodchips production and consumption from 2004 to 2006 with a projection up to 2025. According to the study, total energy values of forest chips are as follows: 6,5 TWh/year in 2007, 20 TWh/year in 2020 and 25 TWh/year in 2025. (Kärhä, 2010). In 2020, the ministry of agriculture and forestry of Finland reported a consumption of 8,2 million cubic meters of forest chips. They were used to generate heat and power. With a solid wood fuels generation of 19,4 million solid cubic meters representing 37,6 TWh, the forest chips are accounted for 15,9 TWh. The difference in energy consumption of woodchips for 2020 is possibly due to the decrease in the use of stumps (Ministry of Agriculture and Forestry of Finland, 2020).

Our work concerns every chipping supply chain where the woodchips can be left in the container in cold winter before transportation or utilization.

2.3 Available methods adaptable to containers

2.3.1 Sustainable chemicals

"Sustainable chemistry is a scientific concept that seeks to improve the efficiency with which natural resources are used to meet human needs for chemical products and services. Sustainable chemistry encompasses the design, manufacture and use of efficient, effective, safe and more environmentally benign chemical products and processes." (Organisation for Economic Co-operation and Development, 2018)

Nowadays, companies use to spray fuel oil on walls as “coating”. Due to its physical properties, diesel is sprayed on containers prior to loading to avoid woodchips to freeze on the walls. The low freezing point and low water solubility of diesel make it a suitable chemical to avoid frozen woodchips adhesion. Unfortunately, as a petroleum product, diesel is toxic for the environment. Different systems will be evaluated in this project.

The selection of chemicals below is accounted for their biodegradation that allows to avoid any contamination in land, their low freezing point that makes it possible to avoid solidification on contact with the container, their sufficient viscosity at low temperature that facilitates the spreading. Their combustion point should also be lower than the furnace temperature of an average burner, which is around 600°C (Torres-Fuchslocher & Varas-Concha, 2015).

2.3.1.1 ***Silicone oil***

Unlike diesel, silicone oil (most common molecule being polydimethylsiloxane, PDMS) has various applications and moderate toxicity for human and environment. It has been found to have no adverse effect on wildlife or nature. (Goddard & Gruber, 1999). It degrades in soil as dimethylsilanediol (DMSD) which then volatizes to the atmosphere where it is decomposed into carbon dioxide CO_2 within a few days by sunlight-induced reactions. (Dow Corning, 1998)

The silicon oil would not affect the burning capacity of the woodchips as the oil degrades at high temperature with an ignition point at 460°C. Its combustion produces CO_2, silicon dioxide SiO_2 and formaldehyde CH_2O. Also, as the quantity of silicone oil on the woodchips would be small, it will mostly degrade during storage. (SANTA CRUZ, 2009)

With a fusion point around -40°C, the silicone oil can be sprayed on the container’s walls easily with a garden pressure pump sprayer. For example, the company Würth produces silicone spray that makes it easier to apply but contains flammable aerosol which can build-up of dangerous fumes in case of fire. (Würth, 2014)

2.3.1.2 ***Rapeseed Oil***

Due to its commercial availability, rapeseed oil would be an interesting alternative to silicone oil. Similar characteristics can be seen in rapeseed oil.

Rapeseed oil has a solidification point between 0°C and -15°C and can be temporary supercooled to -14°C to -15°C before it begins to solidify. Therefore, it should not cause any trouble during the spraying on the container's walls during winter if the oil is stored in a warm room. (Die Deutschen Versicherer, 1998-2021)

Spilled vegetable oils have a negative effect on environment and wildlife. Although unpolymerized vegetable oils are rapidly and completely biodegradable in sediment, the biggest hazard is smothering. Large spills of vegetable oils can cause fatal damage to wildlife. (National Oceanic and Atmospheric Administration, 2019)

The South Australian Metropolitan Fire Service describes the auto ignition of vegetable oils as heat production during their oxidation. If the heat cannot be dissipated, the temperature will increase until reaching the auto ignition point. (Government of South Australian, 2020). Due to the rise of the temperature during degradation, rapeseed oil has the capacity to auto-ignite (self-ignition temperature 424°C). (Buda-Ortins, 2011).

When it comes to the auto ignition of rapeseed oil, this characteristic is not a problem as the quantity of oil used to coat the container is small. Also, the comparison with the previous use of fuel oil never caused any known trouble from self-ignition on forest site or in a container, although it has a much lower auto ignition temperature (210°C).

Considering the low quantity of rapeseed oil on woodchips combined with its burning capability, this solution should not cause any trouble to the boiler as the oil will mostly degrade during the storage of the woodchips. Thus, there should not be any traces of oil left on the woodchips during burning.

2.3.1.3 ***Antifreeze***

The two main antifreezes additives are ethylene glycol and propylene glycol. Usually used in car engine as coolant and to avoid winter freezing, ethylene glycol has a freezing point of -12,9°C and propylene glycol -59°C. For those specificities, they are good candidates to avoid ice formation at the moist woodchips/trailer walls. They are both biodegradables and they break down into carbon dioxide and water (NORA, 2021). But ethylene glycol is hazardous for human and wildlife in case of ingestion (Thermo Fisher Scientific, 2010). On the contrary, propylene glycol has the same properties as ethylene glycol, but is recognized by the Food and Drug Administration (FDA) as generally safe and is also used as food additive. (ATSDR, 1997)

Propylene glycol has a flash point at 101°C, a boiling point at 188°C and an autoignition at 420°C. Those characteristics do not create trouble for woodchips burners. (Ipcs Inchem, April 2014). For the rest of the report the term antifreeze refers to propylene glycol.

2.3.1.4 ***Icewick tile***

The Icewick system has been developed as a tile infused with anti-icing liquid. It uses a porous material incorporated within the tile matrix. The pores are connected to a reservoir of potassium acetate to prevent the formation of ice. The reservoir will feed the tile from below if placed adjacently or if remotely connected with a pump. A key capability of the system is that the meltwater can be absorbed along with the diluted anti-icing fluid, rather than flowing to adjacent surfaces where it could cause problems such as freezing next to the tiles and therefore just moving the ice problem a few centimeters as seen in Figure 1.

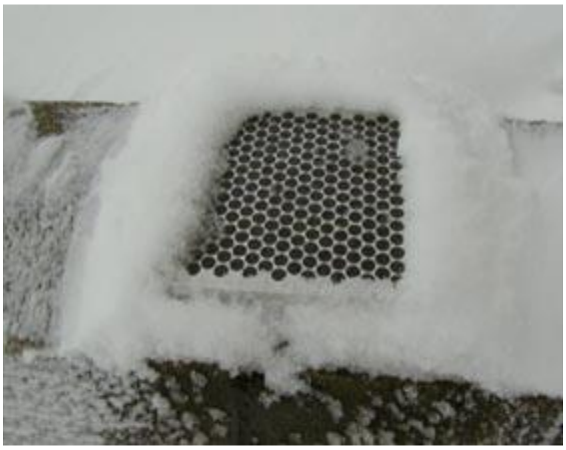

Figure 1. Icewick Anti-icing Grate prototype melting snow (IDI 2007)

Potassium acetate is a freezing point depressant fluid. Its hygroscopic nature maintains the appropriate chemical potency in a changing moisture environment. It cannot dry out or over-dilute from humidity. Since potassium acetate has a sufficiently low corrosivity, it can be used on aircraft runways; it is applied as a liquid to temperatures as low as –29°C. (Innovative Dynamics inc., u.d.)

Potassium acetate is safe for the environment, biodegradable at low temperature and shows a low toxicity to fish, mammals and vegetation. (Chemical Solution INC, 2005)

2.3.2 Polyethylene lining and Composite trailers

The ice adhesion of PE is much lower than stainless steel (Niemelä-Anttonen, et al., 2019). PE lining is used in dump trucks to avoid the adherence of moist materials such as sand or soil during the tipping of the load. Made from UHMWPE, it has a low friction coefficient and therefore does not require any additional release agent (Quadrant Engineering Plastic Products, 2011). Some companies offer the lining of the trailer with UHMWPE to avoid problems in winter. The Swedish company ScanLining AB has tested the Quicksilver lining in northern Canada at -45°C with good results. The experiments have shown that the load does not freeze to the surfaces of the container, which eliminates the need for exhaust heat, vibrators or such other less environmentally friendly solutions as pouring diesel or forming oil into the flatbed before loading. (Scan Lining AB, 2017)

Another solution is to use full composite trailers. The composite containers from Fibrocom ltd are made of combinations of (synthetic) resins and synthetic fibers strongly bound together with diagonal laminate and a cross section of I-beams side by side for reinforcement. A team from Lappeenranta University was conducting tests in a cold room to identify the problem occurring during the transportation of woodchips in composite containers in comparison with metal containers in winter. In both cases, frozen lumps of woodchips are present. The woodchips freeze to the inner surface of the metal container, which make it impossible to unload around 14% of them. Due to the slippery inner surface of the composite containers, the unloading is easier. (Föhr, et al., 2015)

Using a composite container would reduce numbers of troubles during winter. Also, composite containers are lighter, which reduces the total weight of the truck and makes the gas consumption more efficient. The disadvantages, however, include the high cost of materials, manufacturing and assembly. In addition, composite containers have a greater susceptibility to damage and their repairing is more difficult in comparison to metal containers. (Tawfik, et al., 2017)

2.3.3 Surface coatings

2.3.3.1 *Hydrophobic*

On superhydrophobic surfaces, water droplets show a very high contact angle (greater than 150°) that explains the low wettability of the surface. Contact angle is measured at the interaction between the solid, liquid and gas phases. The shape formed by the droplet of liquid shows an angle with various value that depends on the wettability of the surface. The higher the value, the lower the wettability. There is a correlation between a low wettability and a low ice adhesion. (Meuler, et al., 2010)

Commercial superhydrophobic paints UltraEverDry and NeverWet have a low ice adhesion value measured on CAT, which respectively amounts to about 40 and 68 kPa. Their ice adhesion is much lower in comparison to bulk stainless steel, which was measured around 269 kPa (Koivuluoto, et al., 2020). Such product as Ultra-Ever dry is an effective hydro/icephobic coating with low ice adhesion and an improved abrasion resistance of the coating. (MacKenzie, 2013). The paint can become a quick solution due to its high commercial availability. Due to the frictions of the woodchip load, the paint resistance to abrasion must be known. Ultra-Ever Dry company provides results on a Taber Abrasion Method (ASTM D4060-10) that show reinforced durability of the paint when compared to other products.

2.3.3.2 *Icephobic coating*

Studies report that icephobicity is related to the contact angle (Ge, et al., 2013, Stefan Jung, 2011). They show a correlation between superhydrophobic surfaces and icephobic surfaces; when the contact angle increases, the ice adhesion decreases. Later, the relation between ice adhesion and contact angle is valid only at low contact angle hysteresis (CAH). CAH describes the relation between the advancing contact angle Θ_A and the receding contact angle Θ_R, which occurs during the movement of the water droplet as shown in Figure 2. Low CAH indicates high droplet mobility. High CAH reveals higher surface contact that signals of higher ice adhesion. The time of ice formation also plays its role in the icephobicity. By increasing the contact angle on superhydrophobic surfaces, the time of ice formation gets delayed. However, the smoothness of a hydrophilic surface can also affect the icing time. (Irajizad, et al., 2019).

Tampere University ice laboratory team has studied the ice adhesion on thermally sprayed coating (TS-). Flame-spray coating (FS-) is one of the thermal spray techniques. FS-PE shows a low ice adhesion of 54-69 kPa. Its commercial availability makes this coating one of the solutions to help woodworkers. However, FS-SLIPS methods combined with silicone oil have the lowest value of this publication (21-27 kPa), which could make a good advancement in reducing woodchips-Ice problems. (Koivuluoto, et al., 2020).

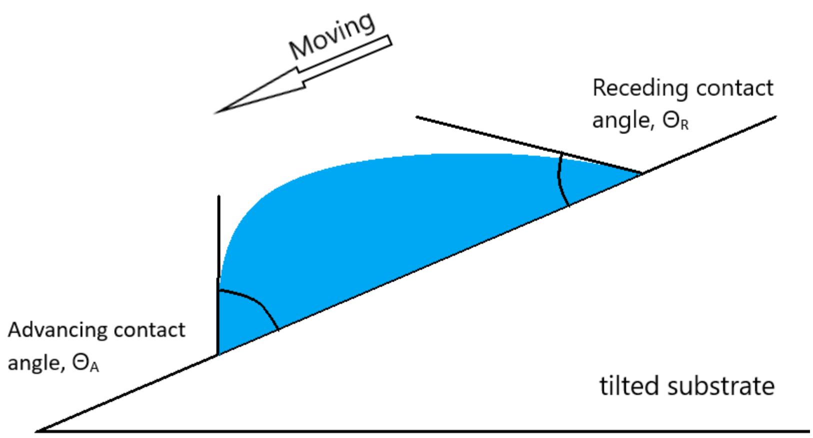

Figure 2. Advancing and receding contact angles on the tilted substrate

2.3.4 Industrial vibrators

The process when loaded material adhere to the truck container is known as carryback. Although the driver can remove the carryback manually by hitting on the container or climbing inside, both techniques can cause damage with time and be unsafe. (Texas Department of Insurance - Division of Workers' Compensation (DWC), 2020).

Using industrial vibrators to ease the dumping of material is therefore recommended as the procedure is very effective on compacted dry matters like dirt or woodchips. The vibration will reduce friction between the material and the wall. This prevents workers from damaging the whole dumping truck container by hitting it. The effect on frozen woodchips have been testified by vibrator companies but there are no scientific studies found that address or proves this solution so far. Only a few companies promote the use of industrial vibrators regarding the ice adhesion.

There are different types of vibrators: rotational that include an unbalanced weight powered either electrically, hydraulically or pneumatically; and linear with a piston. For coarse material like woodchips, it is recommended to choose hydraulic vibrator or piston vibrators (The Act Group, 2019).

There is a system that can be temporary mounted onto the container via a suction system. For instance, system like the Martin VAC-MOUNT portable vibrators is held on the container by vacuum. They do not require any permanent installation and can be connected to the truck's hydraulic system. (Martin Vibration Systems & Solutions, 2021). Those systems are mainly used for grain hoppers but can be adapted on every flat surface.

2.3.5 Heating system

Heat could be efficient if applied at the inner surface of the container to melt the contact layer of the ice between the woodchips and the surface of the container. Ice would be removed therefore making the unloading easier. The energy required to melt 1 g of ice is approx. 335 J/g. Heating the container to 0°C to melt only 1 mm layer of frozen woodchips would be enough to free them from adhesion (Ryerson, 2013).

ThermalWing is a product by Kelly Aerospace Thermal System. It consists of flexible, electrically conductive graphite foil realizing warming cycle to 5°C, which is enough to melt and remove the adhesion layer on the container. (RDD Entreprises , 2020). The product is designed for airplanes and so far, there are no known applications of it for containers. The main concern of using this system in a container is the strength of the material. There is a question if it can hold a load of woodchips.

In 1932, Ferwerda patented a heating system derived from the exhaust pipe. The fume was redirected through the trailer's bed and wall. It allowed to warm all metal and therefore avoid adhesion of frozen material. A flexible pipe connects the exhaust pipe to the trailers, allowing it to pivot to unload. (Ferwerda, 1932). The testimonials of companies using this system to warm up their woodchips in on-site containers revealed the presence of degradation by oxidation from the inside of the exhaust pipe heating system. In winter, the combustion of the wood in contact with the cold container causes condensation.

3 Materials and Methods

In contact with the frozen container, the moisture of the woodchips will freeze on the metal and create a metal-ice-woodchips layer system, illustrated in Figure 3. This layer will make the woodchips attached to the container walls and therefore prevent the complete unloading of the container. The determination of the adhesion is defined by the strength of the ice layer.

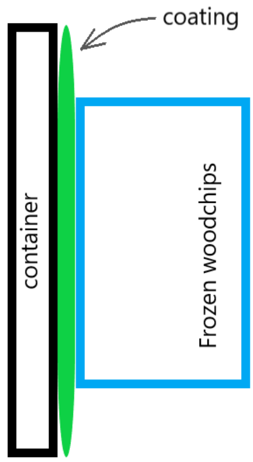

Figure 3. Simplification of the interaction container/coating/frozen woodchips

To understand and measure the adhesion of frozen woodchips to the containers' walls, centrifugal ice adhesion test and shear test are used. The purpose of these tests is to determine what surface has the lowest frozen woodchips adhesion.

To recreate the situation where woodchips are stored in container in winter, the tests are conducted in a cold room set at -10°C with a relative humidity approx. 40% overnight. This corresponds to the lower-than-average temperature of Finland, during the icing period (November to March) and a higher average humidity during this same period. (Pirinen, et al., 2012).

3.1 Materials

3.1.1 Test boxes and Centrifugal Ice Adhesion Test Samples

5 test boxes have been made: 4 from steel plates and 1 from polyethylene Quicksilver. The inner measurements of each box were 300x300x300 mm. The bottom of the box could slide by pushing the side of it after removing the holding screws. The bottom of the metal boxes was reinforced with PE plastic (Quicksilver PE) to prevent metal deformation during the shear test.

Figure 4. Metal box; 1,25 mm sheet

Figure 5. Quicksilver; 12 mm polyethylene sheet

4 metal (cold rolled steel sheet DC01AMO), 1,25 mm thick in Figure 4:

1 uncoated steel which was sprayed with silicone oil or rapeseed oil.

2 Flame-sprayed PE, rough and smooth.

1 Flame-sprayed PE-SLIPS infused with silicone oil.

1 UHMWPE (Quicksilver liner from ETRA Oy), 12 mm thick in Figure 5.

After producing the coating inside the box (see details in part 3.1.3), the boxes are filled with woodchips of known moisture. The full boxes are left in the ice room for the night at -10°C. Once frozen, the boxes are put at room temperature, flipped to the side and prepared so that the bottom part was able to move. The design was made to allow the implementation of a shear test, as shown in Figure 7. This test shows the attachment of the frozen woodchips to the box. Shear tests are more reliable than tension tests because while the metal plate is pulled up, the woodchips may still be attached to the removed part. Shear tests allow to keep the woodchips in the premises of the box while the bottom part is removed, set up shown in Figure 6.

Figure 6. Set up for shear tests at room temperature with force gauge

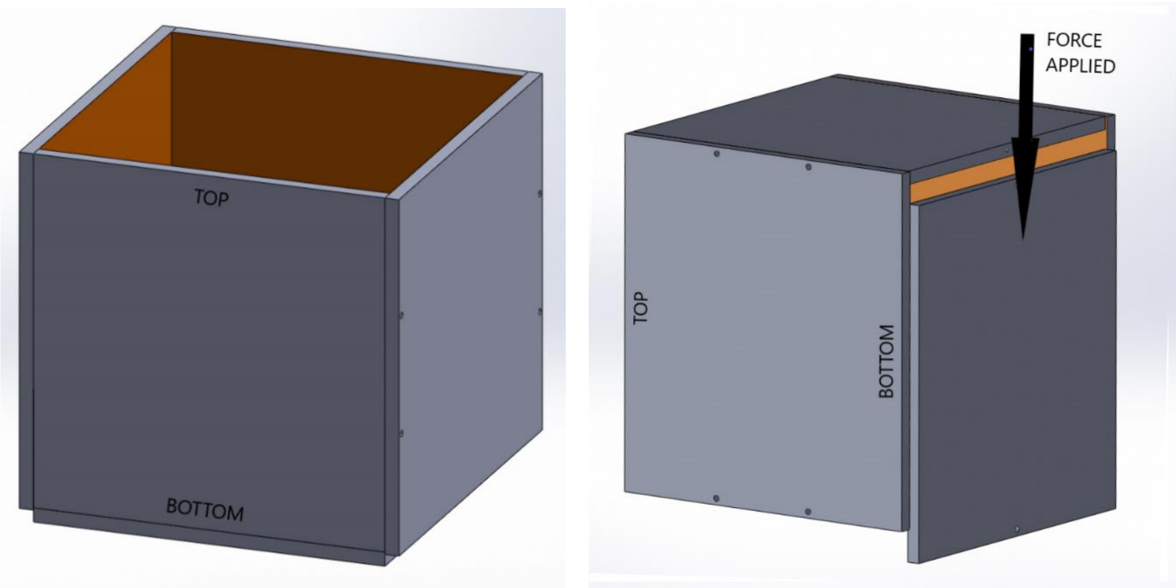

Figure 7. Position of the boxes for shear test

3.1.2 Centrifugal Ice Adhesion test samples

9 series of 4 samples have been made for a total of 36 samples: 32 from steel plates and 4 from PE Quicksilver. The CAT samples are made of 6x3 cm steel plate, coated on one side and pierced through to be installed in the CAT machine. A 3x3 cm plastic mold is also made to hold the woodchips during the freezing process (see Figure 8 and Figure 9).

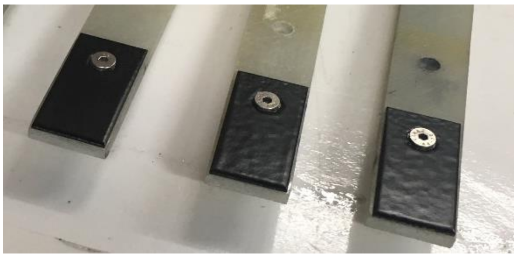

Figure 8. Coated CAT samples

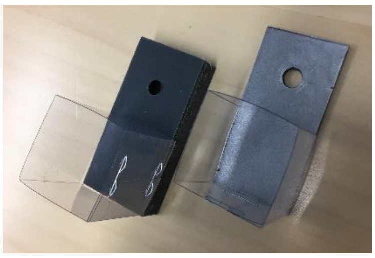

Figure 9. PE Quicksilver and uncoated metal CAT sample with mold

32 metal (cold rolled steel sheet DC01AMO), 1,25 mm thick:

16 uncoated steel which 12 were sprayed with silicone oil on 4, rapeseed oil on 4 and antifreeze on 4.

8 Flame-sprayed PE, 4 rough and 4 smooth.

4 Flame-sprayed PE-SLIPS infused with silicone oil.

4 covered with Teflon (PTFE) tape

4 UHMWPE (Quicksilver liner from ETRA Oy), 12 mm thick.

By using a 3x3 cm mold on the sample plate, woodchips are held in place while freezing. The size of the woodchips was important as the contact surface between the woodchips-ice mixture and the coating determined the strength of the ice adhesion. After this, the mold was removed from the frozen woodchips and they were tested with the CAT in the cold climate room at -10°C, as shown in Figure 14.

3.1.3 Coating

3.1.3.1 ***Flame spray coating***

The coatings have been done in the thermal spray lab at Tampere University. The flame spraying gun is a CastoDyn DS 8000 (Castolin) with a M10 nozzle (Figure 10). It is attached to a mechanical arm from ABB Robotics AB type IRB4400/60. The robot arm is assisting the work with a traverse speed set to 750 mm/s with 5 mm step and a speed of 999 mm/s for a post-heating treatment. The temperature was controlled by a Fluke Thermal Imager Ti300, as illustrated in Figure 11. The coating is low density polyethylene (LD-PE) powder (~150 µm, Plascoat), fed by a Sulzer Metco 4MP (Oerlikon Metco) with indicator 105, pressure 5,0 bar, flow 60 slpm (standard liters per minute). Pretreatment on the steel plate consists of a grit blasting with alumina (Mesh 40). A pre-heating is done before spraying around 90-100°C with flame, which is usually achieved with 1 or 2 heating times. The pre-

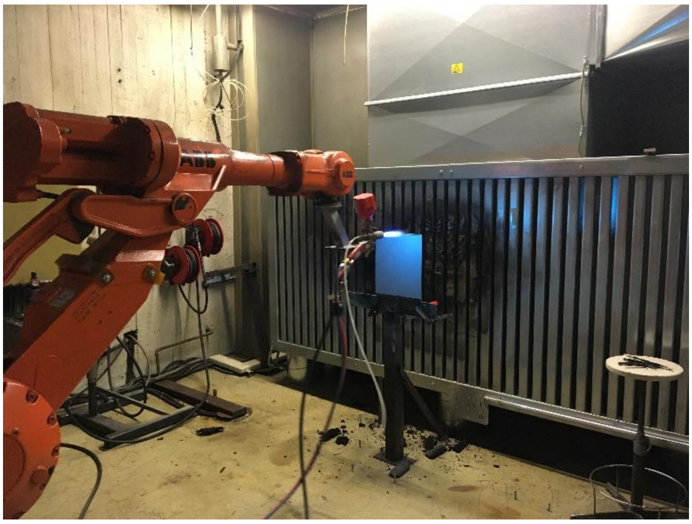

Figure 10. Coating process assisted with robot arm

Figure 11. Temperature control

heating and the post-heating distance and the gases setup depend on the type of coating.

3.1.3.1.1 FS PE-SLIPS

The gases flow is set to 4,0 bar of oxygen and 0,7 bar of acetylene. The substrate is pre-heated with the flame from a distance of 200 mm. A coating of 6 layers of flame + powder is applied from 350 mm, followed by 3 layers just powder without flame at the distance of 100 mm. The SLIPS coating does not require post heating.

3.1.3.1.2 Rough FS-PE

The gases flow is set to 4,2 bar of oxygen and 0,7 bar of acetylene. The substrate is pre-heated with the flame from 250 mm. Then follows a coating production from 250 mm, 6 layers flame + powder with an additional air to the gun of 2 bar. The post-heating treatment is made at a distance of 250 mm with flame (Figure 12).

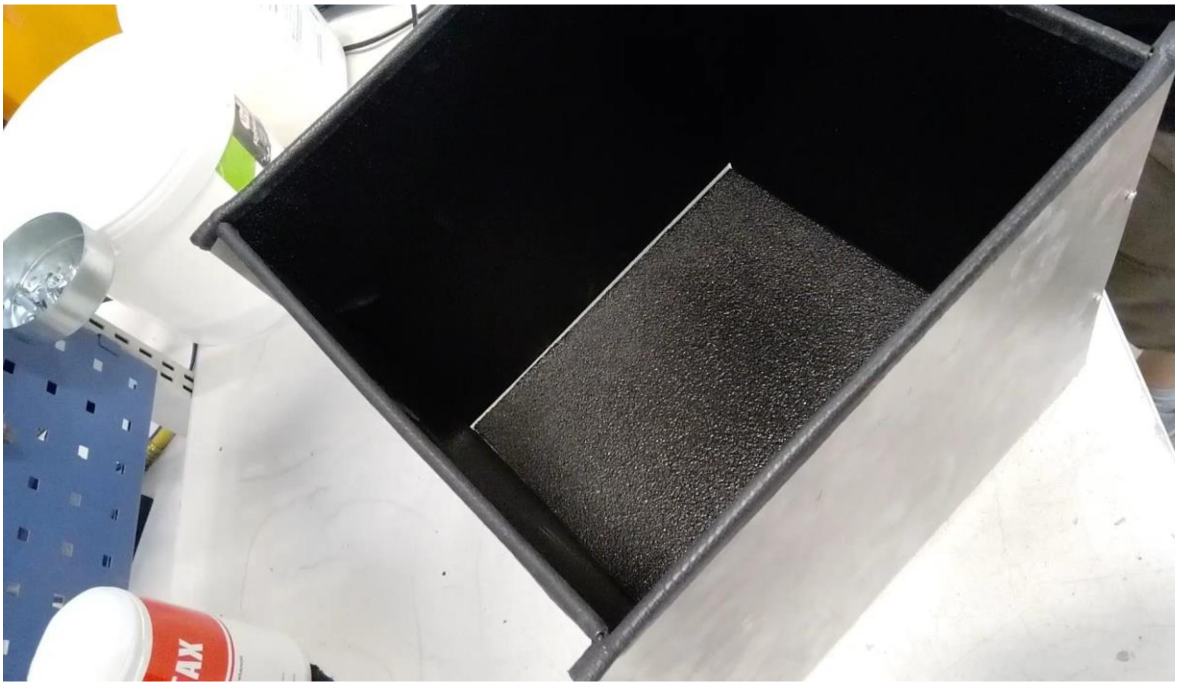

Figure 12. Rough FS-PE coated box

3.1.3.1.3 Smooth FS-PE

The gases flow is set to 4,0 bar of oxygen and 0,7 bar of acetylene. The substrate is pre-heated with the flame from 200 mm, followed by a coating production from 325 mm, 6 layers flame + powder. The post-heating treatment is made at a distance of 250 mm with flame.

3.1.3.2 ***Supplementary coating***

In addition to the thermally sprayed coating, other surface solutions were studied. The boxes were coated with rapeseed oil (Keiju) and silicone oil (500 cSt from Sigma-Aldrich for the FS-SLIPS and Wurth spray). The CAT samples had the same coatings as the boxes and also included antifreeze (Star brite Anti-Freeze propylene glycol -46°C), and Teflon tape (3M) for reference.

3.2 Methods

3.2.1 Shear test

A shear test is designed to apply stress to a test sample so that it experiences a sliding failure along a plane that is parallel to the forces applied. This method is used to study de-icing. Generally, shear forces cause one surface of a material to move in one direction and the other surface to move in the opposite direction so that the material is stressed in a sliding motion. The difference between shear tests and tension and compression tests is that with shear tests the forces applied are parallel to the two-contact surface, whereas in tension and compression tests they are perpendicular to the contact surfaces. (Rønneberg, et al., 2020)

A shear test was realized by pushing the bottom of the box at room temperature. The design has been made to allow this part to slide away from the box.

The box is set at 90° angle in such position as to allow the bottom to slide freely from the box. A force gauge is applied to the side of the box every 5 min to push the bottom down and record the force and the time (in minutes) needed to break the ice-woodchips mixture adhesion at room temperature after staying the night at -10°C.

$$\tau = \frac{F}{A} \tag{1}$$

where τ [N/m^2] is the shear stress, *F* [N] is the shear force and *A* [m^2] is the surface area of the ice.

Equation (1) is used and as we are doing vertical shear test, we need to see the influence of gravity with Equation (2). The self-removing depends on gravity, on the mass of the bottom plate and the area. The mass of the moving plate is around 1 kg for the Quicksilver PE and 2 kg for the steel, and the area is 0,09 m^2, therefore the gravity should have an influence on the ice adhesion. The force gauge is a Kern/Sauter digital FK25 with a readability of 0,01 N and a maximum load of 25 N, mounted on Kern test stand. Due to its low sensitivity, a pressing every 5 min will be done until the release of the plate.

$$\tau' = \frac{F'}{A} = \frac{mg}{A} \tag{2}$$

where *m* [g] is the mass and *g* [m/s^2] is the gravity.

The τ' is measured regarding to the weight of the moving part, (being built from multiple materials, using the density would result in errors) and compared to time for the adhesion of the ice-woodchips mixture to break. Therefore, boxes with different moving plate can be correlated to each other. (Rønneberg, et al., 2020).

3.2.2 Centrifugal Ice Adhesion test

Another test is done to measure the coated surface/woodchips interaction: a Centrifugal Adhesion Test. In principle, this machine shows the relation between the adhesion strength of an ice cube frozen on the CAT blade and the rotation speed at a constant angular acceleration. When the samples detach, they hit the dome and activate a sensor recording the time and the rotation speed. (Koivuluoto, et al., 2020)

According to Koivuluoto et al. (2020), these tests are realized on the 6x3 cm coated sample plate. Woodchips are frozen on the other half of it and then placed in the CAT, as seen in Figure 13.

Initially, it was decided to only freeze woodchips on top of the plate, but preliminary tests showed an inconsistency in the contact area on the surface plate that made the calculation impossible. Therefore, it was decided to use an ice-woodchips mixture to better mimic the wall-ice-woodchips layer that can be found in the woodchips containers.

In the CAT testing, an acceleration rate of 300 rpm/s was used. Ice removal was detected with the acceleration sensor and the shear ice adhesion strength τ_{iwm} [kPa] was calculated by using the following equation (3):

$$\tau_{iwm} = \frac{F}{A} = \frac{m_{iwm} r \omega^2}{A} = \frac{m_{iwm} r (\alpha t)^2}{A} \tag{3}$$

where F [N] is the centrifugal force at the moment of ice-woodchips mixture detachment, A [m^2] is the iced area, m_{iwm} [kg] is the mass of the accreted ice-woodchips mixture, r [m] is the radial spinning length, ω [rad/s] is the rotational speed, α is the constant acceleration rate (300 rpm/s) and t [s] is the time of the ice detachment.

Figure 13. Example of frozen woodchips sample on metal plate

Figure 14. Woodchips sample mounted in CAT device

Since it is important to have a sufficient surface contact between woodchips and the plate. Smoking woodchips (Roast master, basic smoking chips) were used as they have the optimal size to fit the sample's mold. Prior to the test the woodchips were sprayed with water. The moisture test was also conducted to determine the water content.

The samples were made from 6x3cm plate, the Table 1 shows the list of the samples divided into different groups. There were 4 repetitions tests for each sample. The coatings were made on steel plate. The samples are displayed in Figure 15.

Name	Material	Form
	Flame-Sprayed Coatings	
FS-PE smooth	polyethylene	Coating
FS-PE rough	polyethylene	Coating
FS-SLIPS	Polyethylene + silicone oil (500cSt)	SLIPS
	Bulk Materials	
Control	Steel	Bulk plate
Quicksilver PE	Ultra-high molecular weight polyethylene	Bulk plate
	Reference	
Teflon tape	Polytetrafluoroethylene PTFE	Tape

Sprayed layers		
Silicone oil	Silicone oil	Spray
Antifreeze	Propylene glycol	Spray
Vegetable oil	Rapeseed oil	Spray

Table 1. Coatings and surfaces used

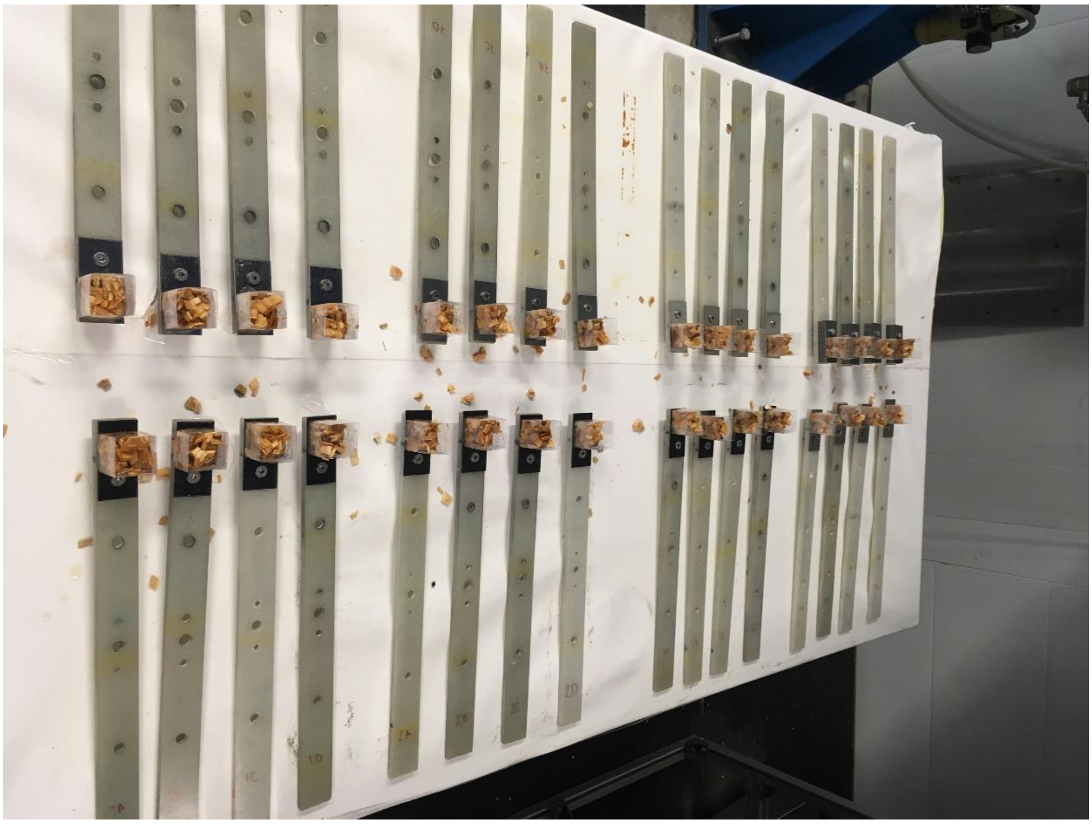

Figure 15. Final samples composition woodchips and ice mixture. 4 samples x 8 testing surfaces.

3.2.3 Determination of moisture content by oven-dry method

The force to remove woodchips frozen on surface is proportional to the moisture content. The higher the moisture content, the stronger the adhesion (Mattsson, 1996). When it comes to water, there are three different ways it appears in woodchips: the fiber water that is bound in the cell, the capillary water that is in the cell cavities, and the water on the surface. The vapor pressure determines the available water to freeze on the surface of the woodchips (Esping, 1977). As reducing the humidity of the forest in winter is not an option, it is nonetheless possible to avoid unnecessary coming of water in contact with the woodchips by protecting them from the rain.

For these tests, it was decided to use woodchips with high moisture content to reproduce the situation of storing woodchips on site (forest) in winter without cover.

The moisture content is the ratio between water and dry wood. To measure the moisture content, the wet woodchips were to stay at 103°C in the oven for 24 hours or until the weight was stable after 2 measurements from 2 hours apart. (Reeb & Milota, 1999)

To simulate natural condition of the woodchips in outside storage or in the wood, the woodchips were sprayed with water prior to the test, and the moisture content was calculated with the following Equations (4) and (5):

$$MC\% = \frac{(m_{wet} - m_{dry})}{m_{dry}} \times 100 \qquad (4)$$

Which can be simplified as:

$$MC\% = \frac{m_{water}}{m_{wood}} \times 100 \qquad (5)$$

where *MC%* is the moisture content in percent of dry mass, m_{wet} [g] is the weight of the wet woodchips sample, and m_{dry} [g] is the mass of the dry woodchips. (Reeb & Milota, 1999)

When sprayed with water every 30 min, the woodchips reached to a moisture content of 98 to 140%. The moisture content was checked before the tests.

Although preliminary test has shown that the moisture content is stable after 6 hours in the oven, the sample was left in the oven over night for 24 hours.

4 Results and Discussion

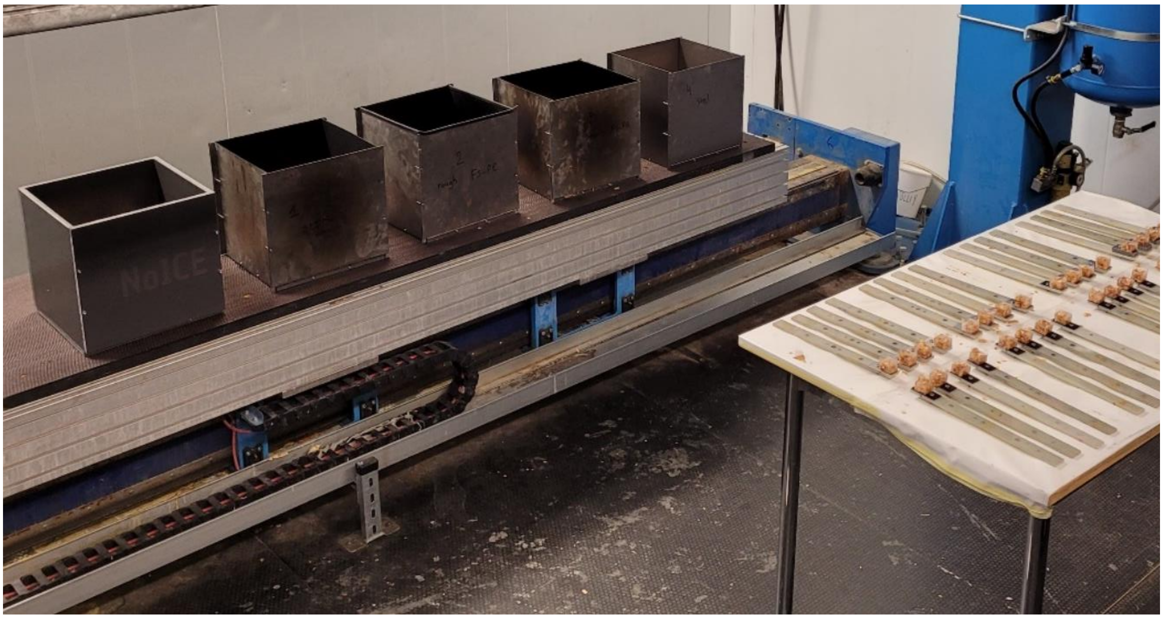

Figure 16. Set up of the icing experiment in ice laboratory

4.1 Results

The tests were divided into two parts: containers shear tests and CAT tests. The Figure 16 shows the setup of the ice laboratory during the freezing period. On the left, are displayed the 5 containers, on the right the first series of CAT samples.

4.1.1 Icing of woodchip containers

Due to sensitivity of the force gauge (max. 25 N), it was not possible to measure the force required to break the surface-ice bond. Broken ice adhesions are shown in Figure 17, Figure 18, and Figure 19. However, the time for the gravity to pull down the plate was measured and give a way to compare the ice adhesion of the different surfaces. As calculated with equation (2), the influence of gravity is 116,9 N/m^2 for the polyethylene box and an average of 212,4 N/m^2 for the steel metal.

Tested samples		Ice removal test for boxes (at 20°C)	influence of gravity τ' [N/m^2]	Time/weight [min/kg]
	plate weight (kg)	Time for ice detachment (min)		
FS-SLIPS (PE+Silicone oil)	1,996	0	217,56	0
Steel covered with rapeseed oil	1,915	0	208,74	0
PE bulk Quicksilver	1,072	44	116,85	41,045
Steel with sprayed silicone oil	1,915	52	208,74	27,154
FS-PE coating rough	1,990	67	216,91	33,668
FS-PE coating smooth	1,929	88	210,26	45,619

Table 2. Overview of the results of ice removal times. this table include the influence of gravity related to the weight of the removable plate and the time per kg allowing to compare the ice adhesion.

Figure 17. Rough FS-PE coating

Figure 18. Sprayed silicone oil on steel

Figure 19. Quicksilver PE bulk

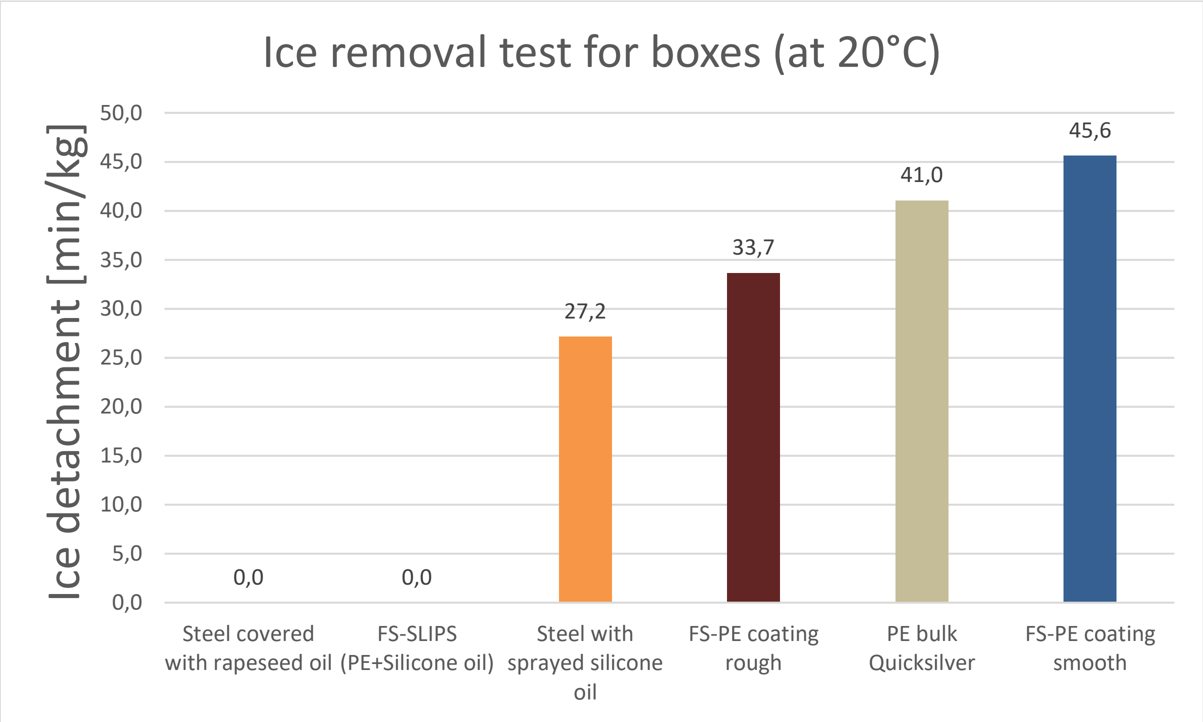

Figure 20. Time of ice removal of different surfaces in minutes at room temperature.

Table 2 and Figure 20 show the time required for the ice adhesion to break under the influence of the gravity at room temperature. As the influence of gravity is similar between all steel boxes (around 210 N/m^2), it is possible to compare the ice adhesion. For the polyethylene Quicksilver, the influence of the gravity is about 117 N/m^2. As the plate weight is different for each box, the way to compare the results is to use the time/plate mass [min/kg].

This test has shown that the highest ice-woodchips mixture time/mass detachment occurred on solid surfaces of FS-PE rough, FS-PE smooth and Quicksilver PE bulk, in opposition to liquid added surfaces. The comparison of solid surfaces highlights the role of roughness. In his thesis, Beeram (2017) shows that correlation between roughness and ice adhesion depends on the wettability of the surface. The ice adhesion increases with roughness of hydrophilic surfaces but decreases with roughness of hydrophobic surfaces. (Beeram, 2017)

The PE has hydrophobic properties (Linda Silvia, 2019), which makes it faster to remove the ice-woodchip mixture from the FS-PE rough surface in comparison to FS-PE smooth surface.

The lowest adhesion is noticeable with spraying rapeseed oil and the FS-SLIPS technology with silicon oil. These surfaces adhesions were so low, that the box plate fell off during the preparation of the test while removing the holding screws.

4.1.2 Centrifuge Ice Adhesion tests results

The 9 surfaces were tested in the Tampere University Ice Laboratory CAT machine. 8 samples are displayed in Figure 21 and Figure 22.

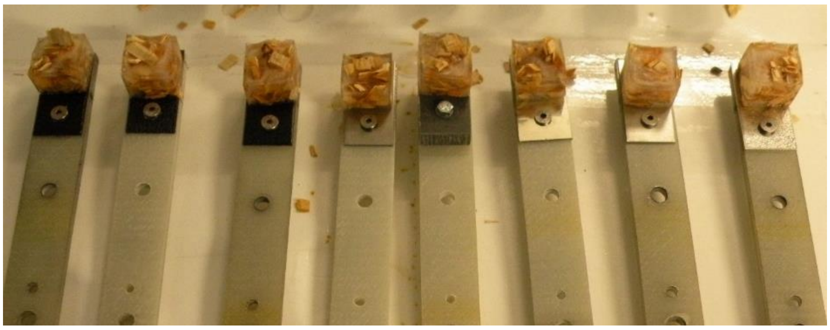

Figure 21. Frozen samples ready to go to the CAT machine. From left to right, TS-SLIPS, rough FS-PE, smooth FS-PE, steel and silicone oil, Quicksilver PE, PTFE tape, steel, steel and anti-freeze.

Figure 22. Results sample after the CAT tests. From left to right, TS-SLIPS, rough FS-PE, smooth FS-PE, steel and silicone oil, Quicksilver PE, PTFE tape, steel, steel and anti-freeze.

FS-SLIPS (PE+Silicone oil)				**Steel with sprayed silicone oil**				**Steel**			
	IA (kPa)	IA_Av (kPA)	IA_STD (kPa)		IA (kPa)	IA_Av (kPA)	IA_STD (kPa)		IA (kPa)	IA_Av (kPA)	IA_STD (kPa)
1A	-	**15**	*7,7*	4A	28	**67**	*41,5*	7A*	85	**78**	*19*
1B	21			4B	35			7B*	61		
1C	18			4C	100			7C*	66		
1D	6			4D	106			7D*	102	*Thin ice layer on the surface after CAT	
FS-PE coating rough				**PE bulk Quicksilver**				**Stainless steel with sprayed anti-freeze liquid**			
2A	116	**98**	*23,5*	5A	55	**34**	*29,2*	8A	8	**6**	*2,4*
2B	116			5B	13			8B	8		
2C	92			5C	-			8C	4		
2D	67			5D	-			8D	4		
FS-PE coating smooth				**PTFE tape**				**Steel covered with rapeseed oil**			
3A	89	**87**	*21,9*	6A	64	**42**	*22,4*	9A	22	**53**	*25,4*
3B	90			6B	20			9B	61		
3C	112			6C	24			9C	82		
3D	59			6D	58			9D	46		

Table 3. Overview of ice adhesion results average (IA_Av) tests results divided per surfaces, Ice adhesion measured per tests (IA) and standard deviation (IA_STD).

There are 4 tests per surfaces; The ice samples broke before going the testing procedure for 1 sample of FS-SLIPS and 2 samples of PE Quicksilver. We observed also a thin layer of ice on the steel samples showing that the ice broke and the ice adhesion on steel was even stronger, as seen in Figure 22.

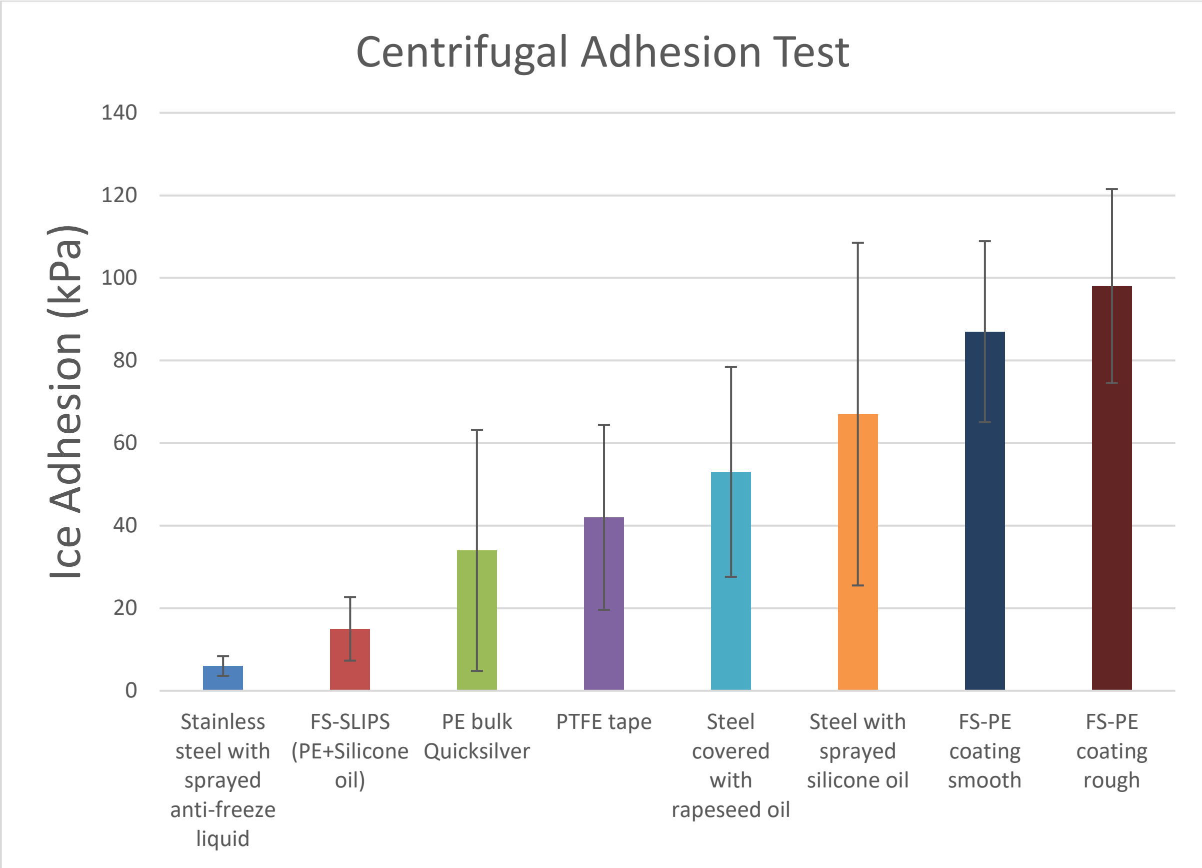

Figure 23. Ice adhesion for different surfaces with standard deviation

Figure 23 compares the ice adhesion results of different surfaces with CAT. It shows a very low adhesion with stainless steel covered in antifreeze and SLIPS technology.

4.2 Discussion and conclusion

Even though those two protocols involve different units, they provided additional results and support each others. The CAT shows surface behaviour of ice adhesion, and the shear test shows the application with larger surface area. No equation is found to relate ice adhesion [kPa], the time of detachment [min], moisture content [%], temperature gradient [°C/sec] and the effect of gravity [N/m^2]. Although it is not possible to directly compare the tests from different units, there are noticeable patterns. The container tests and CAT show similarity. For example, FS-SLIPS surfaces infused with silicone oil have the lowest ice adhesion in the CAT at -10°C and the fastest ice-woodchips mixture removal at room temperature.

Disparity between CAT and container tests occurs regarding the steel covered in rapeseed oil. This could be because the rapeseed oil has a solidification temperature between 0°C and -15°C (Die Deutschen Versicherer, 1998-2021). The CAT testing was carried out at -10°C in the solidification range of the rapeseed oil and the container test was carried out at room temperature 20°C. It is possible that the transportation time and the test preparation time from the cold room to the lab affects the liquifying of the oil that could have started melting before the start of the test. However, the short time is highly improbable to influence the tests.

Difference between silicone oil in FS-SLIPS and sprayed on surface could be explained by the viscosity of the oil. At -10°C silicone oil is still liquid with a very high viscosity (Monteiro, et al., 2018). The viscosity of a liquid is described by the cohesive force between the molecules. The degree of polymerization of the silicone chains is related to viscosity. Bigger polymer chains create friction between themselves. Lowering the temperature affects the movement of the molecules, slowing them down, therefore increasing the viscosity. At the contact with the surface, there is the adhesion force which is defined by the force that is required by a liquid droplet to detach from a surface. Liu et al. (2020) showed that viscosity of silicone oil affects the ice adhesion strength. This article also shows a relative difference between porous anodic aluminum oxide (AAO) used as a SLIPS support infused with silicone oil, and bare aluminum. (Liu, et al., 2020). The same effects are observed during the results. The silicone oil used in SLIPS has lower viscosity than the sprayed silicone oil that is called technical quality silicone oil and contains a mixture of products for example propan-2-ol, hydrocarbons (WÜRTH, 2015). Also, SLIPS structure helps oil to stay on the surface, which is not the case with sprayed silicone oil layer. The viscosity of the silicone oil and the SLIPS surface result in a lower ice adhesion for FS-SLIPS.

Further experimentations should be conducted to define the influence of viscosity on ice adhesion, along with comparison of viscosity of silicone oil, rapeseed oil and propylene glycol between -20°C and 20°C.

This report gives a wide range of solutions for companies to avoid ice adhesion troubles. It provides a comprehensive overview of the existing methods aiming to find a suitable solution and meet the needs of the companies and presents the results of some of our laboratory tests. Every solution tested and/or reported shows a decrease in ice adhesion compared to the raw stainless steel (269±13 kPa) (Koivuluoto, et al., 2020).

The next step is to upscale these tests to see their adaptabilities on full size containers, to determine the quantity of chemicals to spray, and to evaluate the costs of woodchips transportation and the life time of treated surfaces.

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