NTNU – Trondheim
Norwegian University of
Science and Technology

Improving the Performance of Road Salt on Anti-Icing

Wibeke Lende

Civil and Environmental Engineering
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Supervisor: Alex Klein-Paste, BAT

Norwegian University of Science and Technology
Department of Civil and Transport Engineering

NORWEGIAN UNIVERSITY OF SCIENCE AND TECHNOLOGY
DEPARTMENT OF CIVIL AND TRANSPORT ENGINEERING

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Name: Wibeke Lende				
Professor in charge/supervisor: Alex Klein-Paste				
Other external professional contacts/supervisors: Åge Sievertsen				

Abstract:
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An alternative mechanism of the anti-icing wet pavement freezing has recently been introduced. It says that the salt does not only depress the freezing point, but the presence of them also weakens the ice. If the ice is weakened sufficiently, the traffic load on the road will destroy the ice, giving an acceptable level of friction again.
The salts are not so environmentally friendly, and can cause damage to cars and the area surrounding the road. Therefore, alternative chemicals are developed to reduce the amount of salt used on the road. Agricultural By-Products are among these other materials, and are used as an additive to the road salt. This is said to reduce the need for salt, but there is little research that shows how these products work, and whether they work as well as the salts. The objective of this thesis was to test one of these and whether the addition of them would reduce the strength of the ice as much as a salt solution would.

The laboratory experiment performed in this thesis has been developed and used earlier by Klein-Paste and Wåhlin (2013). A new parameter was proposed, called equilibrium brine fraction, which could be used to predict whether the ice where sufficiently weakened. Asphalt substrates covered with solutions with different salt and additive concentrations where frozen in an ice laboratory. Afterwards the strength of the ice was tested with a British Pendulum Tester and the percentage of destroyed ice was noted. Many tests were performed at different temperatures, which gave a dataset showing what brine frictions were likely to destroy the ice.

The probability that an ice sample failed for a given brine fraction where calculated using binomial logistic regression. Then the results from the experiment performed in this thesis and the ones from the experiment done by Klein-Paste and Wåhlin (2013) were compared.

The results showed that the additive of an Agricultural By-Product did neither improve nor have a negative effect on the weakening of the ice.

Keywords:

1. Winter maintenance
2. Anti-icing
3. Road salt
4. Agricultural By-Products

Wibeke Lende

NTNU
Fakultet for ingeniørvitenskap og teknologi
Institutt for bygg, anlegg og transport

MASTEROPPGAVE

(TBA4945 Transport, masteroppgave)

VÅREN 2014
for
Wibeke Lende

Forbedring av ytelse til vegsalt ved anti-ising

Bakgrunn
Salting av veger er fremdeles at aktuelt tema, og det er et sterkt ønske å minimalisere saltets negative konsekvenser på naturen og korrosjon på bilparken, bruer og overbygning. Å finne en erstatning for salt er ikke lett, fordi selv om det flere alternative kjemikalier på markedet er disse som regel betydelig dyrere, og det kreves mer for å oppnå samme virkning. I tillegg kan også alternative kjemikalier ha negative konsekvenser. En populær tilnærming, spesielt i USA og Canada er å bruke tilsetningsstoffer til vegsalt i et forsøk å forbedre ytelse. Disse tilsetningsstoffer er ofte sukkerbaserte biprodukter fra landbruk (agricultrural bi-products). Selv om det er betydelig praktisk erfaring med slike tilsetningsstoffer mangler det fremdeles mer grunnleggende kunnskap om hvordan disse stoffer virker/forbedrer ytelse.

Resent forsking har gitt ny kunnskap om hvordan salt virker i anti-ising av våte vegbaner (Klein-Paste and Wåhlin, 2013). Saltet setter ikke bare ned frysepunktet men bidrar også til å svekke isen som dannes slik at trafikken selv klarer å ødelegge den. Det er fremdeles ukjent om tilsetningsstoffer kan bidrar til denne svekkingen og dermed forbedre ytelse av vegsalt.

Målsetting
Målet med oppgaven er å skaffe mer kunnskap om *hvordan* tilsetningsstoffer virker. Dette skal gjøres gjennom en laboratoriestudie.

Oppgaven
Kandidaten skal:

Beskrive mekanismer bak anti-ising av våte vegbaner.

Utføre laboratorieforsøk med vegsalt (NaCl) med et kommersielt, sukkerbasert tilsetningsstoff.

Sammenlikne data med tilsvarende data på NaCl uten tilsetningsstoff.

Analysere og diskutere om tilsetningsstoffet har forbedret ytelse av NaCl.

GENERELT

Oppgaveteksten er ment som en ramme for kandidatens arbeid. Justeringer vil kunne skje underveis, når en ser hvordan arbeidet går. Eventuelle justeringer må skje i samråd med faglærer ved instituttet.

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Besvarelsen skal inneholde
- standard rapportforside (automatisk fra DAIM, http://daim.idi.ntnu.no/)
- tittelside med ekstrakt og stikkord (mal finnes på siden http://www.ntnu.no/bat/skjemabank)
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Ved innlevering av oppgaven skal kandidaten levere en CD med besvarelsen i digital form i pdf- og word-versjon med underliggende materiale (for eksempel datainnsamling) i digital form (f. eks. excel). Videre skal kandidaten levere innleveringsskjemaet (fra DAIM) hvor både Ark-Bibl i SBI og Fellestjenester (Byggsikring) i SB II har signert på skjemaet. Innleveringsskjema med de aktuelle signaturene underskrives av instituttkontoret før skjemaet leveres Fakultetskontoret.

Dokumentasjon som med instituttets støtte er samlet inn under arbeidet med oppgaven skal leveres inn sammen med besvarelsen.

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Dersom studenten i arbeidet med masteroppgaven skal delta i feltarbeid, tokt, befaring, feltkurs eller ekskursjoner, skal studenten sette seg inn i "Retningslinje ved feltarbeid m.m.". Dersom studenten i arbeidet med oppgaven skal delta i laboratorie- eller verkstedarbeid skal studenten sette seg inn i og følge reglene i "Laboratorie- og verkstedhåndbok". Disse dokumentene finnes på fakultetets HMS-sider på nettet, se http://www.ntnu.no/ivt/adm/hms/. Alle studenter som skal gjennomføre laboratoriearbeid i forbindelse med prosjekt- og masteroppgave skal gjennomføre et web-basert TRAINOR HMS-kurs. Påmelding på kurset skjer til sonja.hammer@ntnu.no

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Faglærer ved instituttet: Alex Klein-Paste

Institutt for bygg, anlegg og transport, NTNU
Dato: 10.01.2014, (revidert: 28.04.2014)

Underskrift

AKleinPaste

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Faglærer

Preface

This report is the end of my master degree at NTNU. It has been written at the Department of Civil and Transport Engineering, Faculty of Engineering Science and Technology. It explores Agricultural By-Products used as additives to road salt in winter maintenance. I was introduced to these additives through an article that I read for a previous subject at NTNU (Petkuviene and Paliulis, 2009). It deals with the corrosion that is caused by the road salt and whether an additive of Agricultural By-Product reduces the damage. This caught my interest and I wanted to know more about these alternative products.

The working conditions at NTNU were well facilitated to carry through the laboratory experiments with all the necessary equipment and materials. I would like to thank Bent Lervik and Jan Erik Molde who has been very helpful with different challenges in the laboratory. Also I want to thank the Norwegian Public Roads Administration for financing this project.

My fellow student Linda Betten has been an avid discussion partner throughout the semester and I would also like to thank her. Last I want to thank my supervisor, Alex Klein-Paste, for patience and good advice during the semester.

Summary

Winter maintenance is a challenging field, especially in parts of the world with a temperate climate. The friction of the road surface has to be kept at an acceptable level so that the demands for traffic safety and mobility are fulfilled. Road salts are used in large quantities for both anti-icing and de-icing objectives, because they depress the freezing point of water.

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The laboratory experiment performed in this thesis has been developed and used earlier by Klein-Paste and Wåhlin (2013). A new parameter was proposed, called equilibrium brine fraction, which could be used to predict whether the ice where sufficiently weakened. Asphalt substrates covered with solutions with different salt and additive concentrations where frozen in an ice laboratory. Afterwards the strength of the ice was tested with a British Pendulum Tester and the percentage of destroyed ice was noted. Many tests were performed at different temperatures, which gave a dataset showing what brine frictions were likely to destroy the ice.

The probability that an ice sample failed for a given brine fraction where calculated using binomial logistic regression. Then the results from the experiment performed in this thesis and the ones from the experiment done by Klein-Paste and Wåhlin (2013) were compared.

The results showed that the additive of an Agricultural By-Product did neither improve nor have a negative effect on the weakening of the ice.

Sammendrag

Vintervedlikehold er et utfordrende felt, spesielt i deler av verden med et temperert klima. Friksjonen av veibanen må holdes på et akseptabelt nivå, slik at krav til trafikksikkerhet og mobilitet er oppfylt. Vegsalter anvendes i store mengder både for anti-isdannelse og avisings formål, fordi de reduserer frysepunktet for vann.

En alternativ mekanisme for anti-isdannelse på frysing av våt asfalt er nylig blitt foreslått. Den sier at saltet ikke bare reduserer frysepunktet, men tilstedeværelsen av disse svekker også isen. Dersom isen svekkes tilstrekkelig, vil trafikkbelastningen på veien ødelegge isen, noe som gir en akseptabel grad av friksjon på nytt.

Saltene er ikke så miljøvennlig, og kan forårsake skade på biler og i området rundt veien. Derfor er alternative kjemikalier utviklet for å redusere den mengde salt som brukes på veien. Biprodukter fra landbruksprodukter er blant disse andre materialene, og brukes som tilsetning til vegsalt. Dette sies å redusere behovet for saltet, men det er lite forskning som viser hvordan disse produkter virker, og om de fungerer like bra som saltene. Formålet med denne oppgaven var å teste en av disse, og om tilsetting av dem ville redusere styrken på is så mye som en saltløsning ville.

Laboratorieeksperimentet utført i denne avhandlingen har blitt utviklet og brukt tidligere av Klein-Paste og Wahlin (2013). En ny parameter ble foreslått, kalt equilibrium brine fraction, som kan brukes til å forutsi hvorvidt isen er tilstrekkelig svekket. Asfalt dekket med løsninger med ulike konsentrasjoner av salt og det alternative produktet ble fryst i et frostlaboratorium. Etterpå ble styrken på isen testet med en British Pendulum Tester og andelen av ødelagt is ble notert. Mange tester ble utført ved forskjellige temperaturer, noe som ga et datasett som viste hvilke equilibrium brine fractions som sannsynligvis ville ødelegge isen.

Sannsynligheten for at en is prøve mislyktes for en gitt equilibrium brine fraction ble beregnet ved hjelp av binomial logistisk regresjon. Resultatene fra forsøket utført i denne avhandlingen og de fra forsøket gjort av Klein-Paste og Wahlin (2013) ble sammenlignet.

Resultatene viste at tilsetningsstoffet hadde verken positiv eller negativ effekt på svekkelse av isen.

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1 Introduction

The conditions of a road surface are always varying, as the temperature and weather will change continuously throughout the day. Regions of the world where there is a temperate climate are especially vulnerable to these changes, and will be particularly challenging during wintertime. In those areas there are big fluctuations in the temperature, which leads to rapid transitions between rain- and snowfall.

It is important that the road is continuously operated and maintained throughout the entire year, with all the challenges that the different seasons has. The roads have to be kept up to a good standard with regards to traffic safety and mobility.

The snow and ice which will occur on the road during winter time has to be controlled, which demands a good operation and maintenance plan. There are three main strategies used for winter maintenance; bare road strategy, winter road strategy and closed road strategy. When using a bare road strategy the road should be completely free of snow and ice throughout the entire year. For a winter road strategy most of the snow is removed, but a small amount of coverage is acceptable as long as the friction is within boundaries. The closed road strategy is only employed if a road has a low amount of daily traffic during the winter and there is an alternative route.

The roads in Norway are divided into five different maintenance classes, graded with letters from A to E. Class A is the highest classification there is, and uses a 100 percent bare road strategy. The maintenance demands is gradually reduced for the following classes, where class E is the lowest classification. This is a 100 percent winter road strategy, with the lowest acceptable coefficient of friction of 0.20. (Vegdirektoratet, 2012)

Various measures are implemented to keep the friction of the road at an acceptable level during wintertime, depending on what condition is on the road surface and which winter maintenance strategy is to be utilized on the road in question. The snow or ice can be removed by using mechanical removal with a snowplow and/or by using salt or other chemicals on the road. Scarifying or sanding can be applied after mechanical removal to increase the friction when using a winter road strategy.

Chemicals applied on the road are employed with two types of objectives in mind, anti-icing or de-icing. Anti-icing are when the chemicals are used to prevent snow and ice from forming on the road surface. De-icing is when the chemicals are meant to melt the ice that has already formed on the road surface. In Norway, anti-compaction is also named as a third objective, but most countries view this as part of the anti-icing objective. Anti-compaction is using the chemicals to prevent snow from compacting on the road, as compacted snow will be more difficult to remove with a snowplow.

In this thesis the focus has been on anti-icing, more precisely certain chemicals used for this purpose. Salt is traditionally been viewed upon as a good anti-icing chemical because of its ability to reduce the freezing point of water. Therefore the ice will not form so easily on the road surface.

Recently a new idea has been presented for how the salt contributes to anti-icing. The idea is that the salt not only has freezing point depression abilities, but that it also reduces the strength of the ice when a wet pavement freezes. An experiment to test this weakening of the ice in the presence of a solute was done for different solutions at different temperatures, with solutes of NaCl and KCOOH. (Klein-Paste and Wåhlin, 2013)

Although salt is mainly employed for anti-icing chemicals, there is a continuous search for better and more environmentally friendly alternatives. Some developed products are used as an additive to the road salt. These are referred to as Agricultural By-Products, and have become very popular in countries such as the UK, Canada and USA. The ABPs are said to give an increased performance to both de-icing and anti-icing. It gives a better melting capacity, a higher melting rate, depresses the freezing point, weakens the ice and increases the longevity on the road. This all leads to a reduction of the total amount of salt used. All this are anecdotal evidence provided by manufacturers and users, therefore the mechanics of these products has to be researched.(Highway-Agency, 2007)

In this project the experiment that was performed by Klein-Paste and Wåhlin (2013) has been done with the addition of an Agricultural By-Product. The objective was to compare the results from the two experiments, to see if the additive contributed to the weakening of the ice.

2 Theory

This chapter gives an overview of the theory behind the laboratory experiment performed in this thesis, to give an understanding as to why the experiment was performed.

2.1 Thermodynamics

This section explains some terms from thermodynamics that are relevant to the freezing of water.

2.1.1 System and surroundings

System and surroundings are two important terms to understand the mechanisms of thermodynamics. The system refers to the one part of the universe that is being studied. The surroundings are everything possible thing in the universe around the system that has an interaction with it of some sorts. A system can be of any size and shape, as long as the boundaries are set for it. Per example an observed system can be on the size of a molecule or as big as an entire planet.

2.1.2 Temperature

The molecules, atoms and subatomic particles in a medium are in a constant state of movement for every temperature above absolute zero. These random motions are referred to as the thermal motion. The temperature and the thermal motion are correlated, as a change in amount of movement in the particles will lead to a direct change in the temperature. The higher the temperature, the more thermal motion it will be in the medium. This means that water particles in a gaseous state will have much more movement than water in a liquid state. Likewise, water particles in a solid state of ice will have less movement.(Zumdahl, 2007)

2.1.3 Heat transfer

Temperature is closely correlated to heat, which is thermal energy transferred from one medium to another. Thermal energy is connected to the thermal movement as well, as it is the energy that is created by this random movement in the particles. The heat transfer will be in the form of infrared radiation, conduction or convection, and will always be moving from the medium with the highest temperature to the medium with the lowest temperature. Therefore, measuring the temperature of two mediums connected to each other makes it possible to determine which way the heat is moving between them. As an example, heat convection will occur as water on a road freezes when the surrounding air is below the freezing point. The thermal energy will be moving from the water to the air, thereby making the water colder and the air warmer. (Zumdahl, 2007)

2.1.4 Latent and sensible heat

Latent heat and sensible heat are two types of thermal energy that are released or absorbed from a thermodynamic system. The terms are related to what process the discussed medium is in. Latent heat will occur in a process of phase transition, meaning during melting, freezing, vaporization or condensation. It simply refers to the amount of energy that is needed to change from one phase to another. As explained earlier, the temperature will be constant during all of those four types of processes. Release and absorption of sensible heat occurs when the temperature is changing, meaning when the medium is not in a phase transition. (Zumdahl, 2007)

2.2 Characteristics of water

Winter maintenance is very much about controlling the water on the road, to keep the friction at an acceptable level. Therefore, an understanding of the mechanisms who determines the different states of water is needed, to research and improve the existing measures.

2.2.1 Phase diagram for water

Water can be found in three different phases; solid, liquid and gas. The phase diagram in Figure 1 show the temperature ranges for each of the different physical states of water. It is unique in the way that it is the only natural material that can exist in all of the three phases in a wide range of temperatures (Nazaroff and Alvarez-Cohen, 2001).

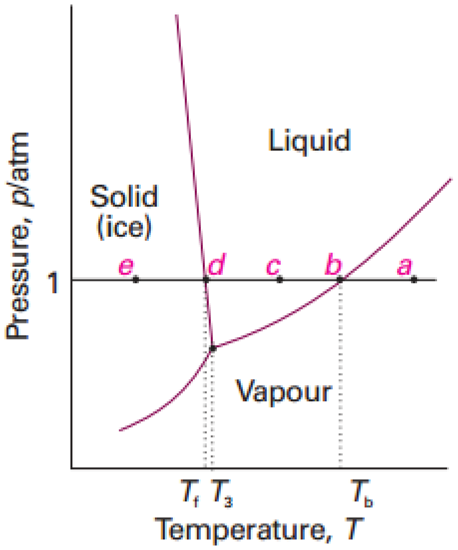

Figure 1 - Phase diagram for water (Atkins and de Paula, 2006)

The points displayed in Figure 1 are marked at a normal atmospheric pressure, 1 atm. This is the average sea level pressure, which means that *a*, *b*, *c*, *d* and *e* show the condition of the water at sea level, at the given temperature.

The water is in a liquid state in point *c*, a solid state in point *e*, and a gaseous state in point *a*. Point *b* displays the water at the normal boiling point and *d* the normal freezing point. At these two points the water will be in an equilibrium state. This means that the medium will be comprised of two different phases; both liquid and gaseous water present at the boiling point, and both solid and liquid water present at the freezing point. The mass of the two phases will not change as long as the pressure or temperature does not. If the temperature change and the atmospheric pressure stay unaltered, the water will transform from comprising of two phases to one singular phase, after some time. Likewise, if the pressure changes and the temperature remain the same, the water will transform to only one of the phases.

2.2.2 Supercooling

In some instances pure water will not turn to solid ice when it reaches its freezing point for the respective atmospheric pressure. It can in fact stay in its liquid state for several degrees below the freezing point. This phenomenon is referred to as supercooling, and can also be found for other liquids.

The reason for the supercooling is that the liquid has not reached the level of organization that is needed for it to turn into its solid state. The water will continue to release sensible heat until the proper level of organization is attained, and then begin to crystallize. As the water starts to solidify, the temperature of the system will rise to the freezing point again. Here the residual water will turn into ice, releasing latent heat in the process until all the water is frozen. Then the ice will continue to cool down until it reaches the temperature of its surroundings. (Zumdahl, 2007)

2.3 Anti-icing

The objective of anti-icing is to prevent ice from forming on the road. Because of the colligative property that is freezing point depression of dilute solutions, it is common to use different types of salts and other types of chemicals for anti-icing measures. Typical chemicals used are Sodium Chloride (NaCl), Magnesium Chloride ($MgCl_2$), Calsium Chloride ($CaCl_2$), Calcium Magnesium Acetate (CMA) and Potassium formate (KCOOH) (Klein-Paste, 2013).

2.3.1 Colligative properties

Colligative denotes to 'depending on the collection', and is a unifying term for four different properties of dilute solutions. What unites these properties is that they will depend on the number of dissolved molecules and ions present in the solution, rather than the characteristics of the solute dissolved in the solvent. The four different properties that are subject to this term are freezing point depression, boiling point elevation, vapor pressure lowering and osmotic pressure.

The existence of solute results in a reduction of chemical potential in the solvent, and is the reason for the changes in the colligative properties. Figure 2 graphically displays the chemical potential for pure liquid and a solution, as a function of the temperature. The lines are in reality curved, but are simplified for this figure. The chemical potential is the potential of a medium to undergo a change in the system. As can be seen from the figure, the changes have a much larger effect on the freezing point than on the boiling point.

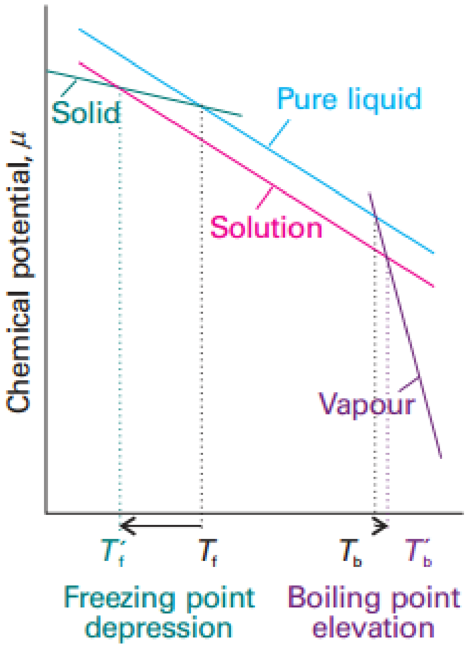

Figure 2 - Chemical potential for a pure liquid and a solvent (Atkins and de Paula, 2006)

As previously explained, the colligative properties will depend of the amount of dissolved particles in the solution. Thus the gap between the lines for pure liquid and solution will increase when adding more solute to the solution, and thereby increasing the changes in freezing and boiling point.

2.3.2 Effect of road salt

Figure 3 shows the phase diagram for sodium chloride dissolved in water, which is the most common salt used in Norway for winter maintenance. In this diagram the different phases are presented as the correlation between temperature and concentration of solute in water. It is compiled of four different phases, separated by the freezing curve, the solubility curve and the eutectic temperature.

The freezing curve shows the freezing point depression when the concentration of NaCl is increased. The solubility curve shows the boundary for when no more salt can be diluted at the temperature in question. The two curves intersect in what is called the eutectic point, which is when the eutectic temperature has been reached. Beyond this temperature no solute can be dissolved in the solvent, thereby giving making it impossible to decrease the freezing point temperature by adding sodium chloride. As an example, a solution of 10% NaCl will reduce the freezing point of water to about -7.5°C.

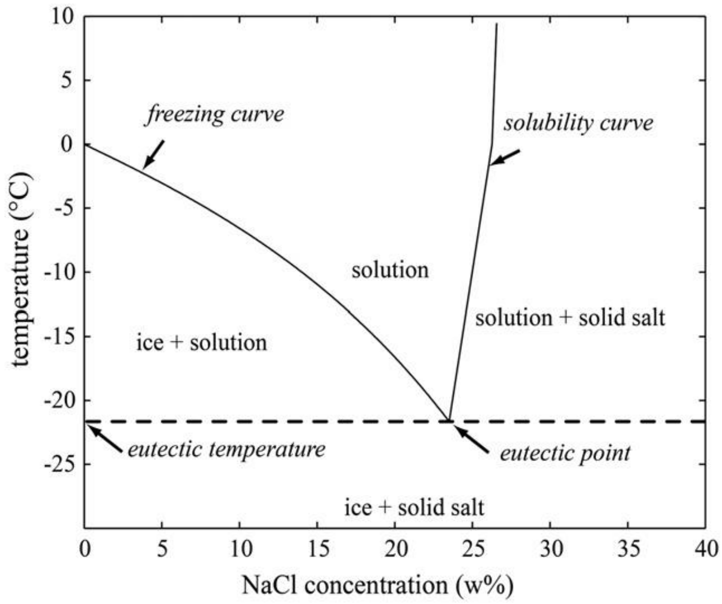

Figure 3 - Phase diagram sodium chloride (Klein-Paste and Wåhlin, 2013)

In addition to reducing the freezing point of water, the solute can retard the freezing process. Figure 4 illustrates an example of how the freezing point of water is affected by the addition of a solute of sodium chloride. In point A the solution is 10°C and has a 5% concentration of NaCl. It is applied on a surface with a temperature of -10°C. The solution will release heat energy to its colder surroundings, thereby becoming colder itself. When the solution reaches zero degrees Celsius it will not start to freeze, as pure water would, because the freezing point of the water has been reduced due to the presence of sodium chloride. Instead the water will stay in its liquid state until reaching the temperature in point B. Here some of the water will start to freeze, but not all of it. As some of the water is removed from the solution when turning to ice, the concentration of NaCl in the solution starts to increase, resulting in further depression of the freezing point. Finally the system will have reached the temperature of its surroundings, and there will become an equilibrium state consisting of brine and ice.

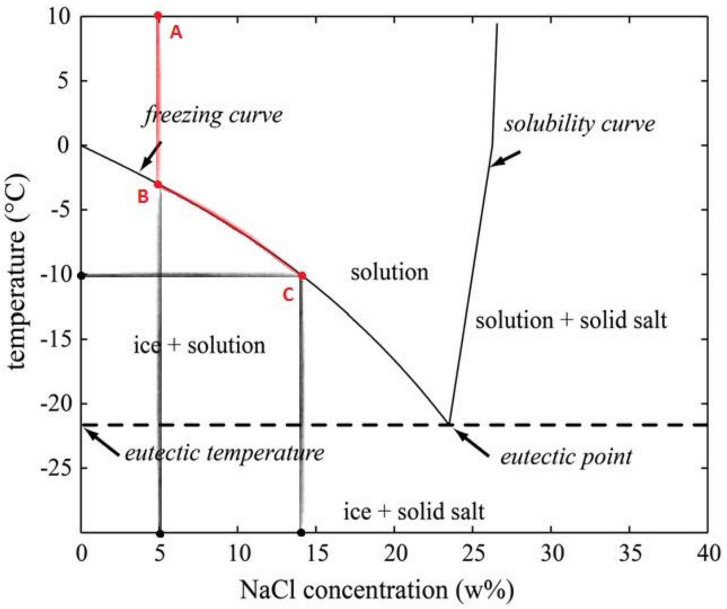

Figure 4 - Illustration of freezing point depression

2.3.3 Alternative mechanism for wet pavement anti-icing

Traditionally the mechanisms of using chemicals for wet pavement anti-icing are only explained with the effect of the freezing point depression, as explained in the previous section. But in addition to these effects, the road salt will also reduce the strength of the ice on the road (Klein-Paste and Wåhlin, 2013). Studies show that the presence of chemicals in itself will soften the ice structure (Haavasoja et al., 2012). Klein-Paste and Wåhlin (2013) proposed the following mechanism for anti-icing:

1. Anti-icing chemicals change the microstructure of the ice that forms during freezing.
2. Anti-icing chemicals thereby weaken the ice.
3. The ice will disintegrate into small pieces under the exposed traffic loading.
4. The re-exposed pavement texture provides sufficient friction.

They also suggested a new parameter called the equilibrium brine fraction F_b, which is the fraction of water that remains unfrozen after the cool-down.

$$F_b(T) = \frac{c}{c_f(T)} \quad (1)$$

The parameter is composed on the basis of a given temperature and chemical concentration in a solution. In equation (1) the numerator c refers to the salt concentration of the solution utilized, before freezing. The denominator $c_f(T)$ refers to the salt concentration given by the freezing point depression curve. That is, the concentration of solute the solution would have in an equilibrium state for the temperature in question.

2.3.4 Additives to road salting

Because of the negative environmental effects of using road salts, other chemicals have been developed to reduce the amount of salt used. Many of these are by-products from agriculture products. They are referred to as Agricultural By-Products and are used as additives to road salt for winter maintenance.

This kind of additive is used in countries like the UK, Canada and USA. A report compiled by Parsons Brinckerhoff and WSP (PSWSP) for the Highways Agency in the United Kingdom, shows that most users justifies the use of ABP in road salting based on only anecdotal evidence provided. Research to back up the producers claims to its effects is not extensive enough. (Highway-Agency, 2007)

Some of the advantages claimed for many ABPs are that they have longer longevity on the road, better adhesion to the road, reduce corrosion damage to the surroundings and they have greater accuracy in spreading. Research has been done to investigate whether an ABP reduces corrosion on metals (Petkuviene and Paliulis, 2009). It showed that for some alloys the additive had a positive effect. The other claimed advantages have to be further researched.

3 Method

The experimental setup for this project has been developed earlier (Klein-Paste and Wåhlin, 2013). The objective of it is to find if using an additive of Agricultural By-Product to road salt would weaken the ice just as much as a solution without ABPs. Testing on sodium chloride solutions had been done previously by Klein-Paste and Wåhlin (2013), and data from that project were provided as a basis of comparison.

The Agricultural By-Product used in the laboratory experiment for this master thesis was Safecote. Solutions consisting of sodium chloride and Safecote were placed on asphalt substrates in an ice laboratory. The laboratory was preset to a specific temperature and the substrates were cooled during the night. Traffic load was simulated on to the ice the following day with the help of a British Pendulum Tester, and the weakening of the ice was registered.

After testing, the collected data was analyzed using binomial logistic regression, and then compared to the data from the previous experiment.

3.1 Laboratory experiment

This section describes the different equipment that was used in the experiment, and the procedure that was followed in order to collect data for further analysis.

3.1.1 Setup for the laboratory experiment (experimental details?)

Figure 5 shows the set up for the asphalt substrates during freezing in the laboratory experiment. 3 mm thick film of solutions were spread evenly on top of eight asphalt substrates (2). The substrates were circular with a diameter of 14 cm, and were each placed on three screws (3) screwed on to a wooden board. By adjusting these screws, the substrates could be leveled correctly so that the water film on top would keep the same thickness over the entire asphalt area. The substrates also had a plate of stainless steel glued to the bottom, which made it easier to get leveled. After leveling the substrates it is important to mark where on the plate they should be placed, so that they will stay leveled and in the right place through every experiment.

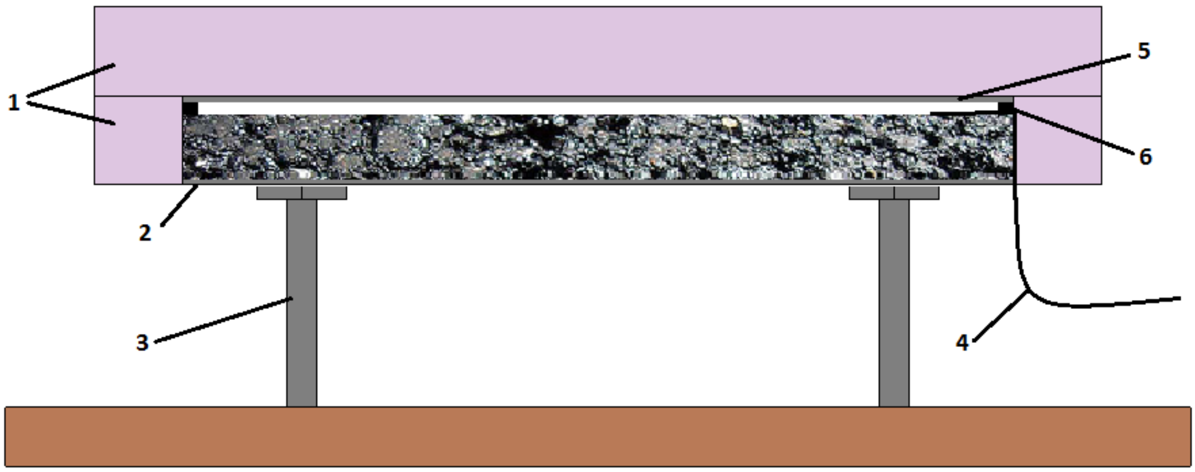

Figure 5 – Setup for the performed laboratory experiment

1 – insulating foam, 2 – asphalt substrate, 3 – screw,

4 – thermocouple cable, 5 – stainless steel plate, 6 – rubber gasket ring

A rubber gasket ring (6) was fastened around the edge with a tape around the side, to keep the solution from escaping off the edge of the substrates. The rubber gasket ring, along with a stainless steel plate (5) placed on top, kept the solution from evaporating or sublimating. 20 mm thick insulating foam (1) was placed around the edge and on top of the asphalt substrates to prevent heat flow in those directions. This way, there would mainly be heat flow to the water film from the bottom of the substrates. Klein-Paste and Wåhlin (2013) also performed experiments with the insulation foam placed on the bottom instead of the top of the substrates. They referred to this as top cooling, while the one used in this thesis was referred to as bottom cooling. They found that it did not matter much what type of cooling was used for the tests.

Every substrate had a thermocouple cable (4) fastened to it to measure the temperature of the asphalt under the water film. The cables were connected to a data logger, and using PC200W Datalogger Support Software gave the possibility of tracking the temperature throughout the freezing process.

Figure 6 – Substrate connected to the data logger

As the picture in Figure 7 shows, the process of setting up the table was done outside the ice laboratory beforehand. Leveling the substrates on the table is precision work, and took some time. Also, the substrates had been used in a previous project, and all had to be tested beforehand, as the thin thermocouple cables were very easily damaged.

Figure 7 - Setting up the table for the laboratory experiment

3.1.2 British Pendulum Tester

After the system had gone through supercooling and reached the temperature of its surroundings, the strength of the ice on the substrates was tested. This was carried out with the help of a British Pendulum Tester, which can be seen in Figure 8. It is a pendulum arm with a 70 mm wide rubber slider attached to the end of it. The instrument is generally used for testing skid resistance, where the friction resistance between the asphalt surface and the rubber slider is measured. In the experiment carried out in this project the friction resistance was not relevant for the analysis. The instrument was only used to simulate the mechanical load on the ice that would otherwise be caused by traffic.

Figure 8 - British Pendulum Tester

The British Pendulum Tester was leveled on to a table using the three screws on the instrument. Afterwards a "frame" for the substrates was screwed on to the table, as can be seen in Figure 9. This was so that the substrates would stand firmly when the rubber slider went over the ice surface. According to Klein-Paste and Wåhlin (2013) the pendulum accelerates to a speed of 3 m/s, which corresponds to a normal load of 22 N and a nominal contact pressure of about 145 kPa.

Before testing a specimen with the British Pendulum Tester the temperature of it was registered. Then the specimen was placed in the frame and the pendulum carefully adjusted so that the rubber slider struck about 55 $\pm$ 5 mm of the ice surface when passing. Then the pendulum was dropped five times on the specimen and then removed from the frame.

Figure 9 - Setup for testing of a solution with the British Pendulum Tester

The damage done on the ice by the rubber slider was visually evaluated and registered in the column for result in the form in Table 2. Three levels of damage were employed, depending on how much ice on the passing area was removed. If there was less than 25 percent ice removed from the area, it was classified as "not failed". If more than 75 percent ice was removed, it was classified as "failed". The tests that showed removal somewhere between (25%-75%) these two was classified as "partly failed". An example of a "not failed" and a "failed" ice surface is shown in Figure 10.

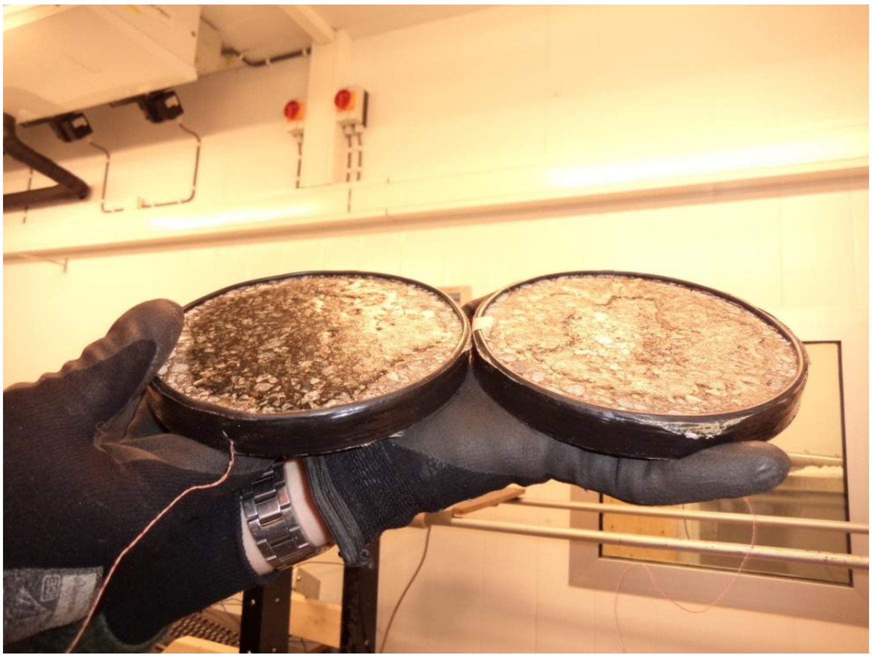

Figure 10 - Substrates after testing with British Pendulum Tester

3.2 Calculations prior to testing

This section describes the calculations that had to be done prior to the laboratory experiments. Tests were performed with different concentrations of solute at six different temperatures, and which ones should be used had to be decided beforehand.

3.2.1 The Equilibrium Brine Friction

The freezing depression curve in Figure 11 is based on a table of properties for sodium chloride solution shown in Appendix A, from the CRC Handbook of Chemistry and Physics (Haynes, 2013).

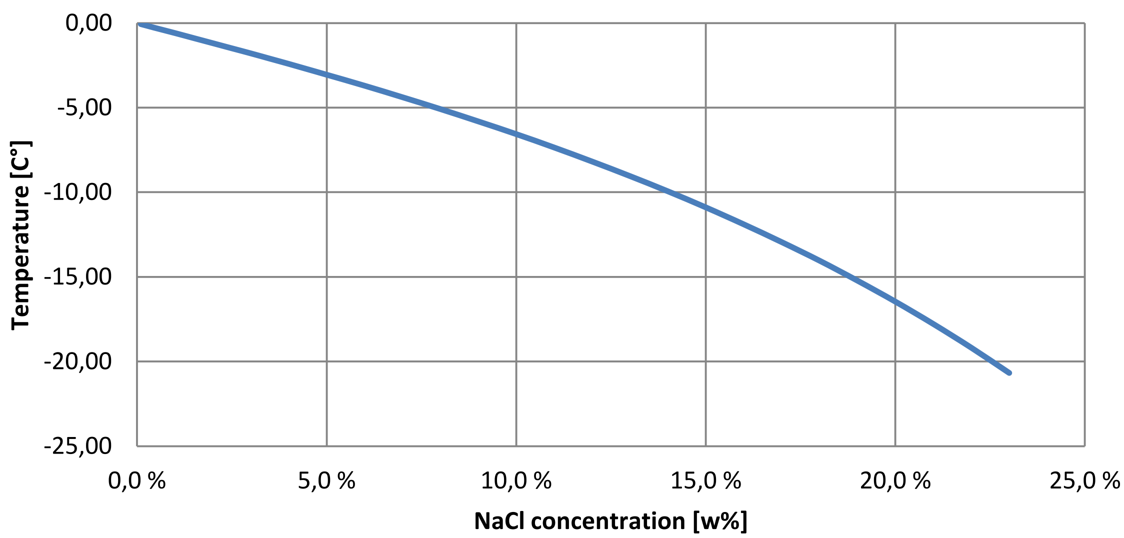

Figure 11 - Freezing point depression curve

By using the regression data analysis tool in Microsoft Office Excel, an equation can be found for the correlation between temperature and NaCl concentration of the given freezing point depression data, which is the equation for $c_f(T)$.

An extract from the Summary Output given by the regression analysis is shown in Table 1, and the complete Summary Output can be found in Appendix B. The extract is a list of the parameters relevant to deriving an equation for the freezing point depression curve.

Table 1 – Extract from the Summary Output of the Excel Regression Analysis

Regression Statistics	
Multiple R	0.999997792
R Square	0.999995584
Adjusted R Square	0.999995111
Standard Error	0.000165957
Observations	32

	Coefficients
Point of intersection	-0.0004
T - variable 1	-0.0178
T - variable 2	-0.0004
T - variable 3	-0.000005

As can be deduced from the coefficient of determination (R Square) in the extract, the applied statistical regression model fits to the acquired data points very well. The equation for the freezing curve that can be derived from the coefficients in Table 1 will therefore become:

$$c_f(T) = -0.000005\ \mathrm{T}^3 - 0.0004\ \mathrm{T}^2 - 0.0178\ \mathrm{T} - 0.0004 \qquad \textbf{(2)}$$

Employing a third degree polynomial trendline to the freezing point depression data, gives the same equation for the freezing curve as the regression. Figure 12 displays graphically how well the derived equation (2) matches the empirical data.

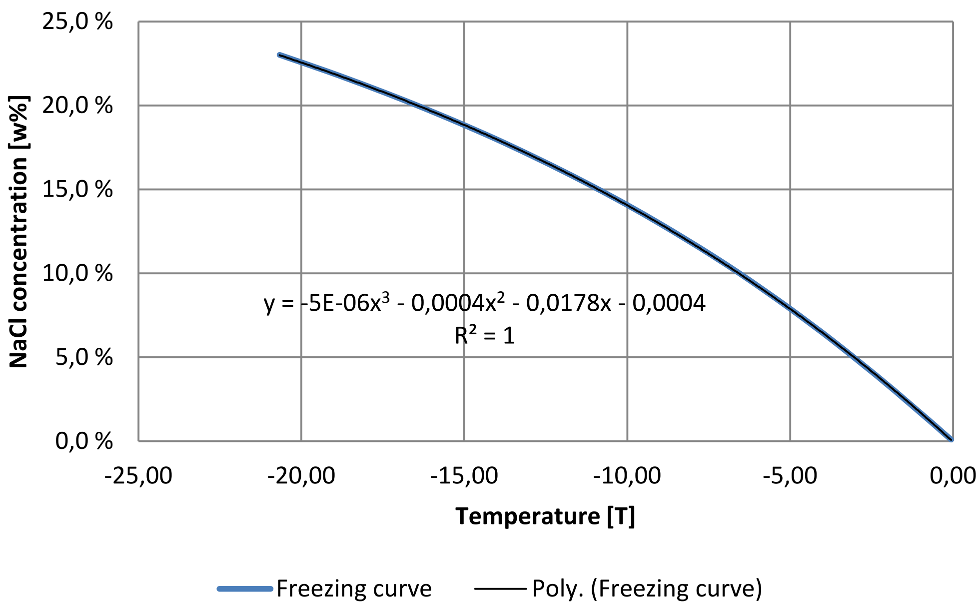

Figure 12 - Transformed freezing curve and its trendline

3.2.2 Solutions

The results from the laboratory experiments performed by Klein-Paste and Wåhlin (2013) showed that when using sodium chloride solutions on the road, using an equilibrium brine fraction of about 0.25 gives a 50 percent chance that the ice will be broken by the mechanical load. Therefore, F_b = 0.25 were used in the new laboratory experiments as a basis for deciding which solutions should be tested first for each temperature.

In the solutions prepared for the experiment, 97% of the solute was sodium chloride and 3% of it was the Agricultural By-Product. A solution with 10% solute was first prepared in a bigger bottle, and then other solutions required for the upcoming test were prepared in smaller bottles of about 50 mL. The bottles had to be stored in a refrigerated room to keep the ABP in the bottle from starting to mold.

The tests were performed at -5°C, -10°C, -12.5°C, -15°C, -17.5°C and 20°C. The equilibrium brine fraction were calculated for NaCl-concentrations between zero and ten percent, with a step of 0.25%. A complete table for all the calculated equilibrium brine fractions is enclosed in Appendix C.

For each new temperature that tests were performed, eight solutions was chosen on the basis of the equilibrium brine fraction of 0.25 found by Klein-Paste and Wåhlin (2013). The concentrations chosen had steps of 0.5% between each, with F_b = 0.25 in the middle. As an example, the first test performed at -5°C with the equilibrium brine fraction and the corresponding concentration is shown in Table 2 - Form used to register data.

Table 2 - Form used to register data, example with -5°C

Concentration brine big bottle		10%						
Volume little bottle		50	mL					
Temperature		-5	°C					
cf (T)		7.9%						
Calculated volume [mL] solute in small bottles	**c**	**F_b**	**Real volume of solute in small bottle**	**Real total volume of solution in small bottle**	**No. Substrate**	**Temperature**	**Result**	
2.5	0.50%	0.06						
5	1.00%	0.13						
7.5	1.50%	0.19						
10	2.00%	0.25						
12.5	2.50%	0.32						
15	3.00%	0.38						
17.5	3.50%	0.44						
20	4.00%	0.50						

The real volume of solute and solvent was registered when making the solutions and the temperature of the substrate was registered before the mechanical load testing. This made it possible to calculate accurate equilibrium brine for the solutions that was tested.

Tests were performed twice for some temperatures, and three times for others. After the first test with one temperature, the concentrations were adjusted for the next test. The concentration where there was a transition between "failed" and "not failed" were used as the next "middle" concentration, and the steps between each was adjusted to 0.25%.

The substrates were all numbered to keep record of what concentration was on them. The same substrate was not used for the same concentration twice, if it was possible to do so.

Tests were performed at six different temperatures, either two or three times. Table 3 shows an overview of the number of tests performed for the corresponding temperature.

Table 3 - Overview of number of performed tests for each temperature

Temperature	-5 °C	-10 °C	-12.5 °C	-15 °C	-17.5 °C	-20 °C
Number of performed tests	2 (-1)	2 (-1)	2	3	3	3

The numbers in the brackets are the number of substrates that were removed from the results for the temperature in question. One of these substrates was not tested with the British Pendulum Tester because the thermocouple cable on it was damaged. Subsequently, the same substrate could not be used for the next test, as the substrate was being repaired during testing. Some of the other thermocouple cables were damaged during later tests, but in those cases the load testing was carried out as usual. Instead the mean temperature of the other substrates in the test was used.

3.3 Binomial logistic regression

Binomial logistic regression was employed to calculate the probability that an ice sample fails for a given brine fraction. The dependent variable F_b would result in one of two outcomes, “sufficiently good friction” or “slippery conditions”, and the outcomes were respectively given the predictive values of 1 and 0. The probability p of the outcome as a function of the dependent variable F_b was therefore given by equation (3):

$$p_{F_b} = \frac{1}{1 + e^{-(\beta_0 + \beta_1 F_b)}} \quad \textbf{(3)}$$

This is the same probabilistic statistical classification model that was used for the previous experiment performed by Klein-Paste and Wåhlin (2013), which gives the possibility of comparing data for NaCl with and without the additive. The “partly failed” and “not failed” data points were classified as “slippery conditions”, and the “failed” data were classified as “sufficiently good friction”.

The calculations were done with the help of the statistical software MATLAB® (The MathWorks, 1994-2014). Both linear fitting coefficients β_0 and β_1 were found, and five different confidence levels of failure. The equation was presented graphically for both new and old datasets.

4 Results

In total there were performed 16 tests, but one of them was removed from the final data collection set because the results for this test diverged completely from the other tests. It was found that the substrates probably had not dried sufficiently enough before starting a new test. Completely filled out forms can be found for each test that was performed in Appendix D.

Figure 13 graphically displays the results for all the tests that were performed. Here a few of the tests were labeled as between "not failed" and "partly failed" or "failed" and "partly failed" because it was difficult to determine the percentage of failure. "Failed"/partly failed" (F/PF) was later on classified as "sufficiently good friction" in the binomial logistic regression, alongside with the "failed" results. All the other data points were classified as "slippery conditions".

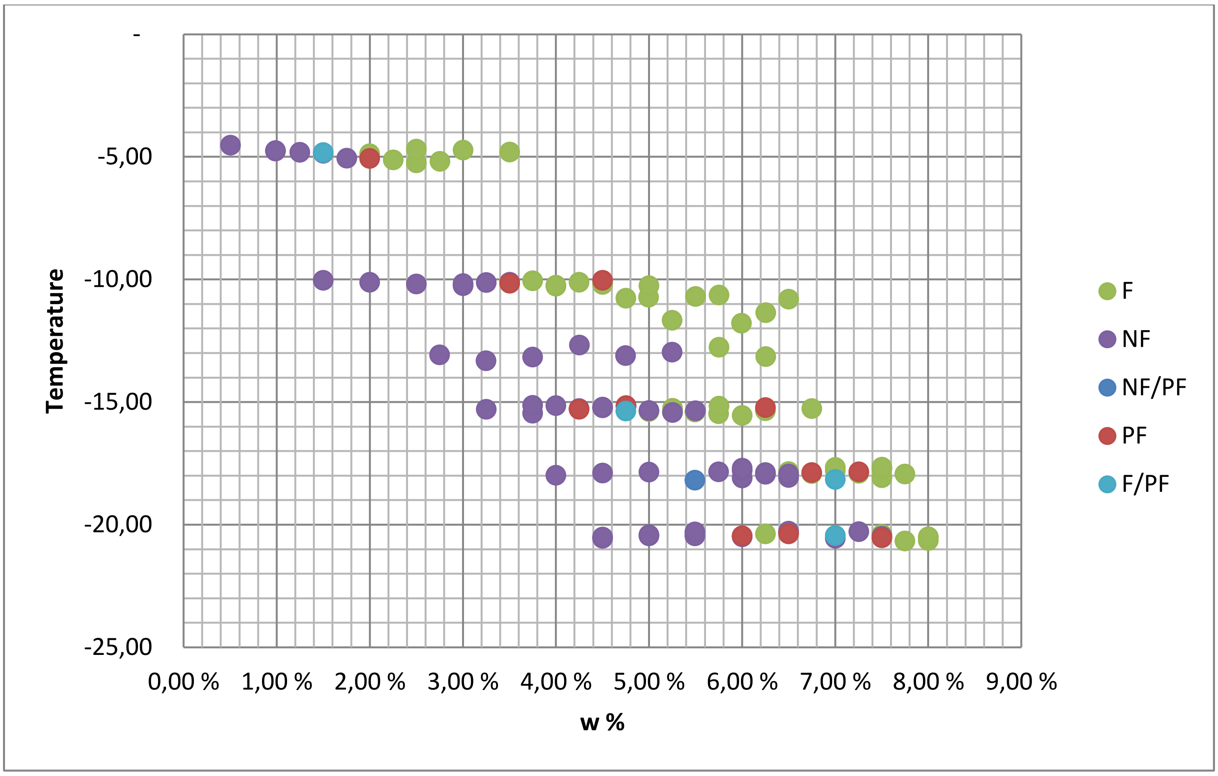

Figure 13 - Complete summary of all tests performed, displayed graphically

Figure 14 presents the collected datapoints in a graph alongside with the freezing curve for NaCl and F_b = 0.28.

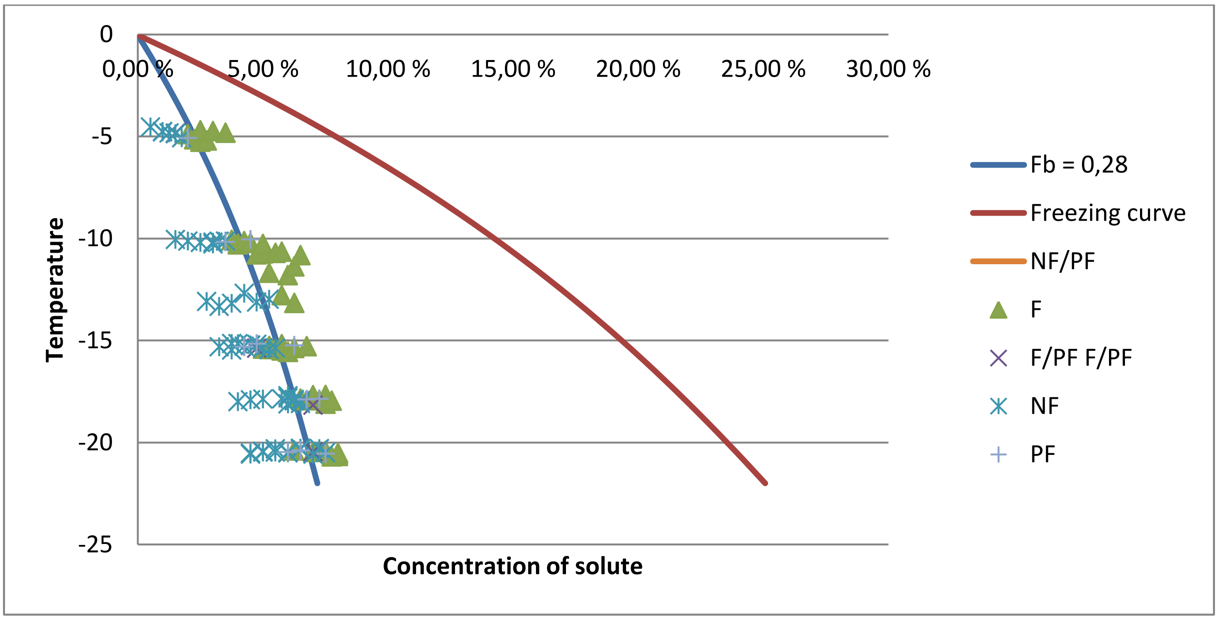

Figure 14 - Data points presented alongside with the freezing curve for NaCl and F_b = 0.28

In Figure 15 the "partly failed" data points are reclassified and all the results are presented with the correlation of equilibrium brine fraction and temperature. A complete calculation of equilibrium brine fraction for all the tests performed can be found in Appendix C.

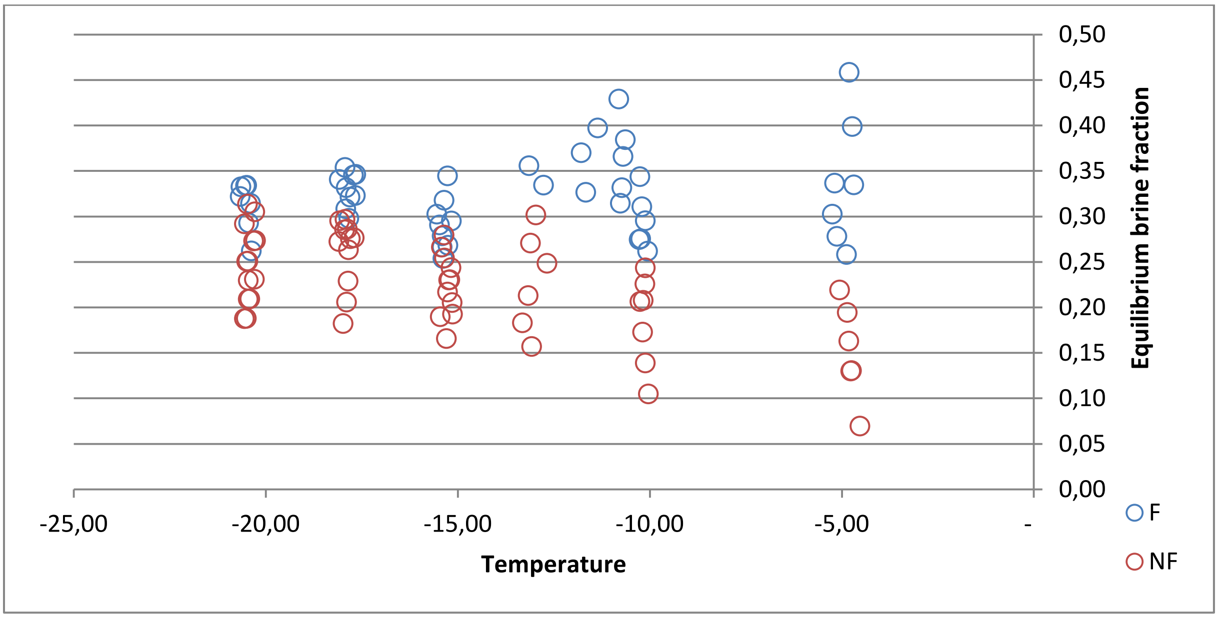

Figure 15 - Results with reclassified datapoints

The binomial logistic regression done in MATLAB (The MathWorks, 1994-2014) gave the linear fitting coefficients $\beta_0 = -10.9028$ and $\beta_1 = 38.1300$. The calculated confidence levels of failure for the performed experiment are shown in Table 4 - Confidence levels of failure for performed experiment

Table 4 - Confidence levels of failure for performed experiment

Confidence level of failure	5%	50%	95%	99%	99.99%
Brine fraction	0.20921	0.28629	0.36336	0.40741	0.52753

Figure 16 shows the distribution for the dataset collected in the laboratory experiment, made with the help of MATLAB (The MathWorks, 1994-2014).

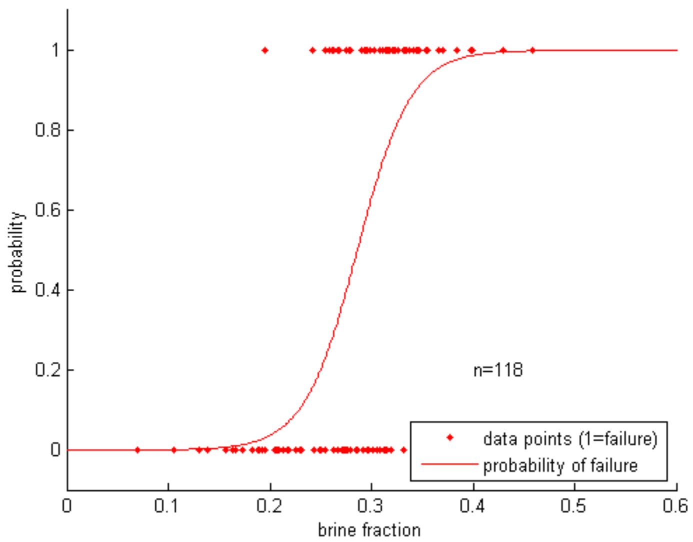

Figure 16 – Probability of ice failure as a function of brine fraction

5 Discussion

The objective of this thesis was to find if a solute where 3% of the sodium chloride had been replaced with an Agricultural By-Product would reduce the strength of the ice to the same degree as brine with only NaCl used as solute would. Figure 17 presents the distribution for the experiment performed in this thesis with a red line, and the distribution from the previous experiment with a black line.

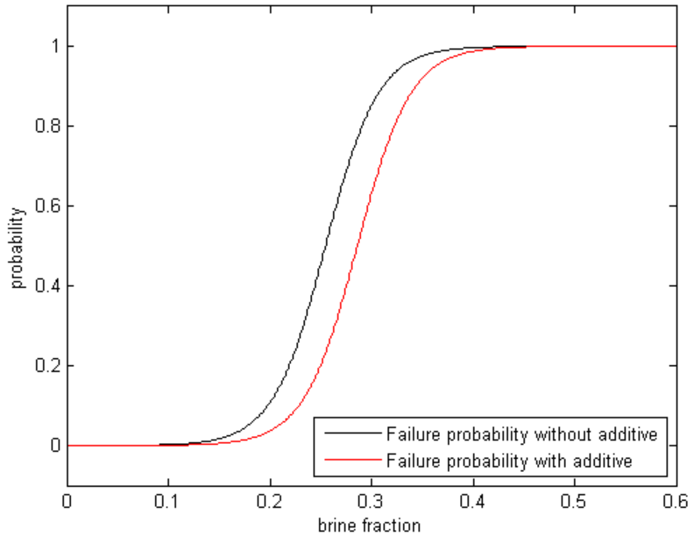

Figure 17 - Results from both experiments

There is a distinctive difference between the two experiments, where the one with ABPs in it clearly needs a bigger equilibrium brine fraction to weaken the ice. Table 5 shows the confidence levels of failure for the previous experiment, which clarifies the difference between the two solutions. For the previous performed experiment a brine fraction of about F_b = 0.255 give a 50% chance of breaking the ice, while the previously performed experiment needed a brine fraction of about F_b = 0.286. In other words one needs 3% more solution with additives to obtain the same weakening of the ice compared to the one without. This can be explained by the fact that by making the solution with additives 3% of the sodium chloride was replaced. This means that there cannot be seen a positive effect regarding weakening of the ice by adding APB.

Table 5 - Confidence levels of failure for previous experiment

Confidence level of failure	5%	50%	95%	99%	99.99%
Brine fraction	0.17918	0.25526	0.33133	0.37437	0.49349

What can also be seen from Figure 17 - Results from both experimentsis that the shape of the s-curve is about the same for the two experiments. By studying the confidence intervals for the two experiments, the variability for the two experiments can be found. The 90% confidence interval for experiment with ABP is shown in (4), and (5) shows the confidence interval for the experiment without ABP.

$$CONF_{90\%} = \{0.20921 \leqq \mu \leqq 0.36336\} \quad (4)$$

$$CONF_{90\%} = \{0.17918 \leqq \mu \leqq 0.33133\} \quad (5)$$

The variability of (4) is $0.36336 - 0.20921 = 0.15425$ and the variability of (5) is $0.33133 - 0.17918 = 0.15215$. The two experiments gave about the same variability, which means that the transition from failed to not failed is unchanged by the additive. This suggests that adding additives does not change the microstructure of the ice in such a way that it further weakens or strengthens the ice.

6 Conclusion

A laboratory experiment has been carried out to investigate if agricultural by-product additives can increase the weakening of ice that forms when a wet pavement freezes. Pure sodium chloride solutions where compared to sodium chloride solutions with 3% additives. There was neither found a positive nor a negative effect of adding the agricultural by-product. It is important to note that ABPs still can have positive effect, for example increased longevity on the road surface.

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APPENDIX A:

Freezing point depression data for sodium chloride solution

Mass%	-Δ/ºC
0,1%	-0,06
0,2%	-0,12
0,3%	-0,18
0,4%	-0,24
0,5%	-0,30
1,0%	-0,59
1,5%	-0,89
2,0%	-1,19
2,5%	-1,49
3,0%	-1,79
3,5%	-2,10
4,0%	-2,41
4,5%	-2,73
5,0%	-3,05
6,0%	-3,70
7,0%	-4,38

Mass%	-Δ/ºC
8,0%	-5,08
9,0%	-5,81
10,0%	-6,56
11,0%	-7,35
12,0%	-8,18
13,0%	-9,04
14,0%	-9,94
15,0%	-10,89
16,0%	-11,89
17,0%	-12,94
18,0%	-14,04
19,0%	-15,22
20,0%	-16,46
21,0%	-17,78
22,0%	-19,18
23,0%	-20,67

APPENDIX B:

Complete Summary Output from Excel regression analysis

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0,99999779
R Square	0,999995584
Adjusted R Square	0,99999511
Standard Error	0,000165957
Observations	32

Analysis of variance

	df	*SS*	*MS*	*F*	*Significance F*
Regression	3	0,174647198	0,058215733	2113724,106	4,64187E-75
Residuals	28	7,7117E-07	2,7542E-08		
Total	31	0,174647969			

	Coefficients	*Standard Error*	*t-Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95,0%*	*Upper 95,0%*
Point of intersection	-0,0004	6,4153E-05	-5,86488809	2,634E-06	-0,00050766	-0,00024484	-0,00050766	-0,00024484
T - variable 1	-0,0178	3,7227E-05	-478,290845	2,527E-56	-0,01788142	-0,0177289	-0,01788142	-0,0177289
T - variable 2	-0,0004	4,7857E-06	-87,1363374	1,2232E-35	-0,00042681	-0,00040721	-0,00042681	-0,00040721
T - variable 3	-0,000005	1,6217E-07	-28,1915762	4,2447E-22	-4,9041E-06	-4,2397E-06	-4,9041E-06	-4,2397E-06

APPENDIX C:

Calculations for equilibrium brine fractions for all performed tests

Real c	**Temperature**	**Result**	**C_f (T)**	**F_b**
5,00 %	-15,39	F	19,7 %	0,2539
2,00 %	-4,88	F	7,7 %	0,2582
3,75 %	-10,06	F	14,3 %	0,2619
6,25 %	-20,38	F	23,8 %	0,2621
5,25 %	-15,41	F	19,7 %	0,2663
5,25 %	-15,26	F	19,6 %	0,2684
4,00 %	-10,28	F	14,6 %	0,2746
4,00 %	-10,24	F	14,5 %	0,2754
2,25 %	-5,13	F	8,1 %	0,2782
5,49 %	-15,41	F	19,7 %	0,2787
5,75 %	-15,48	F	19,8 %	0,2907
7,00 %	-20,45	F	23,9 %	0,2929
5,75 %	-15,17	F	19,5 %	0,2951
4,25 %	-10,12	F	14,4 %	0,2953
6,50 %	-17,84	F	21,8 %	0,2980
6,00 %	-15,55	F	19,8 %	0,3025
2,50 %	-5,25	F	8,3 %	0,3025
6,75 %	-17,92	F	21,9 %	0,3084
4,50 %	-10,21	F	14,5 %	0,3107
7,50 %	-20,40	F	23,9 %	0,3144
4,75 %	-10,77	F	15,1 %	0,3145
6,25 %	-15,36	F	19,7 %	0,3178
7,00 %	-17,81	F	21,8 %	0,3213
7,75 %	-20,67	F	24,1 %	0,3220
7,00 %	-17,67	F	21,7 %	0,3231
5,25 %	-11,67	F	16,1 %	0,3266

5,00 %	-10,73	F	15,1 %	0,3316
7,25 %	-17,91	F	21,9 %	0,3317
8,00 %	-20,65	F	24,1 %	0,3326
8,00 %	-20,53	F	24,0 %	0,3339
8,00 %	-20,50	F	23,9 %	0,3342
5,75 %	-12,77	F	17,2 %	0,3344
2,50 %	-4,69	F	7,5 %	0,3347
2,75 %	-5,19	F	8,2 %	0,3366
7,50 %	-18,09	F	22,0 %	0,3407
5,00 %	-10,26	F	14,5 %	0,3438
6,75 %	-15,27	F	19,6 %	0,3446
7,50 %	-17,72	F	21,7 %	0,3455
7,50 %	-17,66	F	21,7 %	0,3463
7,75 %	-17,94	F	21,9 %	0,3539
6,25 %	-13,15	F	17,6 %	0,3558
5,50 %	-10,70	F	15,0 %	0,3660
5,99 %	-11,79	F	16,2 %	0,3701
5,75 %	-10,64	F	15,0 %	0,3844
6,25 %	-11,36	F	15,7 %	0,3973
3,00 %	-4,73	F	7,5 %	0,3988
6,50 %	-10,81	F	15,1 %	0,4291
3,50 %	-4,81	F	7,6 %	0,4584
1,50 %	-4,83	F/PF	7,7 %	0,1956
4,75 %	-15,37	F/PF	19,7 %	0,2415
7,00 %	-20,45	F/PF	23,9 %	0,2929
7,00 %	-18,16	F/PF	22,1 %	0,3171
0,50 %	-4,53	NF	7,2 %	0,0696
1,50 %	-10,04	NF	14,3 %	0,1049
0,99 %	-4,77	NF	7,6 %	0,1301
0,99 %	-4,75	NF	7,6 %	0,1306
2,00 %	-10,12	NF	14,4 %	0,1389
2,75 %	-13,08	NF	17,5 %	0,1571
1,25 %	-4,82	NF	7,7 %	0,1630
3,25 %	-15,30	NF	19,6 %	0,1658

2,50 %	-10,19	NF	14,5 %	0,1729
4,00 %	-17,99	NF	21,9 %	0,1823
3,25 %	-13,32	NF	17,7 %	0,1831
4,50 %	-20,56	NF	24,0 %	0,1877
4,50 %	-20,51	NF	23,9 %	0,1880
3,75 %	-15,46	NF	19,8 %	0,1898
3,75 %	-15,14	NF	19,5 %	0,1926
1,50 %	-4,86	NF	7,7 %	0,1945
4,00 %	-15,15	NF	19,5 %	0,2054
4,50 %	-17,90	NF	21,9 %	0,2059
3,00 %	-10,26	NF	14,5 %	0,2064
3,00 %	-10,17	NF	14,4 %	0,2078
5,00 %	-20,46	NF	23,9 %	0,2091
5,00 %	-20,42	NF	23,9 %	0,2094
3,75 %	-13,17	NF	17,6 %	0,2131
4,25 %	-15,27	NF	19,6 %	0,2169
1,75 %	-5,06	NF	8,0 %	0,2192
3,25 %	-10,13	NF	14,4 %	0,2258
5,00 %	-17,86	NF	21,8 %	0,2290
5,49 %	-20,46	NF	23,9 %	0,2298
4,50 %	-15,24	NF	19,6 %	0,2302
4,50 %	-15,21	NF	19,5 %	0,2305
5,49 %	-20,30	NF	23,8 %	0,2310
3,50 %	-10,12	NF	14,4 %	0,2435
4,75 %	-15,18	NF	19,5 %	0,2437
4,25 %	-12,68	NF	17,1 %	0,2485
6,00 %	-20,50	NF	23,9 %	0,2507
6,00 %	-20,47	NF	23,9 %	0,2509
5,00 %	-15,35	NF	19,7 %	0,2543
5,75 %	-17,85	NF	21,8 %	0,2634
5,25 %	-15,43	NF	19,7 %	0,2663
4,75 %	-13,11	NF	17,5 %	0,2707
6,00 %	-18,10	NF	22,0 %	0,2724
6,50 %	-20,32	NF	23,8 %	0,2731

6,50 %	-20,27	NF	23,8 %	0,2735
6,00 %	-17,80	NF	21,8 %	0,2755
6,00 %	-17,70	NF	21,7 %	0,2766
5,50 %	-15,36	NF	19,7 %	0,2796
6,25 %	-17,95	NF	21,9 %	0,2854
6,25 %	-17,88	NF	21,8 %	0,2861
7,00 %	-20,56	NF	24,0 %	0,2919
6,50 %	-18,08	NF	22,0 %	0,2953
6,50 %	-17,94	NF	21,9 %	0,2968
5,25 %	-12,97	NF	17,4 %	0,3016
7,25 %	-20,29	NF	23,8 %	0,3051
7,50 %	-20,48	NF	23,9 %	0,3136
5,49 %	-18,19	NF/PF	22,1 %	0,2485
4,25 %	-15,30	PF	19,6 %	0,2166
3,50 %	-10,16	PF	14,4 %	0,2428
4,75 %	-15,15	PF	19,5 %	0,2441
2,00 %	-5,07	PF	8,0 %	0,2495
6,00 %	-20,46	PF	23,9 %	0,2510
6,50 %	-20,38	PF	23,8 %	0,2726
6,75 %	-17,88	PF	21,8 %	0,3089
7,50 %	-20,54	PF	24,0 %	0,3130
4,50 %	-10,04	PF	14,3 %	0,3149
6,25 %	-15,23	PF	19,5 %	0,3198
7,25 %	-17,85	PF	21,8 %	0,3324

APPENDIX D:

Forms with registered results for all performed tests

Concentration brine big bottle	10 %	
Volume little bottle	50	mL
Temperature	-5	°C
cf (T)	7.9 %	

Calculated volume [mL] solute in small bottles	c	Fb	Real volume of solute in small bottle	Real total volume of solution in small bottle	No. Substrate	Temperature	Result
2.5	0.50 %	0.06	2.52	50.01	2	-4.53	NF
5	1.00 %	0.13	5.01	50.77	1	-4.75	NF
7.5	1.50 %	0.19	9.00	60.00	7	-4.86	NF
10	2.00 %	0.25	10.00	50.03	8	-4.88	F
12.5	2.50 %	0.32	12.51	50.03	4	-4.69	F
15	3.00 %	0.38	15.02	50.03	5	-4.73	F
17.5	3.50 %	0.44	17.52	50.01	3	-4.81	F
20	4.00 %	0.50	20.00	50.01	Plate 6 ødelagt	-	-

Concentration brine big bottle	10 %	
Volume little bottle	50	mL
Temperature	-5	°C
cf (T)	7.9 %	

Calculated volume [mL] solute in small bottles	**c**	**Fb**	**Real volume of solute in small bottle**	**Real total volume of solution in small bottle**	**No. Substrate**	**Temperature**	**Result**
5.00	1.00 %	0.13	5.01	50.77	3	-4.77	NF
6.25	1.25 %	0.16	6.25	50.07	7	-4.82	NF
7.50	1.50 %	0.19	9.00	60.00	4	-4.83	PF/F
8.75	1.75 %	0.22	8.77	50.03	8	-5.06	NF
10.00	2.00 %	0.25	10.00	50.03	5	-5.07	PF
11.25	2.25 %	0.28	11.26	50.00	1	-5.13	F
12.50	2.50 %	0.32	12.51	50.03	6	-5.25	F
13.75	2.75 %	0.35	13.78	50.04	2	-5.19	F

Concentration brine big bottle	10 %	
Volume little bottle	50	mL
Temperature	-10	°C
cf (T)	14.3 %	

Calculated volume [mL] solute in small bottles	c	Fb	Real volume of solute in small bottle	Real total volume of solution in small bottle	No. Substrate	Temperature	Result
7.50	1.50 %	0.11	9.00	60.00	1	-10.04	NF
10.00	2.00 %	0.14	10.00	50.03	2	-10.12	NF
12.50	2.50 %	0.18	12.51	50.03	3	-10.19	NF
15.00	3.00 %	0.21	15.02	50.03	4	-10.17	NF
17.50	3.50 %	0.25	17.52	50.01	5	-10.16	PF
20.00	4.00 %	0.28	20.00	50.01	6	-10.24	F
22.50	4.50 %	0.32	22.51	50.01	7	-10.21	F
25.00	5.00 %	0.35	25.02	50.05	8	-10.26	F

Concentration brine big bottle	10 %	
Volume little bottle	50	mL
Temperature	-10	°C
cf (T)	7.9 %	

Calculated volume [mL] solute in small bottles	**c**	**Fb**	**Real volume of solute in small bottle**	**Real total volume of solution in small bottle**	**No. Substrate**	**Temperature**	**Result**
15.00	3.00 %	0.21	15.02	50.03	8	-10.26	NF
16.25	3.25 %	0.23	16.26	50.01	7	-10.13	NF
17.50	3.50 %	0.25	17.52	50.01	6	-10.12	NF
18.75	3.75 %	0.26	18.76	50.03	5	-10.06	F
20.00	4.00 %	0.28	20.00	50.01	4	-10.28	F
21.25	4.25 %	0.30	17.32	40.77	3	-10.12	F
22.50	4.50 %	0.32	22.51	50.01	2	-10.04	PF
23.75	4.75 %	0.33					

Concentration brine big bottle	10 %	
Volume little bottle	50	mL
Temperature	-12.5	°C
cf (T)	16.9 %	

Calculated volume [mL] solute in small bottles	c	Fb	Real volume of solute in small bottle	Real total volume of solution in small bottle	No. Substrate	Temperature	Result
13.75	2.75 %	0.16	13.75	50.00	6	-13.08	NF
16.25	3.25 %	0.19	16.25	50.02	4	-13.32	NF
18.75	3.75 %	0.22	18.75	50.01	8	-13.17	NF
21.25	4.25 %	0.25	21.26	50.00	5	-12.68	NF
23.75	4.75 %	0.28	16.57	34.90	2	-13.11	NF
26.25	5.25 %	0.31	26.26	50.04	7	-12.97	NF
28.75	5.75 %	0.34	28.76	50.00	3	-12.77	F
31.25	6.25 %	0.37	31.27	50.00	1	-13.15	F

Concentration brine big bottle	10 %	
Volume little bottle	50	mL
Temperature	-12.5	°C
cf (T)	16.9 %	

Calculated volume [mL] solute in small bottles	c	Fb	Real volume of solute in small bottle	Real total volume of solution in small bottle	No. Substrate	Temperature	Result
23.75	4.75 %	0.28	23.77	50.03	6	-10.77	F
25.00	5.00 %	0.30	25.00	50.05	1	-10.73	F
26.25	5.25 %	0.31	26.26	50.04	3	-11.67	F
27.50	5.50 %	0.32	27.50	50.00	2	-10.70	F
28.75	5.75 %	0.34	28.76	50.00	4	-10.64	F
30.00	6.00 %	0.35	30.05	50.14	5	-11.79	F
31.25	6.25 %	0.37	31.27	50.00	7	-11.36	F
32.50	6.50 %	0.38	32.52	50.03	8	-10.81	F

Concentration brine big bottle	10 %	
Volume little bottle	50	mL
Temperature	-15	°C
cf (T)	19.3 %	

Calculated volume [mL] solute in small bottles	**c**	**Fb**	**Real volume of solute in small bottle**	**Real total volume of solution in small bottle**	**No. Substrate**	**Temperature**	**Result**
16.25	3.25 %	0.17	16.26	50.01	6	-15.30	NF
18.75	3.75 %	0.19	18.76	50.03	5	-15.14	NF
21.25	4.25 %	0.22	17.32	40.77	4	-15.30	PF
23.75	4.75 %	0.25	23.77	50.01	3	-15.15	PF
26.25	5.25 %	0.27	26.27	50.01	2	-15.26	F
28.75	5.75 %	0.30	28.76	50.00	1	-15.17	F
31.25	6.25 %	0.32	31.27	50.03	8	-15.36	F
33.75	6.75 %	0.35	33.75	50.01	7	-15.27	F

Concentration brine big bottle	10 %	
Volume little bottle	50	mL
Temperature	-15	°C
cf (T)	19.3 %	

Calculated volume [mL] solute in small bottles	**c**	**Fb**	**Real volume of solute in small bottle**	**Real total volume of solution in small bottle**	**No. Substrate**	**Temperature**	**Result**
18.75	3.75 %	0.19	18.76	50.03	4	-15.46	NF
20.00	4.00 %	0.21	20.00	50.01	3	-15.15	NF
21.25	4.25 %	0.22	17.32	40.77	2	-15.27	NF
22.50	4.50 %	0.23	22.51	50.01	1	-15.24	NF
23.75	4.75 %	0.25	23.77	50.01	5	-15.18	NF
25.00	5.00 %	0.26	25.02	50.05	7	-15.35	NF
26.25	5.25 %	0.27	26.27	50.01	8	-15.43	NF
27.50	5.50 %	0.28	27.51	50.03	6	-15.36	NF

Concentration brine big bottle	10 %	
Volume little bottle	50	mL
Temperature	-15	°C
cf (T)	19.3 %	

Calculated volume [mL] solute in small bottles	**c**	**Fb**	**Real volume of solute in small bottle**	**Real total volume of solution in small bottle**	**No. Substrate**	**Temperature**	**Result**
22.50	4.50 %	0.23	22.51	50.01	5	-15.21	NF
23.75	4.75 %	0.25	23.76	50.01	7	-15.37	F/PF
25.00	5.00 %	0.26	25.02	50.05	2	-15.39	F
26.25	5.25 %	0.27	26.26	50.02	6	-15.41	F
27.50	5.50 %	0.28	27.52	50.10	1	-15.41	F
28.75	5.75 %	0.30	28.75	50.02	8	-15.48	F
30.00	6.00 %	0.31	30.01	50.01	4	-15.55	F
31.25	6.25 %	0.32	31.26	50.01	3	-15.23	PF

Concentration brine big bottle	10 %	
Volume little bottle	50	mL
Temperature	-17.5	°C
cf (T)	21.5 %	

Calculated volume [mL] solute in small bottles	**c**	**Fb**	**Real volume of solute in small bottle**	**Real total volume of solution in small bottle**	**No. Substrate**	**Temperature**	**Result**
20.00	4.00 %	0.19	20.00	50.01	7	-17.99	NF
22.50	4.50 %	0.21	22.51	50.01	3	-17.90	NF
25.00	5.00 %	0.23	25.02	50.05	5	-17.86	NF
27.50	5.50 %	0.26	27.52	50.10	4	-18.19	NF/PF
30.00	6.00 %	0.28	30.01	50.01	8	-18.10	NF
32.50	6.50 %	0.30	32.51	50.02	1	-18.08	NF
35.00	7.00 %	0.33	35.02	50.02	6	-18.16	F/PF
37.50	7.50 %	0.35	37.54	50.04	2	-18.09	F

Concentration brine big bottle	10 %	
Volume little bottle	50	mL
Temperature	-17.5	°C
cf (T)	21.5 %	

Calculated volume [mL] solute in small bottles	c	Fb	Real volume of solute in small bottle	Real total volume of solution in small bottle	No. Substrate	Temperature	Result
30.00	6.00 %	0.28	30.01	50.01	7	-17.80	NF
31.25	6.25 %	0.29	31.26	50.01	8	-17.88	NF
32.50	6.50 %	0.30	32.51	50.02	4	-17.94	NF
33.75	6.75 %	0.31	33.76	50.03	2	-17.92	F
35.00	7.00 %	0.33	35.02	50.02	5	-17.67	F
36.25	7.25 %	0.34	36.27	50.00	1	-17.85	PF
37.50	7.50 %	0.35	37.54	50.04	3	-17.72	F
38.75	7.75 %	0.36	38.76	50.02	6	-17.94	F

Concentration brine big bottle	10 %	
Volume little bottle	50	mL
Temperature	-17.5	°C
cf (T)	21.5 %	

Calculated volume [mL] solute in small bottles	c	Fb	Real volume of solute in small bottle	Real total volume of solution in small bottle	No. Substrate	Temperature	Result
28.75	5.75 %	0.27	28.75	50.02	1	-17.85	NF
30.00	6.00 %	0.28	30.01	50.01	3	-17.70	NF
31.25	6.25 %	0.29	31.26	50.01	4	-17.95	NF
32.50	6.50 %	0.30	32.51	50.02	2	-17.84	F
33.75	6.75 %	0.31	33.76	50.03	6	-17.88	PF
35.00	7.00 %	0.33	35.02	50.02	7	-17.81	F
36.25	7.25 %	0.34	36.27	50.00	8	-17.91	F
37.50	7.50 %	0.35	37.54	50.04	5	-17.66	F

Concentration brine big bottle	10 %	
Volume little bottle	50	mL
Temperature	-20	°C
cf (T)	23.6 %	

Calculated volume [mL] solute in small bottles	**c**	**Fb**	**Real volume of solute in small bottle**	**Real total volume of solution in small bottle**	**No. Substrate**	**Temperature**	**Result**
22.50	4.50 %	0.19	22.51	50.01	8	-20.56	NF
25.00	5.00 %	0.21	25.02	50.05	3	-20.42	NF
27.50	5.50 %	0.23	27.52	50.10	7	-20.46	NF
30.00	6.00 %	0.25	30.01	50.01	6	-20.46	PF
32.50	6.50 %	0.28	32.51	50.02	5	-20.38	PF
35.00	7.00 %	0.30	35.02	50.02	2	-20.45	F
37.50	7.50 %	0.32	37.54	50.04	1	-20.48	NF
40.00	8.00 %	0.34	40.01	50.01	4	-20.65	F

Concentration brine big bottle	10 %	
Volume little bottle	50	mL
Temperature	-20	°C
cf (T)	23.6 %	

Calculated volume [mL] solute in small bottles	**c**	**Fb**	**Real volume of solute in small bottle**	**Real total volume of solution in small bottle**	**No. Substrate**	**Temperature**	**Result**
22.50	4.50 %	0.19	22.51	50.01	4	-20.51	NF
25.00	5.00 %	0.21	25.02	50.05	6	-20.46	NF
27.50	5.50 %	0.23	27.52	50.10	3	-20.30	NF
30.00	6.00 %	0.25	30.01	50.01	2	-20.47	NF
32.50	6.50 %	0.28	32.51	50.02	5	-20.32	NF
35.00	7.00 %	0.30	35.02	50.02	8	-20.45	F/PF
37.50	7.50 %	0.32	37.54	50.04	7	-20.40	F
40.00	8.00 %	0.34	40.01	50.01	1	-20.53	F

Concentration brine big bottle	10 %	
Volume little bottle	50	mL
Temperature	-20	°C
cf (T)	23.6 %	

Calculated volume [mL] solute in small bottles	c	Fb	Real volume of solute in small bottle	Real total volume of solution in small bottle	No. Substrate	Temperature	Result
30.00	6.00 %	0.25	30.01	50.01	1	-20.50	NF
31.25	6.25 %	0.27	31.26	50.01	2	-20.38	F
32.50	6.50 %	0.28	32.51	50.02	3	-20.27	NF
33.75	6.75 %	0.29	35.02	50.02	4	-20.56	NF
35.00	7.00 %	0.30	36.27	50.00	5	-20.29	NF
36.25	7.25 %	0.31	37.54	50.04	6	-20.54	PF
37.50	7.50 %	0.32	38.76	50.02	7	-20.67	F
38.75	7.75 %	0.33	40.01	50.01	8	-20.50	F