NEW TEST PROCEDURES FOR SOLID AND LIQUID DEICER

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Topic 5. Operational approaches, equipment and products for winter service

ABSTRACT:

On roads with medium to high traffic volume abrasive gritting materials do often not perform as well as chemical deicers due to a faster material loss in the wheel tracks. Furthermore a passing of vehicles at high speeds causes damages to following vehicles making chemical deicers the weapon of choice in winter maintenance. Due to its good cost-benefit ratio sodium chloride from mainly three origins namely vacuum salt, rock salt or sea salt is used as the major deicing material. While pure sodium chloride behaves the same from all sources, the practical delivered salt from these sources show variations of considerable impact regarding grain size, purity, sulphate concentration, humidity among others. For winter maintenance thawing capacity and thawing speed are the most important parameters together with the resulting costs in the selection of deicers. With other deicers on the market as well it is necessary to provide universal testing procedures which cover the entire product range. With the use of a special designed climate chamber allowing manual testing procedures inside without opening the chamber door, a series of new test methods meeting these requirements have been developed. As a result time- and temperature - dependent thawing capacity curves have been conducted for all common solid and liquid deicers. Also freezing point curves have been validated for typical mixtures of deicers used in Austria. The paper gives an overview of these new testing procedures allowing a determination of all necessary de-icing parameters with moderate effort. Furthermore the results of these test are given as well allowing a comparison of already existing products regarding performance and costs with any new de-icing product which may appear on the market in the future.

KEY WORDS: deicer, laboratory, thawing capacity, freezing curves, cost-benefit

1. INTRODUCTION

The use of abrasives winter maintenance is mostly restricted to urban areas or roads with low AADT. As problems with abrasives like particulate matter and regaining costs grow road authorities turn more and more to de-icing chemicals. To provide a good quality of the used chemicals according to the Austrian regulations a product must fulfil following requirements:

- High thawing capacity (includes sufficient rate and duration of thawing)
- Low environmental impact (Fauna, flora must not be damaged)
- Low impact on surrounding infrastructure (Road infrastructure e.g. bridges must not be damaged)
- Economic use (Costs for material must be too high)
- Ensure friction (must not lower friction to dangerous levels)

These qualitative requirements have no quantitative description in the current regulations which makes it almost impossible to include new de-icing chemicals if needed. Thus the common practise is to test new products in situ which imply very subjective judgement on the criteria's mentioned above. Above these qualitative criteria's there are several specific requirements listed in the Austrian regulations such as:

Free flowing capability, which describes the ability of dry chemicals to remain as separate grains rather than clumping together. Bigger lumps of chemicals can't be spread properly and may even damage spreading machines. In order to avoid such problems usually anti caking agents are distributed over the de-icing chemicals. These anti caking agents have to be certificated for the use in the European Union. As there is no standardised testing method common in Europe, the procedure is rather subjective by testing it by hand.

The chemical compounds of de-icing agents are regulated for three chemicals only. The chemical purity of Sodium chloride for example must not be below 97.5%, Calcium chloride not below 77% and magnesium chloride not below 47%. All chemicals must not contain more than 10g/kg sulphate to avoid damage of concrete structures.

The grain size of dry chemicals affects the quality of distribution both in dry or pre-wetted treatments. Thawing rate is also affected as shown in this paper. Spreading machines are adjusted for a special grain size, other than that the output will not be consistent. To small grains are blown away by wind too easily and to big grains tend to roll over the road surface into the surrounding areas. In Austria the grain size must be between 0.125 mm and 3.15 mm with 5% below or above that limit and all grains below 5 mm.

Heavy metal concentrations must be within the given limits according to the regulations.

Too high humidity might lead to clumping even with added anti caking agents. Therefore the humidity of salt used for road winter maintenance in Austria must be below 0.5%.

If a product matches the specifications it might be used for winter maintenance in Austria. However the physical property most important for winter maintenance, thawing behaviour, is still not specified. As testing methods are not specified as well this paper will present new testing methods and possible limits in order to define the missing specifications.

2. CURRENTLY USED TESTS

The only significant testing method for thawing capacity in Europe is the Inzeller Eisplattenversuch developed at the Federal Highway Research Institute (Bundesanstalt für Straßenwesen) in Germany [1]. To determine the thawing capacity 4 g of the de-icing agent are placed on a plate of ice 3.5cm thick at temperature of -2° C and -10° C for 10 and minutes. As there are 6 ice plates (28x18 cm) required for each combination at least 24 plates have to be tested over all. As a comparable result the thawing capacity is calculated as gram molten ice per gram deicing agent. Main problem with this method is the need of huge machinery and therefore a climate chamber to perform the tests.

In the US chemical deicers are tested using the method described in the "Handbook of Test Methods for Evaluating of Chemical Deicers" SHRP-H-332 [2]. The thawing capacity is tested according to SHRP-H-205.1 for dry chemicals and SHRP-H-205.2 for liquid deicers. Similar to the Innzeller Eisplattenversuch, the deicing chemicals are placed on a defined plate of ice. The weight of the ice plate is measured over the testing period several times. The brine emerged from the molten ice and chemicals will be removed from the plate using a syringe. After weighing the brine is put back on the plate. The testing period is 60 minutes in order to achieve significant results. Problem with this test method is the uncertainty rising from the repeated use of ice plates after removing the brine once.

Another test is the ice penetration (SHRP-H-205.3 und SHRP-H-205.4) analysing the thawing performance on cylindrical ice samples with either dry or fluid chemicals. The sample cylinders are produced using a transparent block of plastic with 35 mm deep holes next to each other. The deicing chemical is put on top of the ice filled holes and melts it way down the ice cylinder. The penetration depth is recorded every 5 to 15 minutes of a 60 minute period. However tests showed the immense impact of grain size on the penetration ability of deicers. Thus it is hard to get reproducible results from this testing method if different manufactures are used with only slightly different grain sizes and forms. Also penetration depth and sample weight of de-icing samples do not correspond linear. Due to the grain size affection this test method rather tests grain size than chemical de-icing performance.

Over all the common tests methods are well developed but have slight drawbacks each. The Inzeller Eisplattenversuch needs expensive equipment and the fact, that the de-icing agent is put only on a spot, not the whole ice plate is disturbing. The SHRP method with repeated use of one ice plate for several tests with removing and regaining the brine brings massive elements of uncertainty.

Thus new methods have been developed at the Vienna University of Technology which deliver reproducible results with low investments in laboratory facilities and equipment. The new test should also be able to measure the pure de-icing performance from either solid or liquid chemicals.

3. NEW TEST PROCEDURES

The complete testing procedure was done inside a climate chamber using access openings sealed with gloves in order to work inside the chamber without opening the door. Thus it was possible to maintain constant temperature and humidity during the tests. Using a climate chamber with such modifications the need for an expensive and bulky climate room can be anticipated. Also the climate chamber can replace other machines used for example when testing soundness of materials.

3.1. Test methods for solid deicers

The principle idea of other testing methods where deicers are placed on ice and thaw brine is measured remains the same in this test procedure. Materials needed for this test are apart from the climate chamber cheap and easy to get. The test setup with the needed equipment is shown in Fig. 1 with the following items:

- 1. Petri dishes with 20cm diameter and 3 cm height
- 2. Electronic balance with working temperature range down to -10°C and 0,05g precision
- 3. Metal Sive 20cm diameter (hole diameter according to grain size)
- 4. Test protocol
- 5. Container for used samples



Figure 1: Tools required for the test procedure on thawing capacity of solid deicers

The main focus of tested deicers was sodium chloride from several vendors with different grain size and humidity. From rock salt with a wider grain size range samples with restricted grain size have been tested separately. Grain size over 3 mm has not been tested due to the limit of 3.15 mm in Austrian regulations.

To make the ice samples the dishes have been filled with around 0.5I water and put in the climate chamber at the predefined test temperature. At the same time all other materials and tools are placed inside the chamber in order to keep the door closed until the end of the test procedure. Thus it is possible to maintain the exact temperature defined. To guarantee sample temperature at defined level the samples have to be given at least 12 hours to completely freeze to solid ice.

During the test development the door has been opened on purpose for a few seconds. Immediately the warm moved into the chamber and affected the thawing capacity considerably.

With the freezing process done and the ice sample temperature at the defined test temperature the actual test procedure starts with distributing 10g of deicer evenly over the sample using the sieve. In order to apply the defined amount of deicer the sample remains on the electronic balance while the sieve is carefully shaken until the 10g are reached. The deviation allowed is defined with $\pm 1g$. Samples with deicer amounts outside this range will not be considered in the analysis. With some experience it is possible to achieve a

deviation of less than 0.5g or even less with salt of almost the same grain size (e.g. vacuum salt). In the analysis the different amount of de-icing chemicals is taken into account as the thawing capacity is normalized per Gramm deicer.

Similar to other test procedures the weight of the molten ice is measured after defined time spans. During development of the procedure time spans of 2, 5, 10, 20, 30, 60, 120 and 240 minutes have been set delivering adequate information at the thawing process. At the end of each time span the weight of the brine generated by thawing the ice on the particular sample is measured as well as the weight of the remaining ice sample. The difference between the weight of an ice sample (including dish) before and after the thawing should be the same as the weight of the brine. This system of double measuring allows removing measures failed to whatever reasons.

To avoid the imprecision resulting from the multiple use of ice samples and brine as done in the SHRP each sample is only measured once. One test series requires 8 samples if the time spans mentioned above are used. With some experience and a good preparation of the climate chamber it is possible to work with 24 samples which allows 3 measurements for each time span. Thus a complete test series can be done in one day respectively 1.5 days if preparation and post processing is taken into account as well.

3.2. Resulting thawing capacity for solid deicers

Results from the new developed test procedure are shown in Fig. 2 for salt with grain size of 2 mm to 3.15 mm and temperature of -2.5°C, -5.0°C and -7.5°C. The figure contains the single measurements as point markers and calculated trends. The anticipated thawing capacity at different temperatures can be seen very clearly. Especially at low temperatures the rate of ice melted by deicers decreases fast within the first 30 minutes. At temperatures of -7.5°C after 30 minutes around 80% of the total thawing capacity measured after 240 minutes is reached.

As information relevant for the practical usage of sodium chloride the thawing capacity determines the application rate in order maintain safe roads. At temperatures around - 2.5°C the thawing capacity still rises after 3 hours. Thus it is possible to use low application rates if the treatment intervals are around 3-4 hours. On the other hand treatment intervals below 30 minutes seem inappropriate due to the fact that at temperatures only a few degrees below zero only 50 to 60 % of the possible thawing capacity is reached.

In Fig. 3 the thawing capacity of different grain size at the same temperature is shown as measured values and calculated trends. One can see that the smaller grain size leads to a higher thawing rate at the beginning of the test. At the end both grain sizes produce almost the same thawing capacity. The reason for this will be explained in a separate chapter later. Also included in this figure is the thawing capacity quotient which describes both, the initial thawing rate and the further development of thawing capacity. As break point of the trend the 10 minute value was chosen.

By comparing Fig. 2 and Fig. 3 it becomes clear that the temperature is the dominating factor determining the thawing capacity of one chemical deicer. To achieve the thawing capacity of fine grains (0.5 mm) with 2mm grains the temperature has to be less than 0.5°C higher. However using these results for sodium chloride as reference new deicer can be tested and compared with common materials in a few days.



Figure 2: Thawing capacity measurements from laboratory tests and calculated trends for sodium chloride with grain size 2 - 3.15 mm at temperatures of -2.5° C, -5.0° C and -7.5° C



exposure time [hh:mm]

Figure 3: Thawing capacity measurements from laboratory tests and calculated trends for sodium chloride with grain size 0.5mm and 2 mm at temperatures of -7.5°C including proposed thawing capacity quotients

The function used to describe the development of the thawing capacity is the power function as seen in Equation 1. Parameter *a* is kind of a scale factor which in this equation describes the overall thawing capacity. Parameter *b* describes the initial thawing rate and further development of the thawing capacity.

$TC = a * x^b$	Equation 1: Development of thawing capacity (Paramter a and b für NaCl according to Tab. 1)			
	TCThawing capacity (ggetaute Sole/gTaumittel)a, bParametersxTime in minutes			

Typical range of the parameter b in tests for sodium chloride is between 0.2 and 0.6 with the value 1 describing a linear function. The lower the parameter *b*, the higher the difference between the initial thawing rate and their further trend. Very low values of b could probably been found when using chemicals with exothermic thawing reactions. Calculated values for these parameters can be found in Table 1.

	grain size 0.5 mm			grain size 2.0 mm – 3.15 mm		
	-2.5°C	-5.0°C	-7.5°C	-2.5°C	-5.0°C	-7.5°C
а	1.517	0.5216	1.452	0.7961	0.3606	0.5576
b	0.2692	0.4781	0.2317	0.4796	0.5697	0.3798

Table 1: Parameter *a* and *b* derived from measurements with sodium chloride

Another way of describing the thawing capacity is to use the quotient as shown in Fig. 3 separating the initial thawing rate and further development as two linear functions. The quotient is calculated using Equation 2.

$TO = \frac{a * x_{t2}^b - a * x_{t1}^b}{a * x_{t1}^b}$	T Q Equation 2: Calculation of thawing quotient		
$x_{t2} - x_{t1}$	T TO	thewing quotient []	
	a h	Parameters [-]	
	2, D X.	starting time of quotient building [min]	
	X _{t2}	end time of quotient building [min]	

Keeping in mind the development of the thawing capacity and also the practical use of deicers it is advisable to split the trend into two parts. The first quotient is calculated from the start of the test procedure to the 10 minute value and the second one from 10 minute to the last measurement at 240 minute value. Initial (0-10 minutes) and Ending (11-240 minutes) thawing quotients calculated in this way are shown in Fig. 4 for sodium chloride with grain size of 0.5mm or 2.0mm and various temperatures.

Figure 4: Initial (TQ1) and Ending (TQ2) thawing quotient for sodium chloride with 0.5 and 2.0 mm grain size and temperatures of -2.5°C, -5.0°C and -7.5°C

It turns out that TQ1 is lower for larger grain size when temperatures drop below -5°C making fine grain size chemicals more efficient at lower temperatures. This correlation is for TQ2 the other way round with coarse grains above fine grains. With the background that the same amount of the same chemical must thaw the same amount of ice under the

same conditions this switch of quotients becomes apprehensible. Larger grains only need more time to melt the same amount of ice compared to fine grain deicers.

3.3. Impact of grain size on thawing

The differences in thawing behavior of different grain sizes measured in these tests are a result of the slightly different thawing mechanisms. Based on an application rate of 10 g/m² and typical grain sizes of salt in Austria with 0.5mm for fine and 2.8mm for coarse salt the amount of grains on 1m² is 70,000 respectively 450. For better imagination Fig. 5 shows the amount of grains in roughly 1:1 scale. Single grains are marked as red dots with their area of influence shown as gray shaded circle. The mentioned difference between grain sizes in the initial thawing rate is a result of the huge difference in the available surface of de-icing chemicals. While 0.5mm grains have 55,000 mm² surface as contact area to ice 2.8 mm grains only have 11,000 mm² [3].

All grains will thaw a cone on top of the ice with a radius depending on the grain size, thawing capacity and temperature. Even though the overall mechanism is the same if all grains are considered the effect is different. While larger grains are separated and melt separated vertical tunnels the finer grains build one layer of brine on top of the ice melting the whole area at once.

Figure 5: Different thawing mechanisms of fine and coarse salt

3.4. Test methods for liquid deicers

Considering the fact that with prewetted spreading already 30% of the applied material is brine and the number of spreaders applying brine only is growing the test procedure was adapted for liquid deicers. The test also allows analyzing the use of various additives used to improve the behavior of liquid deicers. Winter maintenance drivers report problems with salt agglutinating on the distributing plate at temperatures below -7°C raising the question of the optimum brine concentration. Using the following test procedure it's possible to determine the concentration required for a safe and reliable application of brine.

Test procedures and required materials and tools are mostly the same as for solid deicers. Only the ice sample changes its size, shape and making. Also the sieve will be replaced by syringes with 10 ml volume. During the freezing process special dishes are used to produce a defined small basin in the ice sample. The dimensions of the sample are shown in Fig. 6 with an area of 3,320 mm² available as contact area between ice and deicer. In order to maintain evenly distributed thawing activity there are rounded edges instead of a sharp corner between basin wall and floor. While the samples for solid deicers have to be weight in order to determine the applied amount of deicer this procedure can be skipped with liquid deicers as the amount is exactly 10 ml using a syringe. The preparation of the ice samples require an opening of the climate chamber since the dishes forming the basin can't be removed using the gloves. Therefore it is necessary to remove the dishes and wait at least 2h until the temperature of all materials inside the climate chamber has regained the defined test temperature.

Figure 6: Dimensions of the ice sample used to test liquid de-icers and picture of used samples with visible traces of deicers.

The testing procedure itself consists of applying the deicers, weigh the molten ice, weigh the remaining ice, calculate the ice thaw by the deicer. Apart from the distribution method and one weighing process less it is the same test as for solid deicers. Thus they are comparable if it is to determine the thawing capacity of different application methods such as dry or prewetted salt application as well as pure brine application.

Figure 7: Process of testing liquid deicers in pictures with weighing, application of deicer using a syringe, exposure time, another weighing

After defining the test procedure and gaining of experience with handling and analysis the following liquid deicers have been tested. An interesting experience was the test of deicers around their limit of thawing capacity. At very low temperatures combined with low concentrations it occurred that less brine was drained then applied. In these cases the brine froze and built ice rather than thawing the ice sample.

- Sodium chloride brine with 10%, 16% and 20% concentration
- Calcium chloride with 23% concentration 23%
- Molasses product (10% molasses, 90% NaCl-brine with 23% concentration)
- Supermix (70% water, 12% NaCl, 12% molasses, 6% CaCl₂)

With the data gathered from these liquid deicers the optimum test temperature needed some discussion. In order to compare different deicers it seems a good idea to use a reference chemical. However due to the large temperature range chemical deicers can work it is hard to find a constant reference. By limiting the temperature to -2.5°C to -10.0°C it is possible to use sodium chloride as reference with the thawing capacity shown in Fig. 8. The function of other deicers looked pretty similar to the functions of solid deicers in this test also the power function was fitted to the measured points.

If tests at temperatures below -10.0°C are necessary the reference might be calcium chloride as it is a common deicer as well. In terms of practical usage this low temperatures do not matter very much since most snowfall events happen at temperatures between +1°C and -2°C in Austria at heights up to 300m above sea level [4]. With rising elevation the temperature range drops but is still around +1.0°C to -5.0°C at altitudes between 300 and 600m. As a result of this meteorological aspect and the fact that below -5°C some deicers in medium concentrations reach their limits the general optimum test temperature can be advised with -5.0°C. Also the difference in thawing capacity between -2.5°C and - 5.0°C on one hand and -5.0°C and -10.0°C on the other is almost the same while the temperature difference is 2.5K respectively 5.0K. As the temperature is the dominating factor on thawing capacity a comparison at low temperatures (-10.0°C) might not reveal much difference while tests at higher temperatures will.

The impact of brine concentration on the thawing capacity is shown in Fig. 9 for sodium chloride brine with 10%, 16% and 20% concentration at a test temperature of -2.5°C. The differences in thawing capacity can be distinguished with values of 1.40 g with 20% concentration, 0.88 g with 16% concentration and 0.33 g with 10% brine concentration ice thaw per gram de-icing chemical (gram brine) after 180 minutes exposure time. During the winter maintenance season it is difficult to change the brine concentration according to the temperature due to huge storage capacities and fast changing air temperatures. A constant sodium chloride brine concentration of 20% is recommended for general use.

The results of 4 different liquid deicers at a test temperature of -5.0°C are shown in Fig. 10 as calculated trend based on extensive measurements with the equations displayed in the Figure. Compared to sodium chloride brine with 20% concentration the brine with additional molasses and the Supermix show a 19% higher thawing capacity after 180 minutes. Calcium chloride has compared to sodium chloride 27% more thawing capacity after 180 minutes. While the thawing capacity is of calcium chloride is 27% higher the price compared to sodium chloride is 200% to 300% higher. Furthermore the fraction of brine with prewetted salting is only 30% resulting in a minor impact on snow or ice compared to the 70% dry deicer.

Figure 8: Thawing capacity measured and calculated of sodium chloride brine at temperature of -2.5°C, -5.0°C and -10.0°C.

Figure 9: Thawing capacity measured and calculated for sodium chloride with concentration of 10%, 16% and 20% at -2.5 °C

Figure 10: Thawing capacity of 4 different deicers at -5.0°C as trend calculated with the equations based on measurements and shown in the figure

4. RESULTS OF CONDUCTED TESTS

On several thawing capacity tests of deicers the developed procedure delivered plausible and reproducible results. In general thawing capacity and freezing point do not correlate linear. Therefore it is necessary to test deicers for both parameters separately.

In general the tests with liquid deicers show a better reproducibility than comparable test with solid chemicals. The reason might be the exact constant amount of deicer used in the test for liquids. Another reason is the impact of different grain sizes on the thawing capacity of solid deicer. Even with the use of a sieve there is still a grain size range left. A possible solution to this problem is a finer limitation of the grain size using sieves with only small differences in whole width.

4.1. Solid deicer

For the tested solid deicers the following results can be pointed out:

- Thawing capacity has its peak in the first 15 minutes of exposure time and flattens to a steady value depending on temperature and deicers
- After 120 minutes around 90% to 95% of theoretical thawing capacity are reached
- The temperature impact on thawing capacity dominates which requires exact constant test temperature
- Fine grained deicer usually profit from the higher contact area available especially in the first minutes of exposure
- The gradient of thawing capacity is related to grain size

This observations lead to following practical conclusions on solid deicers

- Due to the better distribution on the surface and the higher specific surface fine salt has a faster and more evenly effect on extensive yet thin ice layers like hoarfrost.
- Coarse salt is more capable of undercutting an established ice or snow layer.

4.2. Liquid deicer

For the tested liquid deicers the following results can be pointed out:

- At temperature below -8°C it is possible to use calcium chloride brine or add molasses to sodium chloride brine to achieve a better thawing capacity
- However due to the high price it is more economical to use more sodium chloride
- Besides the almost identical freezing point of a 23% calcium chloride brine and a 23% sodium chloride brine the thawing capacity of CaCl₂ is 27% higher
- The full potential of high thawing capacities can be used only if temperatures are very low (below -5.0°C)

This observations lead to following practical conclusions on solid deicers

- A general test temperature of -5.0°C is advised as standard
- New deicers have to be checked for the freezing point before conducting thawing capacity tests and the test temperature has to be chosen accordingly
- Sodium chloride can be used as a reference deicer in order to quantify the thawing capacity of new chemicals in a test temperature range of -2.5°C to -10°C

5. CONCLUSIONS

The developed test procedures allow more precise and reproducible data on the thawing capacity of chemical deicers in both liquid and solid state. The results show a strong related with practical experience and enable the shortcut from laboratory experiments to practical usage. Thus the application of chemicals can be optimized according to weather conditions. Also the quality of delivered deicers can be tested with the developed process.

Compared to the existing methods this new approach requires only a few laboratory facilities. There is a one-time charge for the climate chamber which is rather cheap compared to the installation of an entire climate room. All other materials used are quit common and available for very small amounts of money.

With the use of a modified climate chamber and the installed gloves there was no disruption in the test temperature for the time of the procedure. The chosen exposure times reproduce the practical effect of salt on the road with an high thawing rate at the beginning and further flattening. With the maximum exposure time of 4 hours it is possible to conduct the tests during one working day. The practical background of the 4 hour limit is the Austrian regulation on treatment intervals which have a minimum requirement of 3 respectively 5 hours in highways respectively state roads.

Potential for further optimisation of the proposed tests for solid deicers can be found in the application of the chemicals on the ice sample. Even with experience it is not possible to achieve an exact dosage. If on the other hand the amount of deicer is prepared in exact dosages it is not possible to distribute it evenly. As the results are normalized the way of keeping the quantity variable in favour for a very even distribution was chosen with acceptable results.

The potential of such distributing optimization becomes clear when the coefficient of determination from solid and liquid deicers are compared. While tests with solid deicers achieve an R² of around 0.79 to 0.92 liquid deicers come up with R² from 0.87 to 0.97.

Not reproducible with this laboratory test is the impact of traffic over time on the thawing capacity. The mechanism behind the discharge of deicers from the road is described in [3,4] and is not relevant in the determination of thawing capacity. However the combination of these two factors along with several others is considered in a holistic winter maintenance model developed at the Vienna University of Technology [5,7,8].

Nevertheless, the results already achieved using this test method allowed an optimization of prewetted salting in Austria [3,6]. Also the results of these tests are more orientated on practical use of deicers than common methods.

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