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Effect of anti-caking agents on flowability of de-icing salts

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Kurzfassung

Winterdienst ist in Österreich sehr wichtig, da an mehr als 100 Tagen die Straßen frei von Eis und Schnee gehalten werden müssen, um sichere Fahrbedingungen zu gewährleisten. Die Gesellschaft ist von ihr stark betroffen, da es um die Mobilität der Menschen und der Fracht während der Winterzeit geht. So wird auch die gesamte Wirtschaft beeinflusst. Unter der Ansicht der Straßenerhaltung repräsentiert sie 20 bis 40% des Gesamtbudgets. Allerdings haben Auftausalze negative Auswirkungen auf die Flora und Fauna im Bereich von Straßen und gelangen durch Versickerung auch ins Grundwasser. Aus alle diesen Gründen ist es wichtig, den Einsatz von Auftaumittel zu optimieren, um die wirtschaftlichen und ökologischen Auswirkungen zu verringern.

Das meist verwendete Auftausalz in Österreich ist Natriumchlorid, dessen Verwendung wird in der RVS 12.04.12 und seine Bestandteile in der RVS 12.04.16 geregelt. Die Rieselfähigkeit von Auftausalz wird durch die Zugabe von Antibackmitteln und durch eine maximale Feuchtigkeit von 0,5% während Transport und Lagerung gewährleistet, damit das Salz nicht verklumpt.

Die Verklumpung von Salz kann mehrere Konsequenzen haben, die zu einer Verzögerung oder Beeinträchtigung der Ausbreitung führen. Dies kann dazu führen, dass die Streustrategie fehlschlägt und die Kosten erhöht werden.

Derzeit gibt es keinen spezifischen Test für die Messung der Rieselfähigkeit. Mit der "Auslaufbox nach Sonntag" steht ein Messapparat zur Verfügung, um die Rieselfähigkeit von Schüttgütern, die in der Regel in Silos gelagert werde, zu bestimmen.

Ziel dieser Arbeit ist es, den Einfluss von unterschiedlichen Antibackmittelgehalten und Feuchtigkeitsniveaus auf die Rieselfähigkeit mit der Auslaufbox nach Sonntag zu bewerten.

Die Ergebnisse zeigen eine direkte Beziehung zwischen der Menge an Antibackmittel, Feuchtigkeit des Salzes und Auslaufrate von Auslaufbox, was die Nützlichkeit des Tests zur Messung der Rieselfähigkeit bestätigen würde.

Abstract

Winter road maintenance is of great importance in Austria. Its operations last for more than 100 days a year and have the aim of keeping the roads clear of ice and snow and in safe drivable conditions. Society is deeply affected by it, since on it depends road mobility of people and freight during the winter period. Thus, affecting the whole country economy as well. From the point of view of road maintenance, it represents from 20 to 40% of its total budget. Environmentally, de-icing agents can have negative impacts on the flora and fauna surrounding roads, besides ground water through filtering of the runoff mixed with de-icing agents. For all these reasons, it is important to optimize the use of de-icing agents and road maintenance operations through all stages, in order to reduce its economic and environmental impact.

The mostly used de-icing salt is sodium chloride, being its use regulated in Austria by RVS 12.04.12 and its components in RVS 12.04.16. Flowability of de-icing salt is guaranteed through the addition of anti-caking agents and by keeping a maximum humidity of 0,5% throughout transportation and storage. If this does not happen, and exceeds this humidity level, salt can form lumps, making inefficient and delaying the spreading of de-icing salts.

The formation of lumps in salt can have several consequences resulting in delaying or hindering the spreading of de-icing salt. This can cause the winter maintenance strategy to fail, and may increases its costs.

Currently the is no particular test for the flowability of de-icing salt. The “Auslaufbox nach Sonntag” is an apparatus designed to test bulk goods that are usually stored in silos.

The aim of this thesis is to test the influence of different salt samples containing different anticaking agent amounts and for several humidity levels on the flowability with the Auslaufbox.

The results obtained show a direct relationship between the amount of anti-caking agent, moisture and outflow ratio, which would confirm the usefulness of the test to measure the de-icing salt flowability.

Resum

El manteniment de carreteres durant el període hivernal és de gran importància a Àustria. Les seves activitats tenen una durada de més de 100 dies l'any i tenen l'objectiu de mantenir les carreteres lliures de gel i neu per garantir una conducció segura. La societat n'està profundament afectada, ja que depèn de la mobilitat viària de persones i la mercaderies durant el període hivernal. D'aquesta manera, afecta també a tota l'economia del país. Des del punt de vista del manteniment de la carretera, representa del 20 al 40% del pressupost total.

Ambientalment, les sals de desgel poden tenir impactes negatius sobre la flora i la fauna que envolten les carreteres, a més de l'aigua subterrània mitjançant el filtrat de l'escolament amb salmorra. Per tots aquests motius, és important optimitzar l'ús de les sals de desgel i les operacions de manteniment de carreteres en totes les etapes, per tal de reduir el seu impacte econòmic i ambiental.

La sal de desgel utilitzada principalment és NaCl, sent el seu ús regulat a Àustria per RVS 12.04.12 i els seus components en RVS 12.04.16. La fluïdesa de la sal de desgel es garanteix a través de l'addició d'agents antiaglomerants i mantenint una humitat màxima del 0,5% durant el transport i l'emmagatzematge. Si això no passa, i se supera aquest nivell d'humitat, la sal pot formar grumolls, complicant l'espargiment de la sal.

La formació de grumolls a la sal pot tenir diverses conseqüències que poden retardar o dificultar l'espargiment de la sal. Això pot fer que l'estratègia de manteniment de les carreteres fracassi i pot augmentar-ne els seus costos.

Actualment, no hi ha cap test en particular per la fluïdesa de la sal de desgel, ja que és una qualitat difícil d'examinar. "Auslaufbox nach Sonntag" és un aparell dissenyat per fer tests a productes a granel que se solen emmagatzemar en sitges, però per als quals les proves estàndards existents no són apropiades.

L'objectiu d'aquesta tesi és provar diferents mostres de sal que contenen diferents quantitats d'agents antiaglomerants per diversos nivells d'humitat i determinar si els resultats obtinguts tenen sentit i podrien ser útils com a prova de fluïdesa per a les sals de desgel.

Als resultats obtinguts es pot observar una relació directa entre la quantitat d'antiaglomerant, humitat i l'outflow ratio que confirmaria la utilitat de l'assaig per mesurar la fluïdesa de la sal de desgel.

Resumen

El mantenimiento de carreteras durante el período invernal es de gran importancia en Austria. Sus actividades tienen una duración de más de 100 días al año y tienen el objetivo de mantener las carreteras libres de hielo y nieve para garantizar una conducción segura. La sociedad está profundamente afectada, ya que depende de la movilidad viaria de personas y mercancías durante el periodo invernal. De este modo, afecta también a toda la economía del país. Desde el punto de vista del mantenimiento de la carretera, representa del 20 al 40% del presupuesto total.

Ambientalmente, las sales de deshielo pueden tener impactos negativos sobre la flora y la fauna que rodean las carreteras, además del agua subterránea mediante el filtrado de la escorrentía con salmuera. Por todos estos motivos, es importante optimizar el uso de las sales de deshielo y las operaciones de mantenimiento de carreteras en todas las etapas, a fin de reducir su impacto económico y ambiental.

La sal de deshielo utilizada principalmente es NaCl, siendo su uso regulado en Austria por RVS 04/12/12 y sus componentes en RVS 12:04:16. La fluidez de la sal de deshielo se garantiza a través de la adición de agentes antiaglomerantes y manteniendo una humedad máxima del 0,5% durante el transporte y el almacenamiento. Si esto no ocurre, y se supera este nivel de humedad, la sal puede formar grumos, complicando el esparcimiento de la sal.

La formación de grumos en la sal puede tener varias consecuencias que pueden retrasar o dificultar el esparcimiento de la sal. Esto puede hacer que la estrategia de mantenimiento de las carreteras fracase y puede aumentar sus costes.

Actualmente, no existe ningún test en particular para la fluidez de la sal de deshielo, ya que es una calidad difícil de examinar. "Auslaufbox nach Sonntag" es un aparato diseñado para hacer tests a productos a granel que se suelen almacenar en silos, pero para los que las pruebas estándares existentes no son apropiadas.

El objetivo de esta tesis es probar diferentes muestras de sal que contienen diferentes cantidades de agentes antiaglomerantes para niveles diversos de humedad y determinar si los resultados obtenidos tienen sentido y podrían ser útiles como prueba de fluidez para las sales de deshielo.

En los resultados obtenidos se puede observar una relación directa entre la cantidad de antiaglomerante, humedad y outflow ratio que confirmaría la utilidad del ensayo para medir la fluidez de la sal de deshielo.

Foreword

I would like to thank everyone that I met in Vienna from the Institute of Transportation and helped me through the writing of this Master Thesis and the tests in the laboratory. Especially thanks also to my tutors from the TU Wien and ETSECCPB.

També voldria donar gràcies als meus pares i a la meva germana. M'han acompanyat durant tota l'etapa universitària que amb aquesta tesi finalitza.

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Frequently used abbreviations

CN: Cyanide

FC: Ferrocyanide $[Fe(CN)_6]^{4-}$

NaCl: Sodium Chloride

RS: Rock salt

SFC: Sodium ferrocyanide

VS: Vacuum salt

YPS: Yellow prussiate of soda

1 Introduction

In countries of cold weather winter service represents a large percentage of the budget for road maintenance. Its economic costs are recompensed by the fact of not having to stop other economic activities that need clear and safe roads. But winter service also has environmental impacts, as the de-icing salts and its additives may runoff, affecting road side vegetation, or infiltrate to groundwater. This means, that both from an economical and environmental point of view it is important to spread the exact necessary amount of de-icing agent to reach the objective of keeping safe drivable roads.

The most commonly used de-icing agent in Austria is sodium chloride (NaCl), because of its low price and proper performance. But its use often implies some other problems also to be considered. These problems are mainly related with the caking of salt during the storage phase that influences the total volume of salt used for winter service and can hinder winter road maintenance.

1.1 What happens during salt storage?

It is necessary, as part of the winter maintenance to have storage facilities for de-icing agents. Salt stocks are essential to have it always available for the winter maintenance activities and it must be ready to be used. This means that salt must be stored in dry conditions, and with the appropriate capacity, so there is no lack of de-icing salt during the winter period. Lacking de-icing agents would imply to close roads to traffic, causing an additional economic and social price.

From the operation point of view, caking in salt during its storage is a problem. It is necessary to store salt during long periods of time, usually months, but it can be even years.

During the storage phase damp can get between salt crystals and dissolve miniscule amounts of the sodium chloride, which encounter other salt particles. As the salt gets dry again, the dissolved sodium chloride forms a structural bridge between salt particles, which results into lumping. This is known as caking [5]. It makes the filling of snowplough trucks and the salt spreading on the road more difficult and can even frustrate salt usage, if salt cannot flow out of the silos.

The current trend is to build road maintenance depots with enough capacity to store the whole amount of de-icing salt needed during the winter period. This means that both silos and warehouses are getting bigger. And so, does the storage period for salt that can be stored for years if the coming winters are mild.

With silos, the problem is that due to the increase in height, the pressures to which salt is subjected are greater. Then, after a few months of the silo being filled, when it is necessary to extract the salt from it, a dome shaped lump inside the silo coinciding with the areas where the stresses are concentrated, prevent the extraction of salt.

This usually happens when the trucks for winter service are about to be filled, and the solution taken is that a worker must hit the “salt dome” with a stick, to break it and let the salt flow out of the silo. This operation is not so convenient, as it delays the salt spreading and is also dangerous for the worker that stands under the silo.

Anticaking agents prevent the formation of lumps, by avoiding crystallization of salt when exposed to humidity. But even though road salt often contains anticaking agent, this problem in silos happens more frequently, due to their increasing size.

Caking in salt during its storage also has its consequences while spreading. If the spreading of salt is done for dry salt, and this salt is lumped, the exactitude of the spreading is diminished. Due to the increase of size, from its normal grain size, to the size of lumps, when centrifuged it is thrown further than expected. This causes the salt not to reach the road surface in the expected quantities and homogeneity. Consequently, it involves an increase on the quantity of de-icing agents used to keep the road clear from slippery conditions. And the surroundings of the road can be reached directly with de-icing salt, affecting environment.

1.2 How can coagulation be avoided?

The formation of lumps can be eluded using additives, called anti-caking agents, which prevent the formation of lumps. These anti-caking agents affect the flowability of the salt in different humidity levels and are supposed to allow its workability even after a storage period that can last from months to years, in which salt is exposed to different humidity levels and wet-dry cycles. Depending if being transported or stored in silos or in a warehouse the water content of salt varies between 0,3 and 0,5% in mass [2].

The most commonly used anticaking agent for de-icing salt is Ferrocyanide $[Fe(CN)_6]^{4-}$ which in its hydrous form is sodium ferrocyanide decahydrate $Na_4Fe(CN)_6 \cdot 10H_2O$ and can also be called hexacyanoferrate or yellow prussiate soda, YPS [7].

FC anti-caking agent acts by inhibiting the crystallization of NaCl when in contact with water, thus preventing the formation of lumps in the salt [8].

This anticaking agent contains cyanide, and tends to accrue in the soils after being washed out of the road by brine, by the direct application of salt that does not reach the road surface, or due to poorly insulated warehouses [9, 7].

FC is also used as an anti-caking agent for table salt and animal feed. This does not mean that the compound cannot release free cyanide. When exposed to sunlight in a dissolved form it can rapidly disintegrate to free cyanide. Being this the main reason of its potential environmental hazard [9]. Further environmental effects of salt application as de-icing are explained in section 1.3.

There are also other kinds of anti-caking agents that are not so commonly used, but that are being introduced as a possible substitute because of their biodegradability and their cyanide-free composition, as the anticaking agent also tested in this Master thesis. This anticaking agent is formed by acetic acid and iron tartrates, derived from by-products of the wine industry [7].

1.3 Environmental effects of de-icing salts

From the environmental point of view there are numerous research papers about the consequences of salt usage to ground water, vegetation, soil, surface waters, etc. surrounding the roads that are treated with salt during winter period.

Known is that de-icing agents, especially salt, NaCl, have some environmental harmful consequences. When chloride de-icers are dissolved in runoff, the anion and cation dissociate. The following section separately describes the environmental effects of de-icing salt based on the affected parties, according on references [10] and class notes from Straßenbetrieb [4].

- **Vegetation:** Road side vegetation can suffer the negative impact of chloride by root absorption. Its effects are similar to those of a drought, having underdeveloped growth, brown and falling leaves and dying limbs. It can lead to even deforestation.
- **Soil:** De-icers reach the soil via run-off, splashing, spraying or ploughing. There can be traces of salts as far as 10 meters from the road edge. Long term accumulation of salts in soil can lead to reduced soil permeability and fertility, along with increased soil alkalinity and density, reducing the ion exchange capacity and the amounts of calcium, magnesium and other nutrients found in soil.
- **Vehicles and Infrastructure**: Salts will initialise and accelerate atmospheric corrosion to metals. Thus, vehicles that drive through salt spread roads and reinforced concrete structures close to the roads can corrode, increasing the economic cost of maintenance.

- **People**: A small percentage of the salt that is applied will remain on the roadway and dry to powder. As the vehicles drives over it, it can be transported by air through fine dust. Apart from people it can also affect neighbouring vegetation, wildlife, soil and water streams.
- **Groundwater:** Chloride does not bind to soils, consequently chlorides can reach groundwater with infiltrating water. Chloride entering groundwater systems is likely to persist for a long time since there is no significance removal mechanism and ground water has slow movement.
- **Surface water:** Chloride concentrations in surface water tend to follow a seasonal distribution. Concentrations usually increase in the winter and decrease in the summer. Salt containing water has a higher density than non-salt containing water and will sink to the bottom of the water body. This can end in chemical stratification and disrupt lake mixing patterns. The adverse effects are directly related to the dilution proportion of de-icing agents, this way, the most impacted by it are small ponds and slow streams, because of lower dilution and dispersion in those locations.

De-icing salt must be transported from production to storage locations, from where it is afterwards distributed. Salt must be kept free of lumps throughout all the process. To achieve it, an anti-caking agent is added to the salt. This is usually sodium ferrocyanide ($Na_4Fe(CN)_6$) and it is also potentially harmful for the environment [7].

The main issue with ferrocyanide, is that even though it is stable and not reactive, when it is in a dissolved form, in contact with sun light it can be fast decomposed to free cyanide. Herein lays the potential environmental hazard of sodium ferrocyanide in its use as anti-caking agent for de-icing salts [9, 7].

There are many natural sources of cyanide, for example Hydrogen cyanide (HCN) is produced and decomposed by some plants, fungi and bacteria, but it is often not a problem, because of their low concentrations. In contrast, this does not happen for anthropologic originated cyanide, that are usually iron complexed, and which accumulates in soil [9]. Aside from de-icing salt, FC can also be found in blue pigments, forest fires control compositions, around gas factories [7].

After ferrocyanide enters the environment it can remain and accumulate for decades, if it does not decompose. In water, it is classified as harmful for aquatic organisms and can cause long term adverse effects [7]. The most common source for cyanide contamination is next to storage places, which may sometimes be not well isolated due to damaged floors, and water and brine could enter the soil. As well next to roadside soils that are periodically salt spread in winter periods. Contamination is only discovered where monitoring of soil and water is carried out [7].

Some studies show that cyanide levels rise significantly next to highways, with levels reaching up to 21 mg CN/kg in soil, after an especially snowy winter, although it did not reach the maximum level admitted by law in Germany for total cyanide of 50 mg CN/kg, it is a considerably higher level than what is considered normal for a natural originated CN level of 1 mg CN/kg [9].

Best practices with de-icing salt help prevent burdening of the environment from cyanide complexes. Such as using the minimum quantity of de-icing agent needed and keeping the salt in good storage conditions. This is also applicable to the contamination of cations and anions released by salts, as well to reduce the economic cost of winter maintenance [9].

There are also other anti-caking agents that are biodegradable and could be a good solution to prevent high concentrations of cyanides in soil along highway, where the application of de-icing salt is intensive during winter periods [7].

1.4 Use of de-icing agents in Europe

The use of de-icing agents is strongly related to the climate of the country. Therefore, each country has its own regulation about thawing salt, or even not particular regulation at all, as in the case of Spain. The points in common are that the mostly used de-icing agent is NaCl, and the most commonly used anticaking agent is sodium ferrocyanide, even though there are remarkable differences in the thresholds. In the following table, it is possible to compare the salt purity, expressed in % of NaCl and anti-caking agent amount, according to the regulations of different countries. See the following Table 1.

Table 1. List of countries and regulations on anti-caking agents in de-icing salt.

Country	Regulations	Salt (NaCl) content	Anti-caking agent (FC ion concentration)
Europe	EN 16811-1:2016 08 - IDT	≥ 97,5%	> 3 mg/kg; <125 mg/kg
Austria	RVS 12.04.16	≥ 97,5%	Rock and sea salt <200 mg/kg Vacuum salt <10mg/kg
Finland	Finish Transport Agency [11]	≥ 97%	≤150 mg/kg
France	NFP 98-180 [12]	Class A ≥ 98% Class B ≥ 91%	<200 mg/kg
Germany	TL-Streu [13]	> 96%	≤200 mg/kg

Country	Regulations	Salt (NaCl) content	Anti-caking agent (FC ion concentration)
Spain[1]	R.D. 1424/1983	-	≤10 g/kg
Sweden	Trafikverket [14]	≥ 97%	≤100 mg/kg
UK	BS 3247 [15]	> 89 %	> 30 mg/kg
USA	ASTM D 632-99 [16]	≥ 95%	≤100 mg/kg

Most of countries determine a purity of NaCl over 90% and a maximum anticaking agent content of around 100 mg/kg. Despite the differences, in European countries, the regulations should adapt soon to the European Standards from 2016 EN 16811-1:2016 08 – IDT, which also sets a minimum amount of anticaking agent in NaCl of 3 mg/kg.

In regulations, it is also limited the heavy metal content, the moisture, the grain curve distribution and the sulphate content of the salt.

About the grain size of the salt, each countries' guidelines are adapted to the salt available in their area. For example, it is stablished in Austrian guidelines a maximum grain size for rock salt of 5 mm [2], while in the United Kingdom up to a grain size of 10 mm for salt is allowed [17].

It is noted that in some standards it is also regulated the use of other substances as de-icing agent to act in the lower temperatures in which NaCl is not sufficient. For instance, in Austria the guidelines stablish the use of NaCl for temperatures up to -10°C, and $MgCl_2$ for up to -15°C and $CaCl_2$ up to minus 20°C [1].

1.5 Salt types depending on origin

Due to the degradation or the production, the salt is divided into three main types: vacuum salt, rock salt and sea salt [18, 17].

Vacuum salt: Pure sodium chloride prepared by the evaporation of brine under vacuum conditions. The brine is usually extracted by controlled solution mining of rock salt beds, but may be naturally occurring. Its grain size is fine.

Rock salt or halite: is a mineral compound with a very high sodium chloride content, which, however, varies depending on the deposit. Sodium chloride mined from natural salt beds must be mechanically degraded. The rock salt deposits originated about 230 million years ago as the result of the evaporation of what were once oceans. It may contain impurities

[1] The data shown in the table for Spain belongs to the law regulating edible salt, there's no regulation regarding winter maintenance.

such as Calcium Sulphate and Silicates. Its grain size is in the order of millimetres.

Marine salt or sea salt: Sea salt, sometimes also called solar salt, is obtained from so-called salt gardens through crystallization. During crystallization, the effects of the sun and wind play a major role. Sea salt also contains, in addition to sodium chloride, small quantities or traces of other substances, including potassium, magnesium and manganese salts.

1.6 How do different anti-caking agents influence the salt flowability?

As it has been explained in previous section 1.1, caking of salt happens when moisture gets between salt particles and dissolves salt crystals creating new crystal bridges between particles when dried [5].

Anticaking agents as FC inhibit crystal growth, avoiding solid bridge formation when the NaCl is exposed to moisture. Thereby, the formation of lumps does not happen and the flowability of the salt can be preserved [8].

There is not a wide research referring to flowability of salt, and there is no specific test intended to measure the flow properties of salt.

1.7 How do anti-caking agents influence thawing?

Anti-caking agents are not supposed to influence thawing capacity of salt. Based on the experience there are other aspects that are more decisive aspects that influence the thawing capacity of salt, as can be if the salt is applied dry or pre-wetted, the temperature of the road surface, the thickness of ice or snow to be thawed, vehicle traffic, the combination of the spreading within a winter maintenance strategy, etc [19].

2 Problem definition

There is not abundant research about the effects of the anticaking agents in the flowability of salt and often salt providers and countries guidelines differ in anti-caking agent content in salt. According to German law TL-Streu the content of anticaking agent must not exceed 200 mg/kg for rock and sea salt and 10mg/kg for Vacuum salt, but other countries as Sweden or the USA determine a 100 mg/kg threshold (See Table 1. List of countries and regulations on anti-caking agents in de-icing salt).

Currently a specific test to measure the flowing capacity of salt does not exist. In the context of winter road maintenance, the flowability of salt is determining and an issue in silo storage and salt spreading. Caking of salt is caused by humidity during salt transportation and storage and is avoided by the addition of anticaking agents. The formation of lumps increases the economic and environmental cost of winter road maintenance. Therefore, it is necessary to find a test which can correlate the anticaking agent and water content in salt to the flowing capacity of salt.

The most used anticaking agent for NaCl is sodium ferrocyanide. Considering the possible negative environmental effects of FC and the fact that if is exposed to sun light can release free cyanide to the environment [9], the study of the flowability performance of salt containing a cyanide-free biodegradable anticaking agent is interesting.

3 Objectives and methodology

The aim of this Mater Thesis is to test the flowability of de-icing road salts of the types vacuum salt and rock salt for containing different anti-caking agent amounts from 0 to 120 mg/kg of ion FC and for humidity levels ranging from 0 to 0,75% in mass, based on previous research on the humidity at which salt is exposed during transportation and storage [2].

The method used to test the flowability capacity of salt is the ‘Auslaufbox nach Sonntag’. The test was developed to test bulk goods for which standardised flowability test were not appropriate for bulk materials due to the broad grain size distribution and coarse grain, as it can be caked salt [3].

The ‘Auslaufbox’ is a cuboid shaped steel box with lateral glass panels. The test consists of filling the box with the bulk material and letting the flap gate open to let the material flow out. With this test two parameters related to flowability are obtained, the outflow ratio, the discharged mass respect the total mass in the box, and the internal friction angle. The test is not meant to be useful for scientific purposes, but for practical uses [3].

4 Winter road maintenance in Austria

Winter road maintenance operations require a large portion of the budget designated for road maintenance. In Austria, there are on average 120 to 150 winter maintenance days per year, this represents a 20% of operating costs on regional roads, and 25 to 30% on Austrian highways [19]. In the Tirol federal state, in 2009, the costs for winter road maintenance represented 41% of the total maintenance costs of roads [4]. This is a matter that countries with milder winters do not have to cope.

In Austria, there is averagely one maintenance depot every 150 km of roads and one worker every 23 km [4]. These infrastructures must serve for the maintenance and safety of roads, and the can be of public or of private domain.

The tasks that these facilities must carry out are standardised by different laws and technical guidelines that determine when, how often, what kind of activity, in what facility and by whom every maintenance operation should be conducted.

Between all the tasks that are necessary to keep the safety on the roads, one of the main ones is the winter road maintenance. The road maintenance depots act as the storage place for de-icing agents, vehicles and as a building for administrative and organisational tasks. This operation requires organisation and foresight for the needs of the winter, since it is not always possible to get de-icing agent salts during winter period.

Winter maintenance means a lot of work and responsibility by the administrations to ensure road safety. The main beneficiary is the user, but also society in general, since the road transportation is an important economic sector. At the same time, it ought to be considered the possible negative effects of operations on the environment.

The affected parties in winter service can be explained through the following Figure 1.

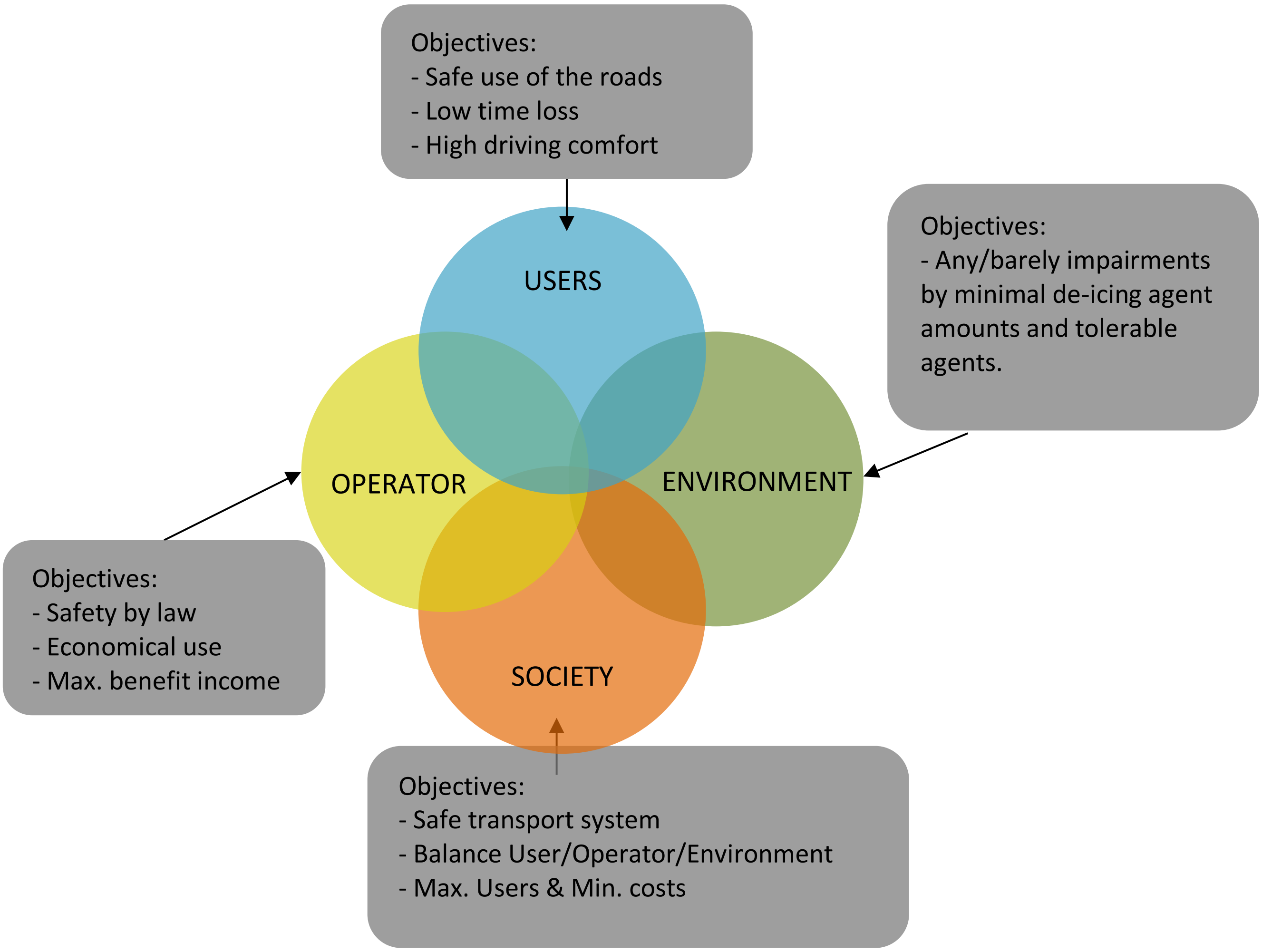


Figure 1. Parties affected by winter road maintenance service [4]

4.1 What is understood by winter road maintenance?

Under the concept winter road maintenance, it is understood all the operations carried out to provide accessibility and safety under winter conditions, that may affect the road conditions with ice and snow.

With temperatures below zero, when it snows or rains, water on the road can freeze and create a film that fills road surface texture and reduces the contact surface between tires and road, reducing grip capacity progressively, which enlarges braking distances and makes controlled driving harder, and if no measures are taken, impedes road accessibility.

The main winter maintenance tasks involve snow ploughing and de-icing agent application. Although it may change from one country to another, in Austria the usual policy is to spread de-icing or gritting agent and plough snow. The aim with these actions is to keep the road grip, and avoid the effects of ice, water and snow. The physical impact of de-icing agent is to lower the freezing temperature of the solution of the agent and the precipitation on the road, below the road surface temperature. To do so the snow must be cleared continually and the de-icing agent losses must be compensated by spreading anew.

The use of de-icing agents has the goal of avoiding, reducing or eliminating icy roads. Frost on the roads can be originated by different reasons:

- Through snow densifying by regular passing cars or people over a snow accumulation on a traffic area.
- Slippery frost originated by the congelation of air moisture on the road surface that is at low temperature.
- Black ice originated by the congelation on a traffic area of pre-existing humidity, e.g. moisture from snowmelt.
- Slippery frost made by sleet, freezing rain, or rain over an over-chilled road surface that creates a homogenous ice surface.

Finally, to keep the safety on the road during winter it is necessary a good winter maintenance service united to an adapted driving to the winter conditions.

4.2 Technical guidelines in Austria

Winter road maintenance techniques are determined in Austria by the technical guideline RVS 12.04.12 (2010) [1], and the characteristics of the agents spread are determined by the guideline RVS 12.04.16 (2011) [2]. These guidelines determine the basics for snow plough and de-icing and grit agent spreading, by defining road categories into which Austrian Federal, country and local roads are divided. As well as determining organizational, clearing equipment, and infrastructure requirements to put into practice all the different winter maintenance activities. Further, it is also defined the characteristics that spreading agents must fulfil to be used on Austrian roads.

4.2.1 Road categories in Austria

In the RVS 12.04.12 (2010) roads are divided either if they belong to the city area or not. Depending if they are federal, country and local roads they will belong to the categories A to D, and for urban areas the categories will range from P1 to P7. The maintenance requirements specified in the winter service road categories (cycle times, care periods, practicability, safety, etc.) set a minimum standard according to each category.

A decisive influencing factor for the allocation of the traffic areas to the individual winter service categories is the traffic volume, average annual daily traffic (AADT) in vehicles / 24h.

In addition to the AADT value, other factors of influence are average daily traffic (ADT) value during the winter period from 1 November to 31 March, truck share, public transportation systems, tourism, outdoor area or urban area, official regulations, etc. They are also included in the allocation of the traffic areas to the individual winter service categories. Hence, the allocation can be made in another category, regardless of the AADT value, in justified cases. The road categories in Austria and their specifications are in the Appendix of the RVS 12.04.12 and summarised in the following Tables.

Table 2. Winter maintenance service categories for roads out of Austrian urban areas

Category	***A***	***B***	***C***	***D***
Description	*Motorways, expressways and their connections*	*Country roads with local traffic importance or with an AADT>5000 vehicles*	*Country roads with an AADT between 1000 and 5000 vehicles*	*Country roads with an AADT<1000 vehicles*
Period of service	*0-24 hr.*	*4-22 hr.*	*5-20 hr.*	*8-20 hr.*
Category	*A*	*B*	*C*	*D*
Response time	*3 hr.*	*Max. 5 hr.*	*Max. 5 hr.*	-
Practicability	*Accessibility of continuous lanes, connecting points and access roads to service areas*	*Humid or dry roadway, icing remains and ruts cannot be excluded*	*Usually snowy roadway*	*Dry roadway, ice remains and ruts not to be excluded, gravel possible*
Spreading material	*Thawing agent – road clearance*	*Preferably thawing/de-icing*	*De-icing or gritting*	*De-icing or gritting*
Impairments	*none*	*Between 22-6h*	*From 20 to 6 hr. and possibly weekends and holidays*	*Severe impairments possible*

Table 3. Winter maintenance service categories in Austrian urban areas

P1	**P2**	**P3**	**P4**
Inner-city main roads, access roads, roads with lines / tram, access to hospitals and fire stations	*Roads with subordinate traffic significance, access roads in residential and commercial areas, mountain roads*	*Streets with a subordinate traffic significance. Rural roads with a rural character (routes of freight and connections, access roads, etc.)*	*Separately managed bike lanes as a connection of localities and/or of importance for rush-hour traffic or school transport.*
P5	**P6**	**P7**	**Sledding streets**
Separate guided cycle lane as a connection with a local development function or leisure traffic	*Designated walkways, pedestrian zones, shopping streets, school paths, paths in the area of hospitals and the like. Facilities, stops of public transport, as far as these are within the scope of the municipality*	*Parking lots, storage areas, parkways, other traffic areas*	*Streets that are kept without maintenance as a leisure area for sledding*

The definition of these categories establishes a hierarchy of zones in order that make easier to organise the ploughing routes, and distribute the personnel, vehicles, material etc. The final aim of this is to minimize the reaction time to spread the de-icing material and ploughing activities, as well as minimise the number of empty trips and making possible to make more trips for winter service.

The categories are regularly revised and actualised with real information from last winter experiences, that let plan certain condition that may originally not have been taken in to account and they may lead to a change of the vehicles and staffs or a redefinition of the road/street category.

4.2.2 What kind of de-icing and gritting agents are regularly used in Austria?

The choice on the spreading strategy is made depending on road, according to the categories defined in RVS 12.04.12 (2010) [1], and the current weather conditions. The kind of agent is as well a basic choice on the spreading strategy. The ploughing strategy and the de-icing or gritting agent reload possibilities must also be considered.

The spreading materials on the road can be divided by gritting and de-icing agents, given what is determined by the RVS 12.04.16 (2011) [2].

<u>Gritting agents</u>

These agents are natural mineral spreading medium (gravel, sand) consisting of particles of stone that are used to increase adhesion between tires and the road surface, therefore preventing skidding when there is road frost or snow.

The main inconvenience of these agents is that their effect is lost in a short time with passing cars. Hence, their use is allowed only (according to the regulation RVS 12.04.12 [1]) for roads belonging to categories C/D and P2 to P7. That is, in the kind of roads with low traffic intensity. Its use also, does not help ploughing, since it does not create a layer of brine that separates snow from the road, as de-icing agents do.

Their use may also generate fine dust particles, which means that it is recommended to use low dust developing agents. For this reason, the remaining agents on the traffic areas must be collected after the winter period. This increases the cost of the gritting agents.

Gritting agents used in Austria are crushed stones of grain size 4/8. As an exception, in urban areas other grain sizes can be used, for example 2/4 or 3/8. They cannot have any smearing content, and if possible are to be stockpiled in dry conditions.

<u>De-icing agents</u>

De-icing agents are used to lower the freezing point of water and either avoid the creation of a frozen layer over it, or they thaw the already existing snow, ice or frost. In this second case, the thickness of the frost or snow that can be thawed by the application of de-icing agents is strongly limited, ranging from 0,5 cm for $NaCl$ to 0,8 cm for $CaCl_2$.

De-icing agents can be used for a preventive strategy, if snow or frost is forecasted in few hours, or in a thawing strategy, to thaw frost or snow on the road. This can be combined with mechanical methods as plough. Further concepts of these strategies are explained in section 4.2.3.

The main de-icing agents are Sodium chloride (NaCl), Calcium chloride ($CaCl_2$) and Magnesium chloride ($MgCl_2$) either in a dry or wet mixture. Other de-icing agents could be used if proven practical and that they have been tested to accomplish the requirements for safety, health and environment, which are:

- High thawing efficiency (thawing performance, effective period)
- Low environmental impact (living beings, plants)
- Low surroundings impact (infrastructure, vehicles)
- Economical resources used (cost for purchase, replacement, stock and spreading)
- No lasting reduction of grip on the traffic surface

It is essential to avoid a reduction of the grip due to overdone use of the spreading material. It is always recommended to use the minimum effective quantity of spreading material for this reason, as well as environmental and economic reasons.

The composition of the de-icing agent is the key to reach the optimum thawing. In the guidelines, aspects to take into account are pointed out and in some cases limit values are fixed. They are:

- Maximum humidity: 0,5M.-%
- Thawing effective substance must be always as high as possible and the quantity of insoluble substance should be as least as possible. The minimum proportion of the de-icing agent material according to the guidelines:
  - NaCl 97,5 M.-%
  - CaCl2 77 M.-%
  - MgCl2 47 M.-%
- Sulphate concentration should be as minor as possible, so as to avoid the damage of concrete. In the guidelines, it is determined that the maximum value of sulphate (anion SO_4^{-2}) is 10.000 mg/kg.
- The grain size has influence on the thawing velocity, being fine grain salts faster effective. Therefore, is Vacuum salt faster in thawing than Rock salt if used dry. But with bigger grain salts the brine bottom layer is achieved, which is convenient when the spreading is followed by ploughing (see Figure 2. Influence of the grain size in thawing). When wet salt is used, the bottom brine layer is the same as rock salt even with vacuum salt due to the formation of lumps.

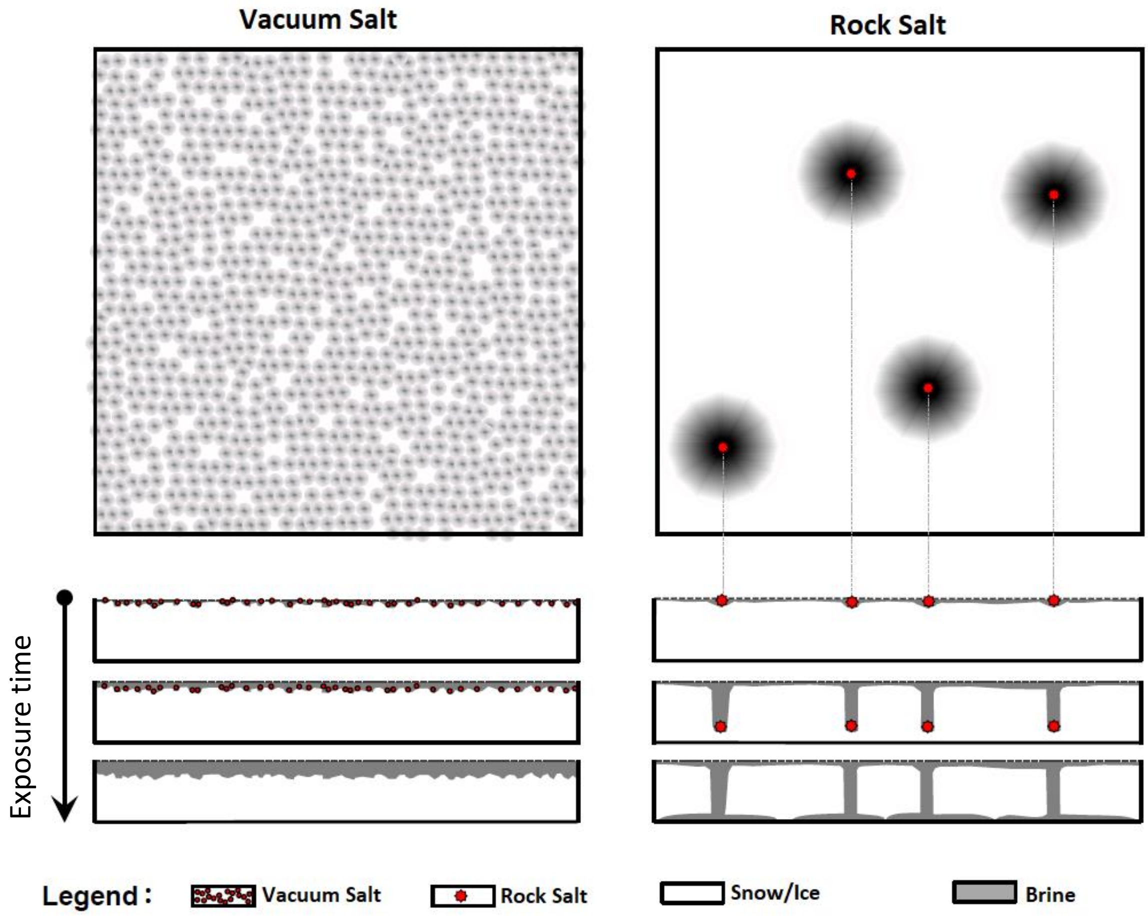


Figure 2. Influence of the grain size in thawing [4]

- Depending on the de-icing agent the guidelines set requirements for the grain size distribution:
  - NaCl:
  - Portion M.-% <0,125mm Maximum value 5 M.-%
  - Portion M.-% 0,125 mm to 3,15 mm Minimum value 95 M.-%
  - Portion M.-% > 3,15 mm to 5,00 mm Maximum value 5 M.-%
  - CaCl2, MgCl2:
  - Maximum grain size: 8mm
- Use of de-icing agent depending on the temperature of road surface:
  - NaCl: -10 °C
  - CaCl2: -20 °C
  - MgCl2: -15 °C
- The costs of $CaCl_2$, $MgCl_2$ are approximately three to four times the costs of NaCl, which costs approximately 90 €/t [4].

De-icing agents' mixtures

These mixtures are composed by portions of two or more kinds of de-icing agents. Wherein, individual components may also be admixed in dissolved form [1].

Wet mixes (prewetted salts): is a mix of NaCl (Dry salt) and a humidifying solution (Brine). The humidifying solution is usually composed by NaCl in 20% or $CaCl_2$ in 23%, or $MgCl_2$ dissolved in water.

Saturation of the prepared brine should be avoided so that a rapid reaction (thawing) is achieved when the salt is applied with the dry salt. Commonly the proportion for the wet mix is a 30% of mass of brine and 70% dry salt so called 'Feuchtsalz' FS30.

This damp salt mixes are the most efficient in terms of losses, since the salt sticks to the roadway and stays longer than for dry salt or liquid salt mixes. The texture of the roadway is also influent in the time that the salt stays on the road surface. And it may encourage rapid thawing action, especially for dry air conditions.

The brine needed for these mixes can be purchased premade, but this is not convenient since most of the product is water and it is expensive. There is the possibility of mixing the brine manually, but it requires staff costs for a low mixing efficiency. There is also the option of a mixing plant, with an approximate production capacity of 10000 l/hr, which can be attached to the salt silo and functions automatically. The drawback of this system is that it is only profitable for high brine consumption.

Dry mixes: Under consideration of the roadway and the weather conditions NaCL and $CaCl_s$ can be used in an appropriate blending ratio.

Liquid de-icing agents

In winter road maintenance salt solutions, or brine, are used predominantly when their freezing point is below the prevalent roadway temperature and they do not cause a rise of the slipperiness on the road. The disadvantage with this de-icing agent are the losses and the fact that salt amount on the roadway decreases much faster with the passing cars than if compared with dry or wet de-icing agents [4].

4.2.3 How are the de-icing agents spread?

Following the spreading routes and categories of every zone, different spreading materials and vehicles are used adapted to the conditions. The spreading materials used in Austria are explained in section 4.2.2. This section is going to focus on the kind of vehicles used to spread the materials and their security requirements as they are defined respectively in the guidelines RVS 12.04.12 [1], and ÖNORM EN 13524 [20].

Spreading materials [1] should be applied evenly to the road surface by means of adjustable and speed-dependent spreading devices (spreading quantity, spreading width). The spreading devices generally consist of a storage container, a conveying and dosing device and a spreading agent distributor. (See following Figure 3)

Spreader for wet salt Spreader for brine

Figure 3. Types of spreaders [4]

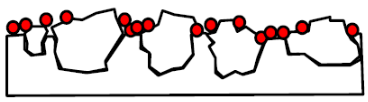
Spreaders must be reliable operative during winter service under extremely low temperatures and other singular burdens. The type and design of the spreaders must correspond to the material used and must be adapted to the carrier vehicle used, in particular to its load carrying capacity. The technical design of the spreaders must ensure corrosion protection and low wear. For an economical and effective use of spreading material during the spreading process, it is possible to provide controlled spreading devices with remote control in the driver's cab of the spreading vehicle.

The spreading is often combined with a ploughing plan depending on the road condition and the weather forecast to ensure a clear and safe road, as explained in the following section.

Spreading strategies

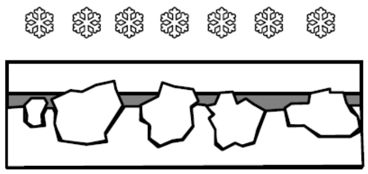
There are two kinds of spreading to be considered, preventive and thawing or late spreading combined with ploughing. They have already been introduced in previous sections.

Preventive treatment has the aim of avoiding the formation of frost or thawing snow, before the event has happened. This serves the purpose of avoiding potentially slippery roads (e.g. hoarfrost) and eases snowploughing in the next treatment. To do so the amounts applied on the road surface range 5 to 10 g/m^2. This layer of brine is to be replaced after each treatment and prevents the adherence of snow or ice on the road surface, without creating large amounts of snow slush. A summary of preventive spreading can be seen in Figure 4 [19] [4].



1. Preventive treatment before snowfall event

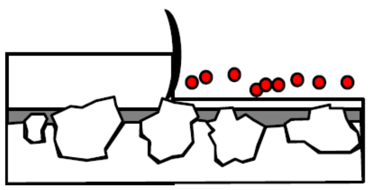
e.g. 10 g/m^2 on the eve of a snowfall event of 1 cm/h road surface temperature = - 5°C with treatment intervall = 3 h results in 3 cm snow depth per treatment interval



2. Dilution and formation of 2 phases

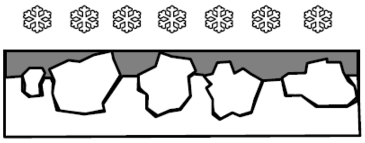
First phase snow/ice above (about 0% salt) density 0,1-0,9 g/cm^3.

Second phase bottom brine below (8% salt) of density 1,1 g/cm^3. The applied salt is dissolved gradually, until an equilibrium concentration is reached at 8% and -5 °C.



3. Snowploughing and salt application

The brine film prevents adhesion of the snow on the road surface and relieves further snowploughing. For salt application applies: remaining snow + applied salt = brine 8% (salt consumption depends on quality of ploughing and road condition)

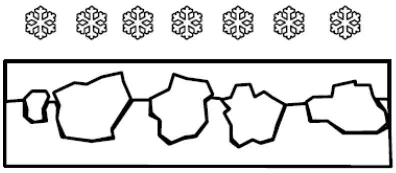


4. Thaw residual snow (continue with 2.)

Thaw of the remaining snow and brine formation > 8%; ongoing development according to 2 until end of snowfalls (discharge loss due to traffic and mixing not considered)

Figure 4. Preventive treatment model of spreading-ploughing [19] [4]

Delayed spreading has the purpose of thawing already existing frost or snow on the road surface. To reach this, it must be known that the thickness layer that can be thawed is limited, with NaCl 0,5 cm and with $CaCl_2$ 0,8 cm at a temperature of -3°C [4]. Otherwise, the operation should be repeated until the frost or snow on the road is completely removed. The usually amounts range between 10 and 40 g/m^2, depending on several conditions. If snow can no longer be thawed due to the thickness, the one possibility is to reach the brine bottom layer with the spreading of 10 g/m^2 of coarse grain salt as seen in Figure 2 and then ploughing, but this needs time to form the brine bottom layer. Another possibility is to plough directly, which may require more energy and time, due to the absence of the brine bottom layer. And then go on with spreading as seen in following Figure 5.

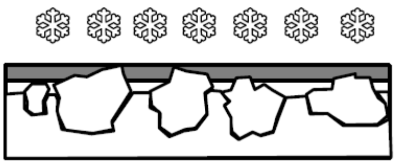


1. Snowfall + formation of ice frost

Snow / ice 0% of de-icing salt. Density 0,1 – 0,9 g/cm^3 freezing due to pressure of traffic to the asphalt

2. Ploughing followed by spreading

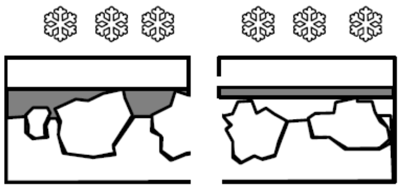
More energy needed for ploughing, larger residual snow amount, more salt Residual snow + salt = saline solution> 7% (total residual snow must be thawed)



3. Road frost during thawing process

Salt solution> 8%. Density 1,1 g/cm^3

Snow / ice 0%. Density 0,1 – 0,9 g/cm^3.



4. (Partially) melt residual snow

or remaining road frost if the amount of salt in the brine is too low to thaw all the residual snow.

Figure 5. Delayed road treatment model for spreading-ploughing [19] [4]

Every road maintenance depot needs to have defined routes on the area served, so as to optimize the trips and try to treat the served streets or roads as quick and efficiently as possible. These routes are revised and actualised, if necessary, with the real information of past winter period to improve them, and apply changes if necessary.

The routes have the aim to reduce the time of reaction, and the number of empty rides, and if possible to make additional rides and have a clear idea of what needs to be done. The routes need to take into consideration particularities of the area, and the vehicles, equipment and personnel available.

In urban areas, to plan the routes it must be considered that every time that spreading or ploughing is carried out it causes traffic jams and that a lorry must drive through the streets, which may be not possible depending on the street width.

Another aspect to be considered for the routes of winter maintenance is where to leave the removed snow until it can be picked up. This must be organised by each road maintenance depot, regarding the local characteristics.

Several recommendations of the strategy to be carried out depending on the weather and roadway conditions (such as skid resistance, temperature, and weather forecast that are followed in Austria can be seen in the Table 4 extracted from Hoffmann, M et al. (2011) [19] and [4].

Table 4. Typical weather and road conditions with each winter maintenance recommendations. [19] [4]

Picture documentation	Road conditions	Treatment recommendations
Dry Road	Very good (usually no problems)	Minimal salt application only with hoarfrost:
	High skid resistance µ = 0,7 – 1,0 Road surface temperature -30°C to + 60 °C No sleekness due to hoarfrost expected Sleekness due to hoarfrost possible (generally 02:00 - 04:00).	No treatment required No treatment required Preventive Treatment 5 - 10 g/m^2 with beginning hoarfrost (02:00 - 04:00)
Wet road	Good (black ice possible)	Treatment only at temperatures below 0°C
	Road surface temperature ≥ 0 °C no dampness Medium skid resistance µ = 0,4 - 0,7 Damp roadway Medium to low skid resistance µ = 0,3 - 0,6 Road surface temperature < 0°C Risk of black ice; very low skid resistance µ = 0,1–0,3	No treatment required check lane grooves (risk of aquaplaning) Preventive treatment is crucial Ploughing & salt application from 20 to 40 g/m^2 & warning messages

Snow next to wheel tracks	Fair (problems when changing lanes)	Ploughing and salt application as required
	No snowfall Wheel tracks dry or wet skid resistance μ = 0,3 – 0,5 Snowfall, Snow remains in wheel tracks (grey - white surface) low skid resistance μ = 0,2 – 0,4	Ploughing and salt application 20 - 30 g/m² Ploughing and salt application 10 - 20 g/m² when less than 0,5 cm snowfall in treatment interval With snowfall > 0,5 cm ploughing & salt application of 10 g/m² until end, then 20 g/m² to 30 g/m²
Snow in wheel tracks:	Bad (very low skid resistance)	Ploughing and salt application as required
	No snowfall, cleared low skid resistance μ = 0,2 - 0,3 road surface temperature ≤ 0 °C Snowfall, cleared, not cleared, precipitation < 0,5 cm in treatment interval (≈ 3 - 5 mm snow) Snow > 0,5cm in treatment interval low skid resistance μ = 0,2 - 0,3 road surface temp. -20°C to 0°C	Treatment with ploughing and salt application to clear the road of snow Preventive treatment prior to preciptitation event if possible, then ploughing and salt application With snowfall > 0,5 cm ploughing & salt application of 10 g/m² until end, then 20 g/m² to 30 g/m²

Black ice	Critical (almost no skid resistance)	Mechanical removal & maximum salt application, closing of roads:
	No precipitation Roadway surface satin silk to Reflective Almost no skid resistance µ=0,05–0,2 Precipitation (Snow) Road surface temperature ≤ 0 °C	Preventive treatment if possible, maximum salt application at icy parts. Staggered treatment. Closing of roads only in consultation with the police, then mechanical removal combined with maximum salting to remove the ice. Best strategy to avoid accidents is to close the roads until a sufficient skid resistance is reached again.

4.2.4 How are spreading agents stored?

Enough storage capacity is fundamental to guarantee the winter service maintenance. In the Austrian guidelines [1] it is recommended to have enough storage capacity for at least 50% of the needed spreading agents for the whole year. Although the current tendency is to build maintenance depots with enough capacity to store the whole winter period consumption of salt and gritting agents.

The requirements for an efficient stockpiling are several. From organisational point of view, to decide which spreading agents are going to be needed and being able to store the needed amounts on time before the winter season begins is basic. As well as to ensure through contracts with providers quick additional needed deliveries during winter period, although this is decreasingly common due to the current tendency to enlarge storage capacity. In addition, the location of the maintenance depots must be at the optimum locations of the road network.

From an operational point of view, it is basic to guarantee that the stored spreading materials are kept in dry conditions and that the storage capacity is sufficient. A quick and easy load of the vehicles is also very convenient.

The main storage units are silos and warehouses that can be seen in Figure 6.

Silo (from 30 m^3 to 300 m^3)

Warehouse (from 400 m^3 to higher)

Figure 6. Storage of spreading agents [4]

Silo storage

Used silos for storage are made of timber, metal or plastic. They have mainly a capacity between 30 m^3 and 300 m^3. Although they can be of higher capacities.

They consist of a reservoir, outlet cone, pusher, fill and breather pipe, possibly a level measure or weight measure and foundations with supports.

An advantage of the use of silos as storage units is the easy and quick way to fill the vehicles, the driver can do it all alone and by gravity force, without the need of any additional equipment. To take into consideration is the higher price of this storage type and is that it is possible for the silo to get stuck if the conditions for flowability of salt are not appropriate. i.e. Anti-caking agent amount of salt and maximum 0,5% of mass humidity during the whole process as it is explained in section 1.1.

Warehouse

Warehouses can be made of timber or concrete in any desired capacity. The possibility of manipulation is to be guaranteed by means of appropriate ceiling heights and gate widths. Sufficient ventilation must also be ensured. For the filling of spreaders there is always a charger in the warehouse, i.e. wheeled loader with corresponding dumping height, conveyor belt or hall crane.

In certain exposed locations like gradient areas it is possible to place small containers of about 1 m^3 capacity to spread gritting agents.

4.2.5 Other aspects of winter maintenance

Weather information is often necessary to react within hours lapse of time, to carry out the preventive treatment of the road. Reliable weather information is essential to carry out an appropriate winter service.

Given the deep impact in society of winter road maintenance, it is also very important all the tasks that have to be done to keep the citizens informed. At the beginning of the winter season it is important to inform in advance about possible disruptions in order to avoid possible future complaints. Public opinion can be influenced by press reviews, so it is important to have a service able to answer the questions of users and press regarding the actions of winter road maintenance.

Besides information and a correct winter service strategy, to keep safety on the road it is basic also to adapt the driving to the weather conditions.

5 Laboratory tests

5.1 Description of the flowability tests

There are different standardised methods that can be used to determine the flowability of a material, for fluid concrete it is usual to use the Abrams cone (BS EN 12350-2), and for plastics there is the Determination of pourability (ISO 6186: 1998) through flow time, or the measurement of the angle of repose defined by ISO 4324:1977 for powder and granules.

These standards define methods that can describe flowability, but are not adequate for materials as de-icing salt, granulates which tend to cake [21]. The reason is that due to the funnel shape of the apparatus used to carry out the tests, the outlet can be clogged by lumps, leading to wrong results of the real flow capacity.

For salt, there is not a certain method generally used, although its flowability is strongly affected by moist and storage conditions. In this thesis, the test carried out to measure the flowability of salt is known as 'Auslauf box nach Sonntag' and which obtained parameters to characterise flowability.

Auslaufbox is an apparatus which use is recommended for bulk goods, and the tests can be easily carried out in a short period of time [21]. The obtained parameters with this test are outflow rate expressed in percentage and internal friction angle [22]. More about the performance of tests is described in section 5.1.1.

The use of this test is recommended for bulk goods such as bran, beet seeds, plastic granules, grinded rubber, salt, hazelnuts, etc. and its aim is to determine the flow rate, between the discharged amount of material and the remaining material inside of the box [21].

The test is meant to be used for practical purposes, and so it is its accuracy. As it has been seen, depending on the humidity level and anticaking agent quantity, the results of different tests for the same sample may vary quantitatively.

With this test performed on salt, two main flowability parameters are obtained, the discharge ratio and the friction angle of the salt are intended to be related to its moist level, characterised as percentage of water content in mass of the salt and the amount of anti-caking agent.

Other parameters seen to have influence on the result of tests are time, the sample preparation and the grain size of the sample.

In this case, the experiments were carried out on NaCl rock salt and vacuum salt, using different amounts of anticaking agent and different moisture levels on the salts. The samples were obtained through contribution of some salt companies, and have been renamed with the aim of keeping their anonymity.

5.1.1 Flowability test 'Auslaufbox nach Sonntag'

The measurements of the flowability were carried out using the "Auslaufbox nach Sonntag". The "Auslaufbox nach Sonntag" stands in its narrow sides. The flap gate of the box is also located on the front side. This gate can be opened quickly because of a counterweight fixed to it. The superior side is open, through which the box is filled. Its dimensions are 20cm x 10cm x 20 cm resulting in 4 l volume.

The procedure of the test is the following, first the box is placed on a horizontal and vibration-free surface with the front side slightly above an edge and a tray must be placed under the box, so it can collect the discharged material as it can be seen in the following figure.

Figure 7. Position of test apparatus

The box is then filled through the superior open side with the help of an adapted funnel. It must be rather overfilled, so that the excess can be removed with a ruler or a similar straight object, resulting in a surface as smooth as possible and the box filled to the edge. The standard time used in this thesis has been 5 to 10 minutes between the box filling and the test is carried out. Secondly, the flap gate is opened, letting the studied material flow out of the box into the tray. The box after the test can be seen in Figure 8.

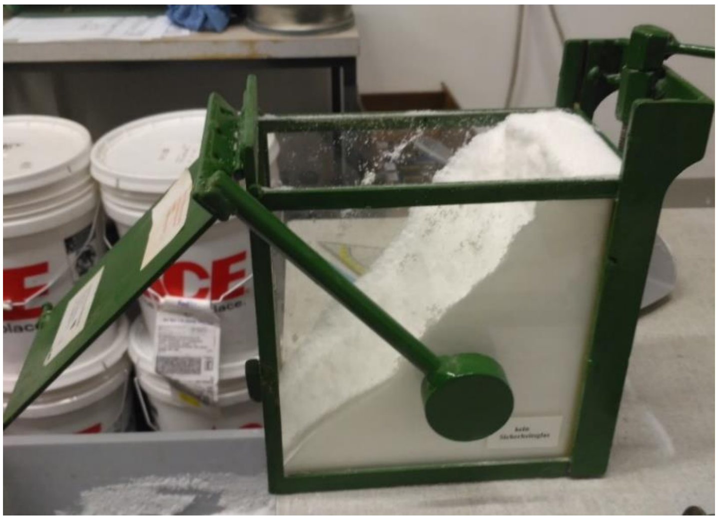


Figure 8. Auslaufbox after a test

When there is no more material flowing out of the Auslaufbox, the internal friction angle can be measured through the glass side using a protractor or directly if the box has marked angles on the glass. It should be noted that the experiments for this thesis have been carried out in an Auslaufbox without marked angles. To obtain the friction angle of the sample, it was done as seen in Figure 9, before the remaining salt in the box is weighted.

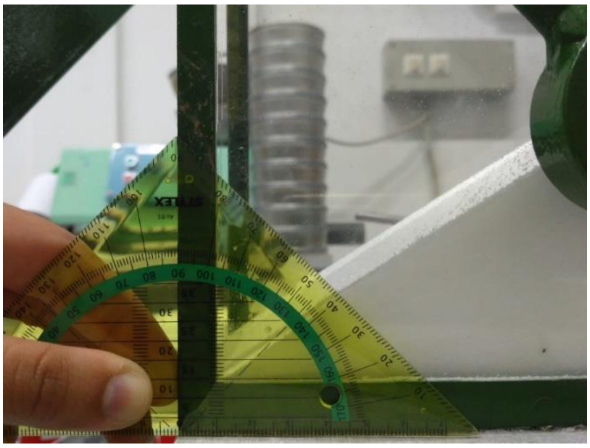


Figure 9. Measure of the friction angle

The discharged material from the box must be weighed to determine the proportion respect the total weight of material inside the box. This way the outflow-ratio can be determined. This is a material specific parameter, directly related to its flowability.

The calculation of the outflow-ratio can be summarised by the following formula.

$$Outflow\ (\%) = \frac{\mathrm{D}}{D+B} \times 100 = \frac{D}{T} \times 100$$

D: Discharged mass of salt on the tray (g)
B: Mass of salt in the Auslaufbox after the test is carried out (g)
T: Total amount of salt in the Auslaufbox before the test (g)

To weigh the discharged and total salt from the box it was used a scale with an accuracy of 2 g. Given that the usual amounts weighed around 5000 g it is an error greater than the one from standardised pourability test ISO 6186:1998 that uses a scale of 0,1 g accuracy to weigh 150 g of plastics, but still acceptable for a test intended for practical purposes.

After the discharged material is weighed, then the remaining material in the box must be also weighted. The outflow ratio percentage is obtained by dividing the discharged mass by the sum of the discharge and the remaining in the box, the total mass in the box.

For this thesis three to two determinations under the same conditions were made, cleaning off any residue adhering to the inner surface of the Auslaufbox.
Another parameter that can be extracted from the tests is the bulk density, dividing the total mass by the volume of the Auslaufbox, which is 4L.

5.1.2 Sample preparation

The salt that is going to be tested must be adequately prepared to obtain reliable results of flowability. To carry out the tests it is important to know the samples' exact humidity level. Therefore, the salt was dried at a 120°C temperature until mass constant is reached. This usually occurs already after the samples are left 12-24 hours in the drying cabinet when the mass constant is reached.

Once the salt sample was dry, it was necessary to add moisture to reach a known certain humidity content. In this thesis either 0,25%, 0,5% or 0,75% quantity of water in mass. This could be done by calculating the weight of the sample that should be water based on the humidity percentage in mass. Then the exact amount of water was sprayed over the sample in a tray placed on a scale with an accuracy of at least 0,1 g.

The process followed was to spray 5 g of water and then mixing the salt with a plastic ruler or a similar object to change the first layer of salt, as seen in Figure 10. This operation was repeated until the desired exact amount of water was sprayed over the salt sample. It is important to clean the ruler in order not to lose material during the process, and keep the accuracy as far as possible.

Figure 10: Sample preparation - water spraying

5.1.3 Main Test Programme

The programme for flowability tests can be seen in the following table Table 5. Test programme for the determination of flowability. The humidity levels for the tests find their justification on the previous work referenced [23], where it was determined through experiments in different salt storage facilities as silo, warehouse and transport truck that at every stage of storage and transportation the humidity levels are located between 0,35 and 0,5 % of mass of the salt.

Additional tests were carried out with the humidity of salt directly from the storage conditions for some salt samples. The aim was to compare if the behaviour of the salt was similar whether having been dried or not. Showing that it was in general similar. This humidity is shown in the first place on the column of humidity of samples from Table 5.

Table 5. Test programme for the determination of flowability

Salt	Grain size [mm]	Name	Anti-caking agent	Anti-caking agent [mg/kg]	Humidity of samples [%]					
Rock Salt	0/3	RS1	without	0		0		0,25	0,5	0,75
	0/2	RS2	without	0		0		0,25	0,5	0,75
	0/3	RS3	Sodium ferrocyanide	70-100		0		0,25	0,5	0,75
	0/2	RS4	Sodium ferrocyanide	70-100		0		0,25	0,5	0,75
Vacuum Salt	fine	VS1	without	0	1,2	0		0,25	0,5	0,75
	fine	VS2	without	0	0,01	0	0,1	0,25	0,5	0,75
	fine	VS3	without	0		0			0,5	

Salt	Grain size [mm]	Name	Anti-caking agent	Anti-caking agent [mg/kg]	Humidity of samples [%]					
Vacuum Salt	fine	VS4	Sodium ferrocyanide	8	0,01	0		0,25	0,5	0,75
	fine	VS5	Sodium ferrocyanide	8	0,01	0		0,25	0,5	0,75
	fine	VS6	Biodegradable Agent	12	0,01	0		0,25	0,5	0,75
	fine	VS7	Sodium ferrocyanide	22		0		0,25	0,5	0,75
	fine	VS8	Sodium ferrocyanide	95		0		0,25	0,5	0,75

5.1.4 Additional examinations

Several results from the main Test programme led to questions that required further additional tests, which are detailed in this section.

<u>Tests for longer period with applied weight</u>

During the execution of tests, it was observed that the time of the sample inside the Auslaufbox could also be an influencing factor on the results. For this reason, it was decided to carry out some further tests on certain salt samples. Another influencing factor considered to affect the flowability results was the compaction of the salt through extra weight over the sample.

The method followed to carry these additional tests out was to apply some weight (the load used for the tests was 1,336 kg) on the filled apparatus to simulate the conditions of a silo as seen in Figure 11. Then the box was let stay closed for a longer period, about 20 hours, as it was left overnight at the laboratory.

Figure 11: Tests outside the program

The programme and the samples used for these additional tests are detailed in the following table.

Table 6. Test programme extra tests for salt with an applied load and longer period in the Auslaufbox

Name of Sample	Anticaking agent content [mg/kg]	Humidity content [%mass]	Load applied [g]	Time between filling and test [h]
VS3	0	0,25	1,336	18
VS4	8	0,75	1,336	17
VS7	22	0,25	1,336	23
VS8	95	0,5	1,336	18

Additional tests on sample VS6 vacuum salt with bio anticaking agent

The sample VS6 containing a biodegradable anti-caking agent showed an irregular performance with the flowability tests. It was alleged that the cause may be the evaporation of the anti-caking agent during the sample preparation in the drying cabinet. Because the anticaking agent is composed of acetic acid and iron tartrates, obtained from by-products of the wine industry. It was decided to carry out the same tests in the programme a second time but skipping the drying cabinet, for the tested salts.

To reach the desired humidity level, one sample of salt was dried, to know the humidity of the salt after being stored in the laboratory. Another sample obtained from the same place was used for the flowability tests. The water sprayed in this case was the difference of the current water content of salt, and the desired water content to carry the tests out.

It was computed as shown in the following formula.

$$W_W = \frac{(X - H) \cdot W_S \cdot (1 - h)}{100}$$

W_W: Water needed to reach the desired humidity level (g)
X: Desired Water content (%)
h: Current humidity content (%)
W_S: Weight of the salt sample (g)

The tested humidity levels were 0,017% as the original humidity of the sample, 0,25% and 0,5%. The results of these additional tests are presented following those from the main test programme of salt sample VS6 in “5.3.10 VS6 – Vacuum salt with 12 mg/kg biodegradable anticaking agent”.

5.2 Test execution

The tests were executed from May to July 2017 in the Laboratory at the Institute of Transportation at the Technical University Vienna. The samples were sent by different salt companies from Europe.

5.2.1 Materials and tools for Flowability tests

The tools used to carry the tests out and prepare the samples are shown in the following photographs from Figure 12.

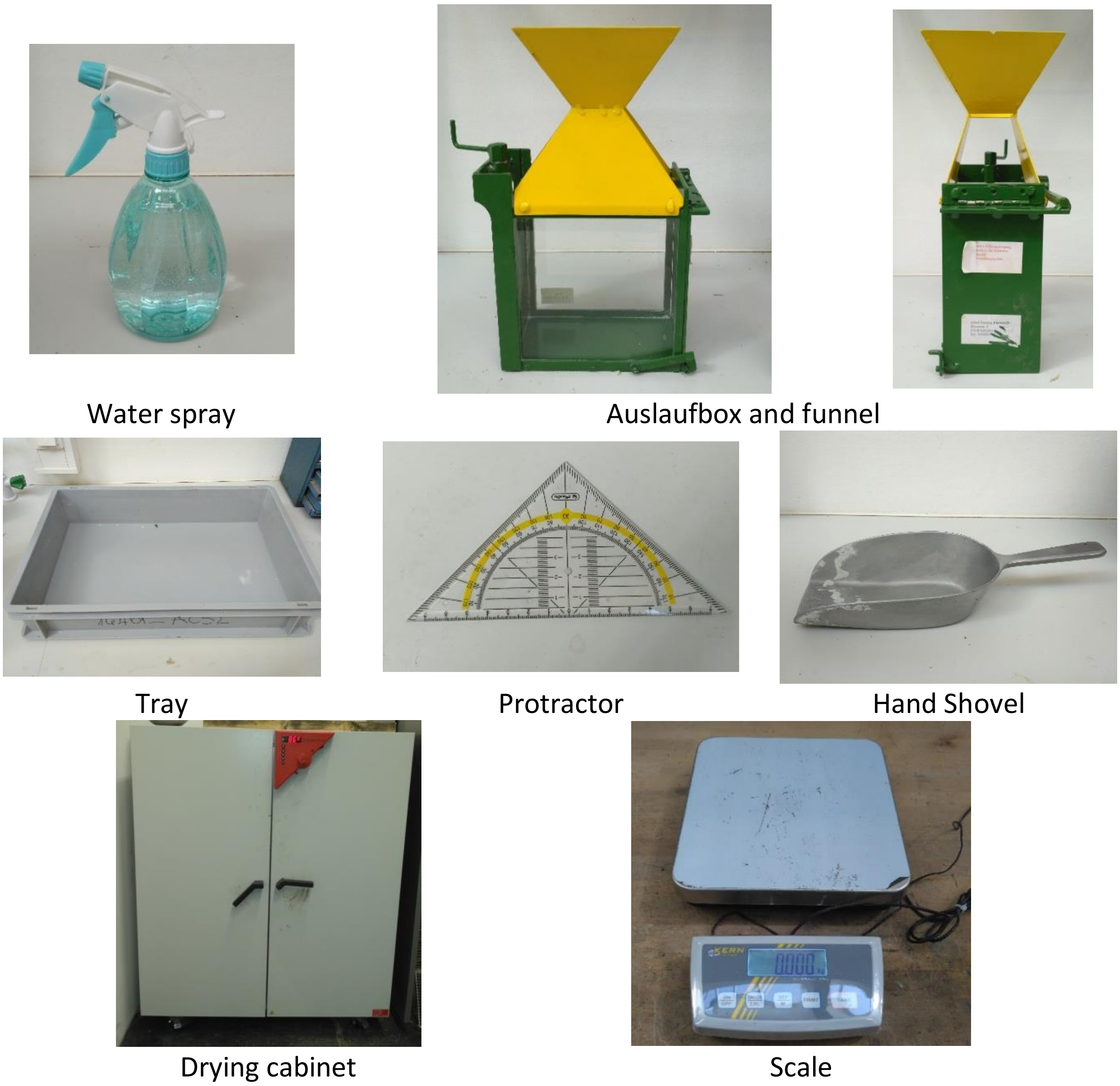


Figure 12. Materials and tools used for the tests

5.2.2 Flowability tests

To start with the proper flowability test firstly the Auslaufbox must be filled completely with the help of a funnel over the top and then levelled with the help of a ruler or a similar straight object, without pressing the sample inside of the box. Then, if the sample is damp, as it has been seen on the laboratory, so that the sample settles in the box, it is recommendable to wait from 5 to 10 minutes until the real test is carried out.

After the test, the first step is to weight the amount of salt that has discharged the Auslaufbox, and the amount that remained inside the box. This way the percentage of discharged salt can be calculated.

5.2.3 Sample characteristics

Each sample can be seen in the Annex IV. The samples of rock salt contained visible impurities as it can be seen in the picture samples RS1, RS2, RS3 and RS4. Vacuum salt samples not containing anti-caking agent VS1, VS2, VS3 can be seen to present a slightly caked appearance.
Sample VS6, containing a biodegradable anti-caking agent has a slightly yellowish colour if compared with other samples, which was more obvious live than in pictures.
VS1 sample, containing 1,2% of humidity was delivered in a bucket seen in the bottom right from the storage of samples in the laboratory.

5.2.4 Incidences during tests

There have not been remarkable incidences while conducting the tests, further than the own drawbacks from the test. As it has been seen, there is difficulty to measure the angle when the sample has in some degree water content. The problems often experienced were irregular shape of the slope, and two different angles in each side of the box. The solution carried out for the first case was to measure the average angle with the help of a ruler. For the second case, the two angles at each side of the box were measured, and then the average was taken as the result.

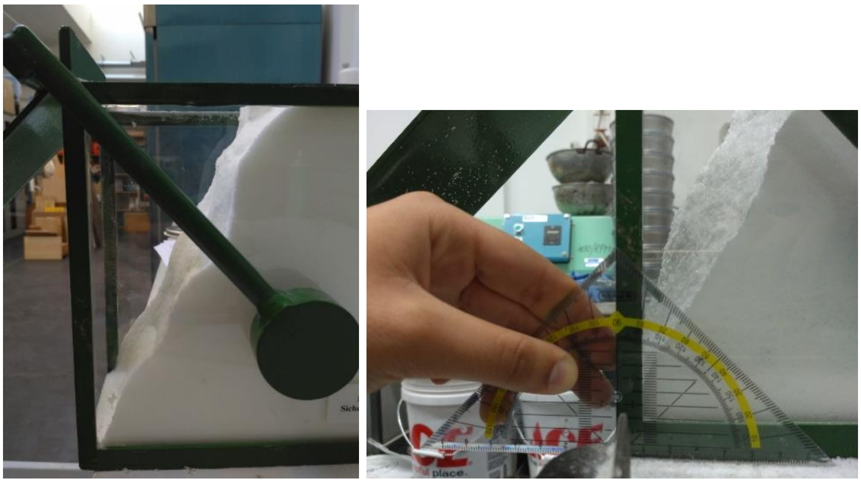


Figure 13. Problems with measurement of internal friction angle

Besides, the test results may vary, different results were obtained for the same conditions of humidity and anti-caking agent content in some cases.

5.3 Results

The results obtained for the flowability tests are presented in this chapter in graphs showing the percentage relation between discharged mass and total mass in the test box, and the salt repose angle for different water content of samples. Each sample was tested following the test program detailed in Table 5. Test programme for the determination of flowability. Then also extra tests were carried out as detailed in section 5.1.4.

5.3.1 RS1 - Rock Salt 0/3 mm and no anticaking agent

This sample is Rock salt of grain size ranges from 0 to 3 mm. It does not have anticaking agent content and the humidity to which it was received was 0,04%. The appearance of this salt is visibly not so pure, as it can be seen it contains impurities see Figure 14.

Figure 14. Sample RS1 appearance

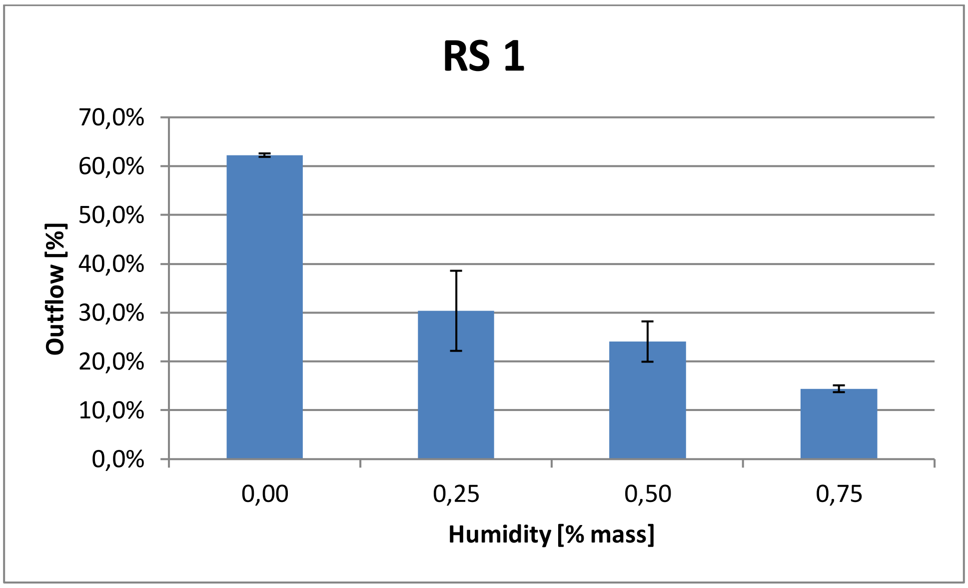


Figure 15: Ouflow ratio results for RS1

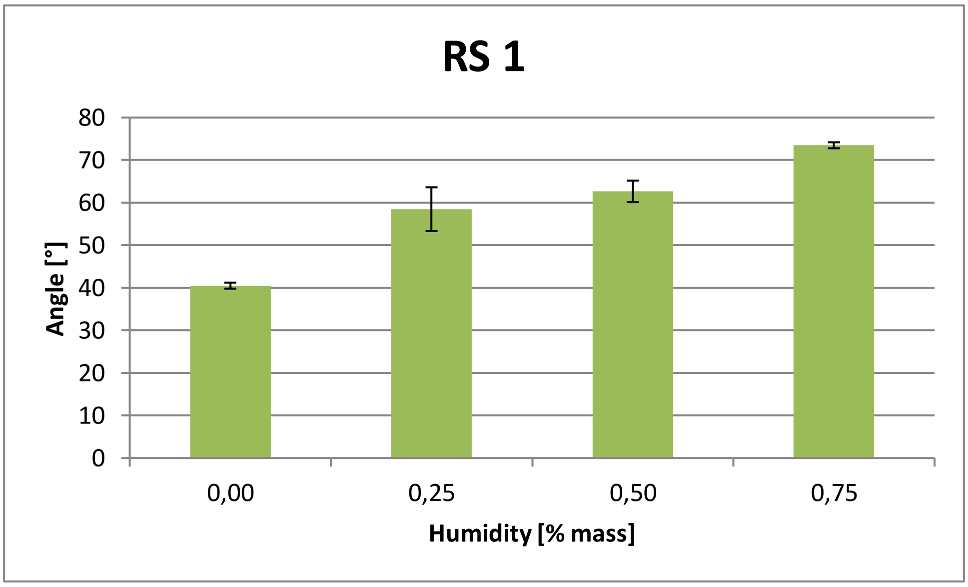


Figure 16: Friction angle results for RS1

As it can be seen in the results for the outflow ratio, it decreases with the increasing humidity. The greatest jump occurs between the samples with 0 humidity and the sample with 0.25% humidity, that have on average an outflow ratio of 62,3% and 30,4% respectively. Then it decreases almost linearly until reaching an outflow ratio of 14,4%, for a humidity content of 0,75% of mass.

As for the friction angle, it increases from an initial 41° to 74°, being consistent with the results for the outflow.

5.3.2 RS2 - Rock salt 0/2 mm and no anticaking agent

This sample is of a grain size of 0/2 mm without any anticaking-agent content. The humidity level to which it was delivered in the laboratory was 0,04% of water content, which is almost 0. The appearance of this sample is like the one seen in Figure 14, only of a finer average grain size.

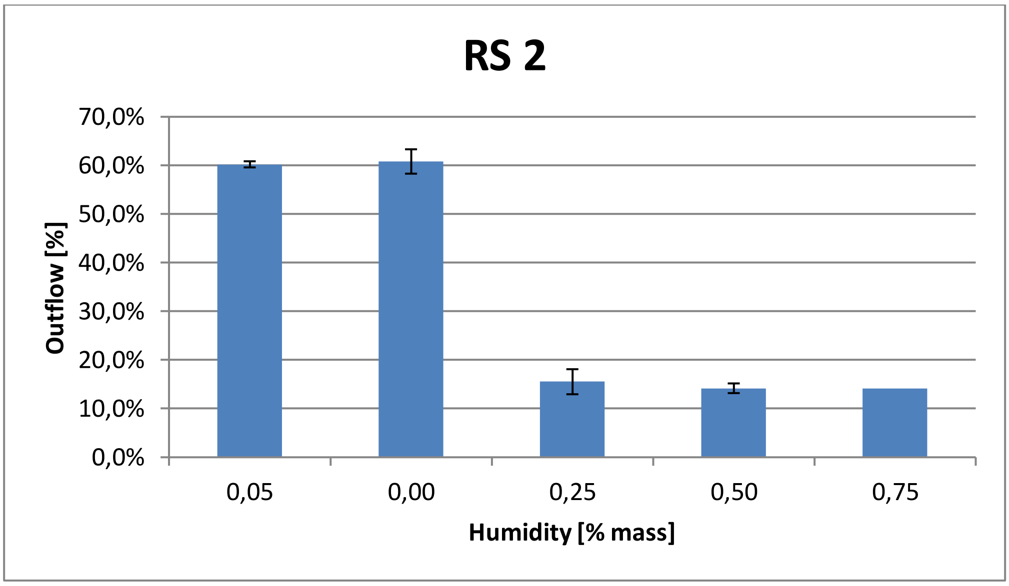


Figure 17: Outflow ratio results for RS2

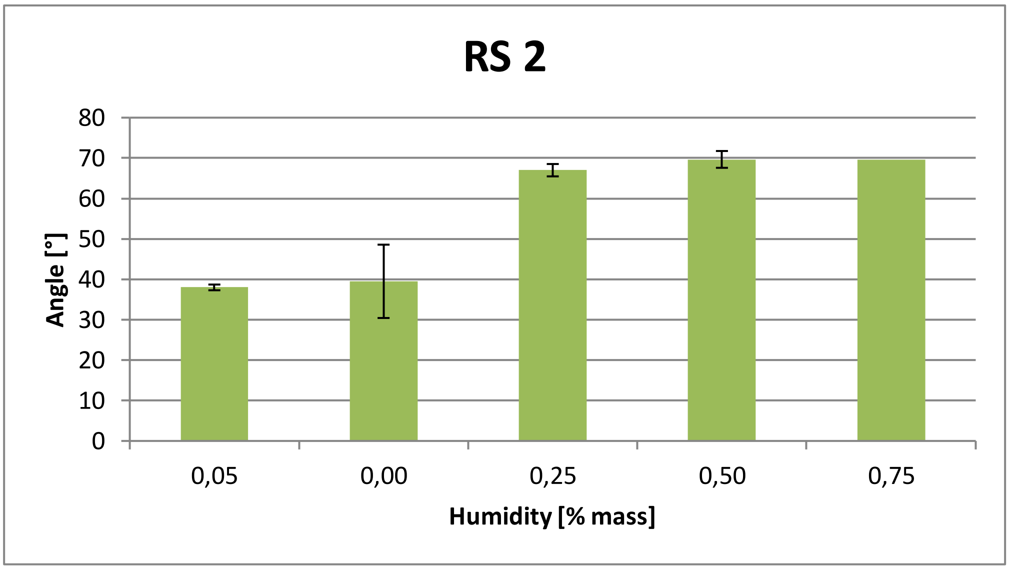


Figure 18: Friction angle results for RS2

As it can be seen in the graphs of the results, the influence of humidity for a sample of a finer grain size are more pronounced, as between moist content of 0,00% the average outflow ratio is 60,8% and the angle is 40°, and for the sample with a 0,25% of humidity the outflow sinks to 15,5% and the friction angle raises to 67°. Then, for higher humidity levels both the outflow ratio and the friction angle are kept almost constant.

This sample was also tested without being dried and the tests yield a similar result as for 0,00% humidity.

5.3.3 RS3 - Rock Salt 0/3 mm and 70-100 mg/kg SFC

This sample is Rock salt of grain size between 0 and 3 mm, containing 70-100 mg/kg of anti-caking agent Sodium ferrocyanide. The humidity content of the salt by the time of delivery was 0,15%.

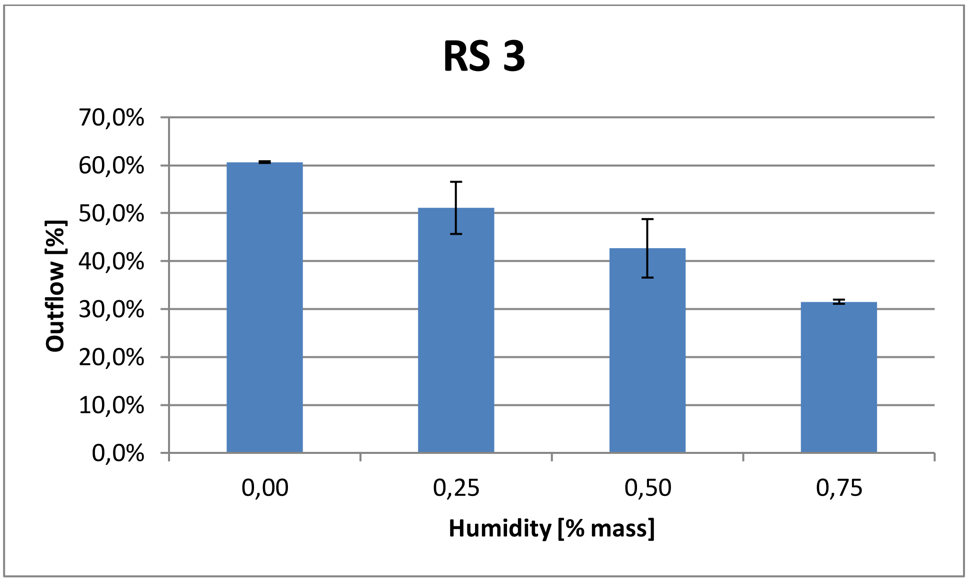


Figure 19: Outflow ratio for RS3

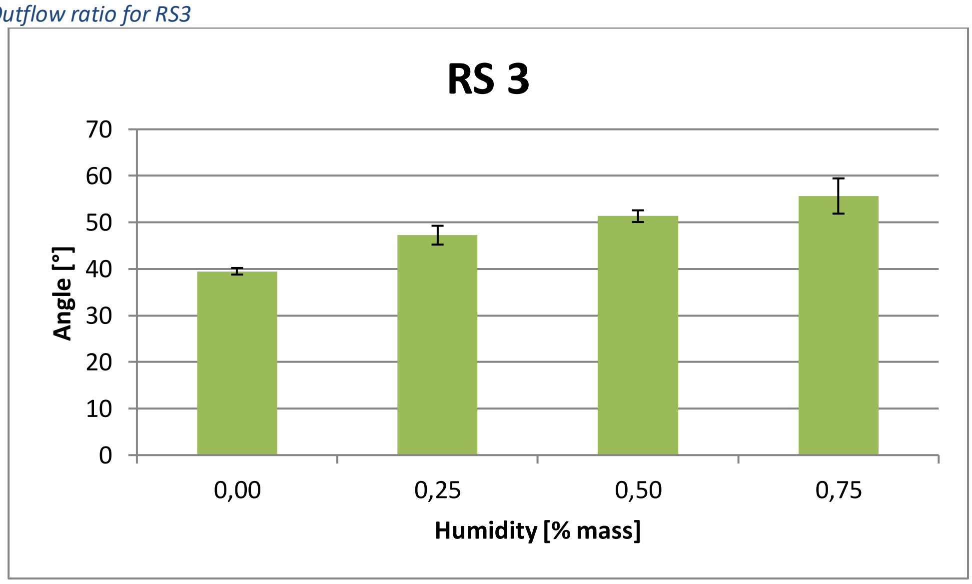


Figure 20: Friction angle results for RS3

As it can be seen by the results, the effect of the anticaking agent is notable in the results for 0,00% and 0,25% humidity. If compared with sample RS1 it can be observed that the outflow ratio does not decrease as fast as for the sample without anti-caking agent content. Reaching only 31,4% of outflow ratio with a friction angle of 56° for a humidity of 0,75% in mass. The results for the sample without any water content are similar to those of the sample RS1 and RS2.

The friction angle increases with the dampness content, but not as fast as it would for the sample without anticaking agent. Without humidity, the friction angle is 40° and with a humidity of 0,75% in mass it reaches 56°.

5.3.4 RS4 - Rock Salt 0/2 mm and 70-100 mg/kg SFC

The sample RS4 is a rock salt of grain size 0/2 mm containing 70-100 mg/kg of anti-caking agent sodium ferrocyanide. The humidity level it had at its delivery was 0,09%.

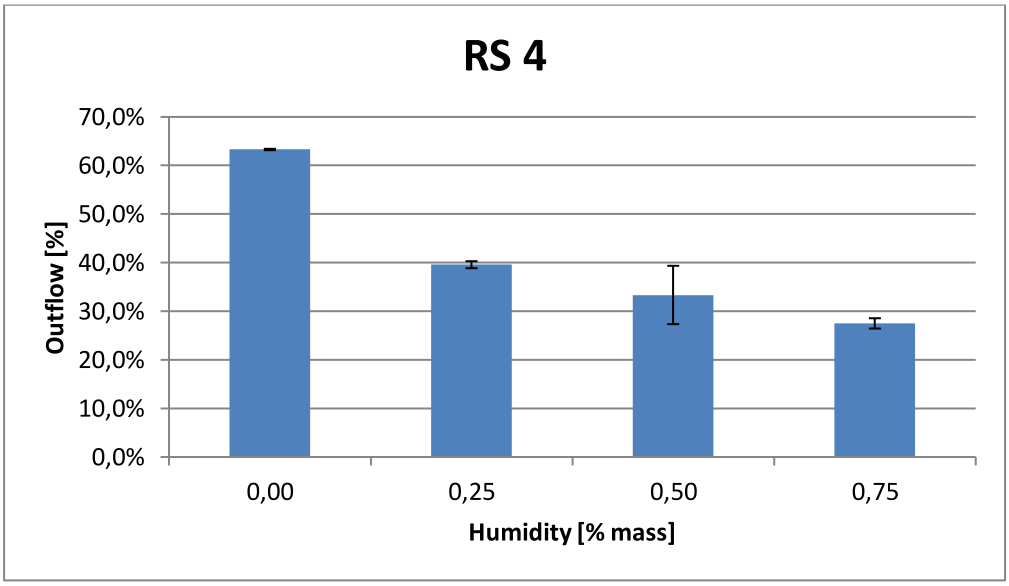


Figure 21: Outflow ratio for RS4

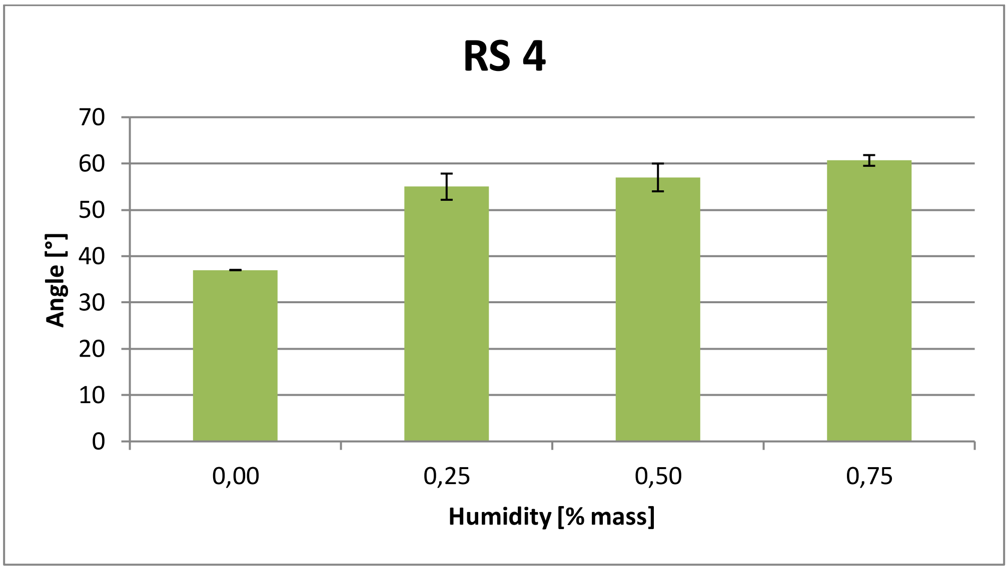


Figure 22: Friction angle results for RS4

The outflow for this sample with no humidity is 63,3%. The results for the outflow decrease not proportionally with the increase of dampness level. And for a humidity of 0,5% in mass the results vary from 40% to 28,3%, which results in a remarkably high standard deviation of 6%.

By the graphs of the results it can be understood that for a finer grain size of rock salt and without humidity in the salt, the outflow ratio is a little higher than the sample RS4, that is a rock salt of bigger grain size also containing anti-caking agent. It is also higher than the same salt with same grain size with neither water content or sodium ferrocyanide, which means the anti-caking agent is also effective without any moisture content. Although both differences are not highly remarkable.

5.3.5 VS1 – Vacuum salt with no anticaking agent

VS1 is a vacuum salt that does not contain any anti-caking agent. This sample arrived in the laboratory with a humidity level of 1,2%, which is above the level of humidity admitted by Austrian and guidelines [1], established in 0,5% for de-icing salt NaCl. This and the fact the sample contained no anticaking agent, caused the sample to completely cake after it was dried in the oven. See Figure 23.

To be able to carry out the tests with this sample it was necessary to sieve it manually with a mesh size of 1,25 mm applying force. Afterwards the water application process and tests were conducted as usual, without further complications.

Figure 23. Sample VS2 after being dried in the oven, forming one big lump

As it can be seen in the results, the fact that the salt was strongly caked and had to be sieved did not significantly affect the results. It has the same pattern observed for salts without anticaking agent content, a strong effect of little humidity level 0,25% in mass, but for higher humidity contents, the outflow and the friction angle change at a lower rate, decreasing and increasing respectively.

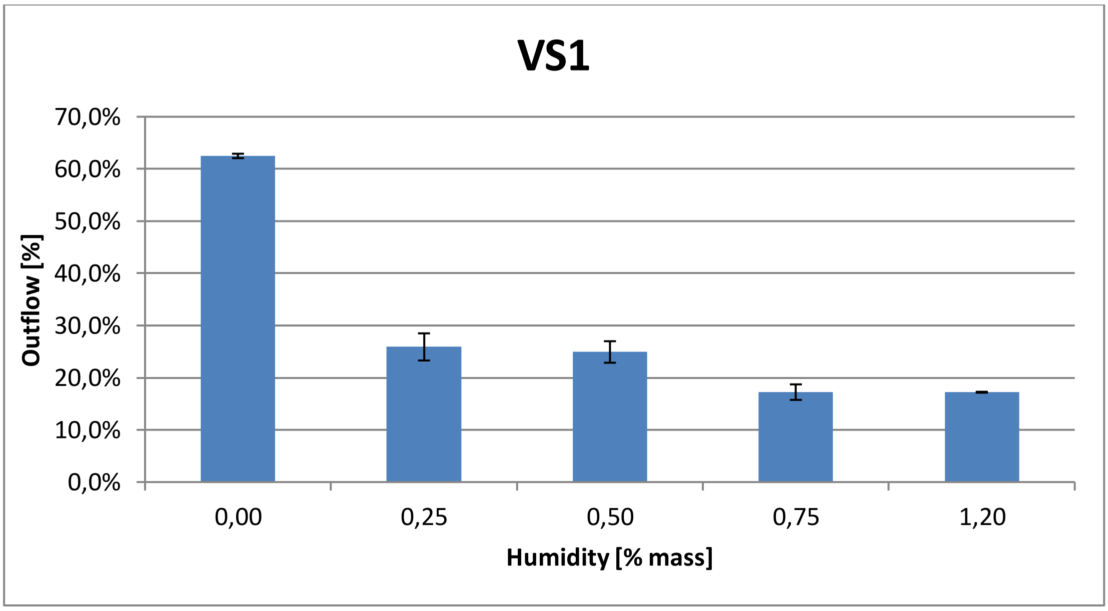


Figure 24: Outflow ratio results for VS1

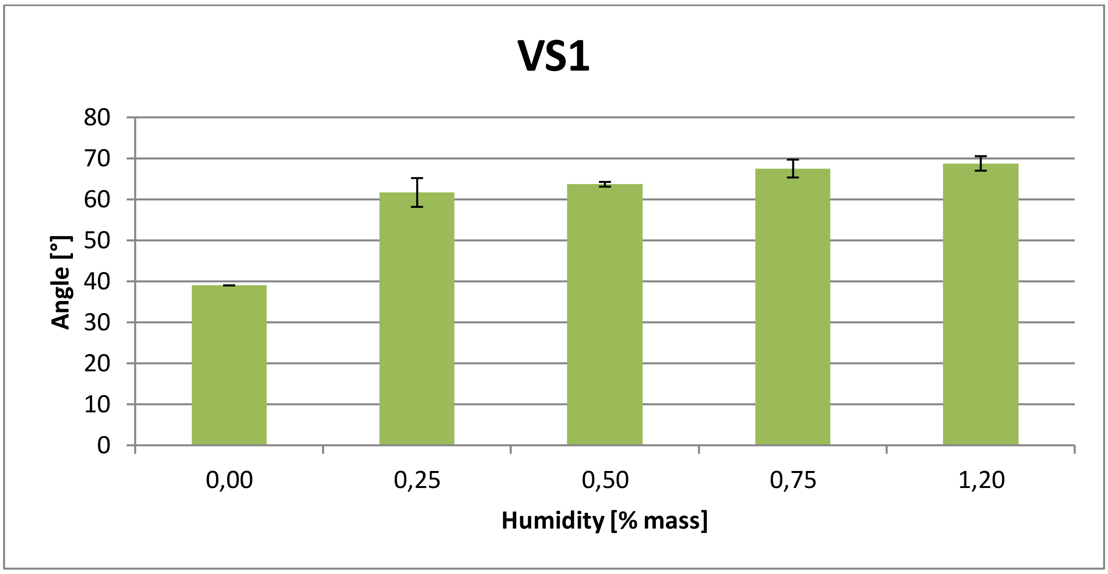


Figure 25: Friction angle results for VS1

If the results obtained for the sample real humidity in the laboratory (1,2%) and the 0,75% humidity they are quite similar, but standard deviation from 1,2% humidity is lower.

5.3.6 VS2 – Vacuum salt with no anticaking agent

This sample of vacuum salt does not contain anticaking agent and it was delivered with a humidity of 0,01%, which is practically zero.

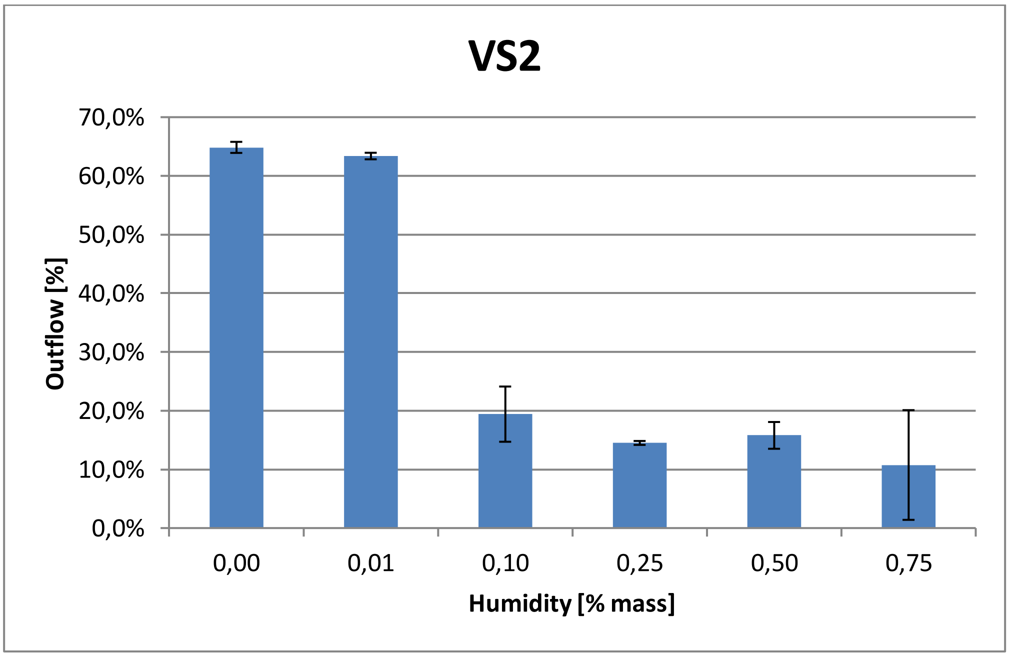


Figure 26: Outflow ratio results for VS2

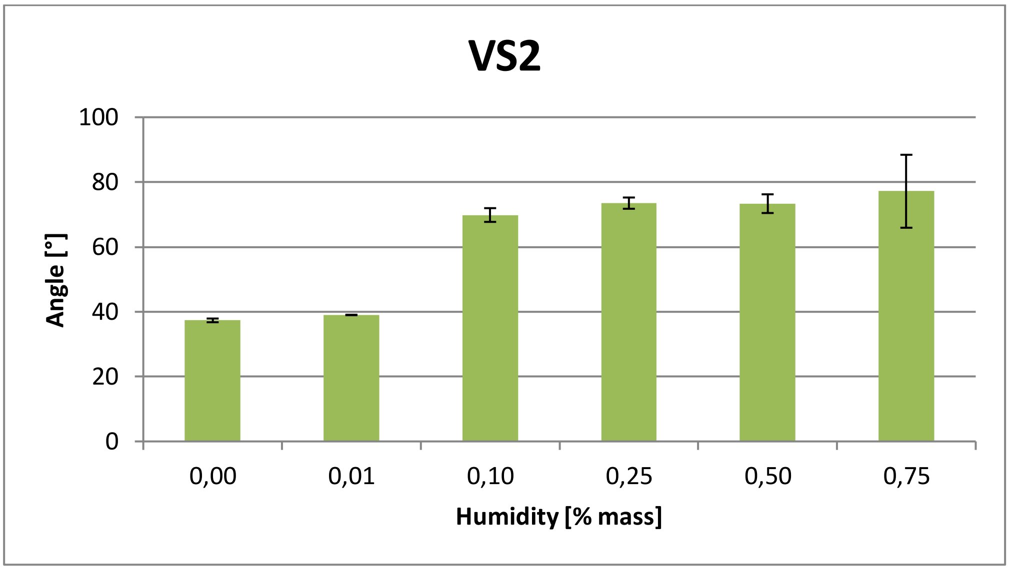


Figure 27: Friction angle results for VS2

As it can be seen, the outflow rate and the friction angle is like those from other vacuum salt samples. But once it contains a bit of moisture, the outflow sinks clearly more than in other samples, being for a dampness of 0,25% in mass 14,5%. This is lower than the outflow rate at a dampness of 0,75% in mass for the samples VS1, VS4 and VS5. The same can be said for the friction angle, that is clearly higher than the other samples.

It was also tested for a humidity level of 0,10%, to see if the results were between those yielded for 0,0 and 0,25% humidity. And as it can be seen, they are much closer to those from 0,25% humidity.

From the results obtained for the real humidity of the sample at the laboratory, they show that they are almost the same as for those of 0,00%.

For the 0,75% in mass humidity there is a significantly higher standard deviation due to the fact that in one test the outflow ratio was 0 and the friction angle 90°. As it can be seen in Figure 28. Auslaufbox with 0% outflow.

Figure 28. Auslaufbox with 0% outflow

5.3.7 VS3 – Vacuum salt with no anticaking agent

This sample does not contain any anticaking agent, was delivered in the laboratory with a humidity of 0,024% in mass. It was slightly caked, even though the lumps could easily be broken with the hands.

Figure 29. Sample VS3 with lumps from caking

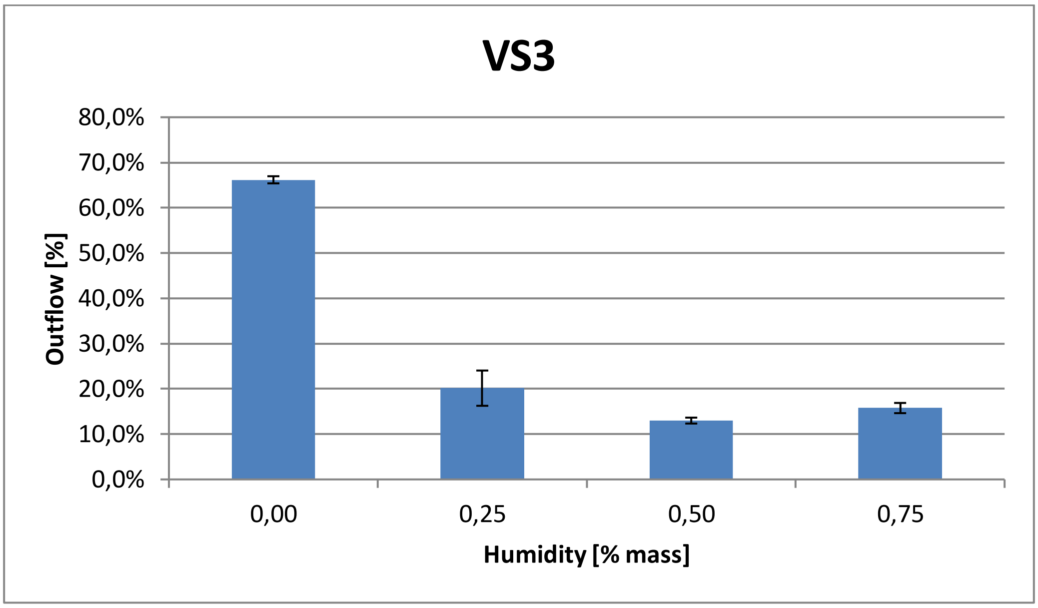


Figure 30: Outflow ratio results for VS3

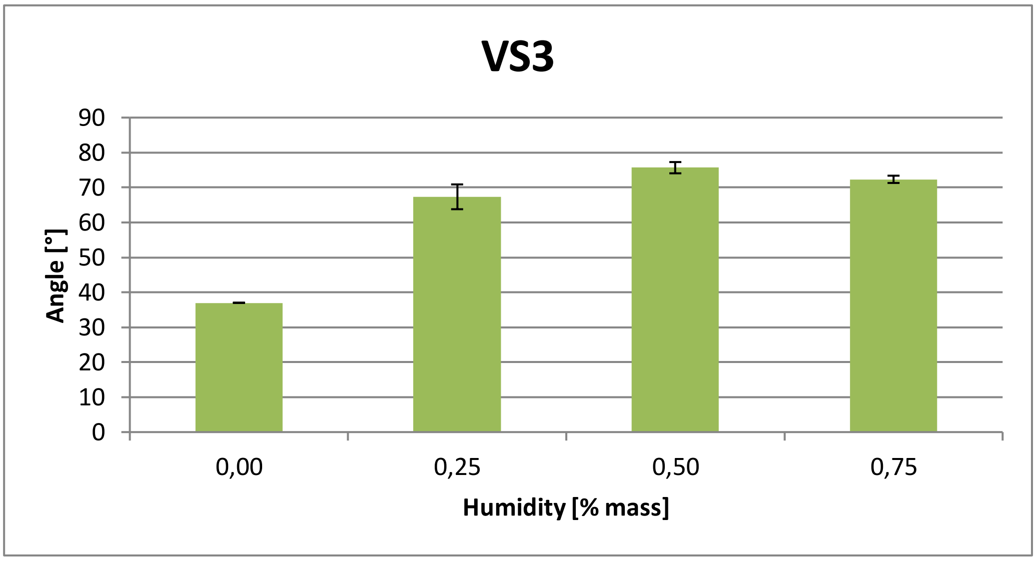


Figure 31: Friction angle results for VS3

As other samples without anti-caking agent, the behaviour of the sample varies as soon as it contains some humidity, reducing drastically its outflow ratio and increasing the friction angle. As it has been seen, the results from this sample have the particularity that for a humidity level of 0,75% it shows better results than for a lower 0,5%.

This sample was also left in the Auslaufbox for one hour for a humidity level of 0,75% before performing the test. And the results were 0% outflow ratio and 90° friction angle, which shows the importance of the time in the box for this test when it is performed on salt containing humidity.

5.3.8 VS4 – Vacuum salt with 8 mg/kg SFC

This sample of vacuum salt has particularly an anticaking agent content of 8 mg/kg. It was delivered with a humidity level of 0,01% in mass.

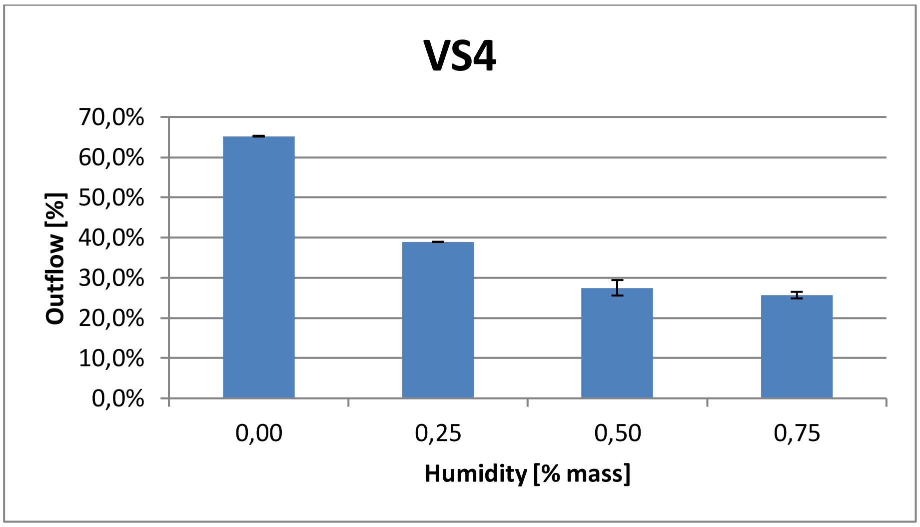


Figure 32: Outflow ratio results for VS4

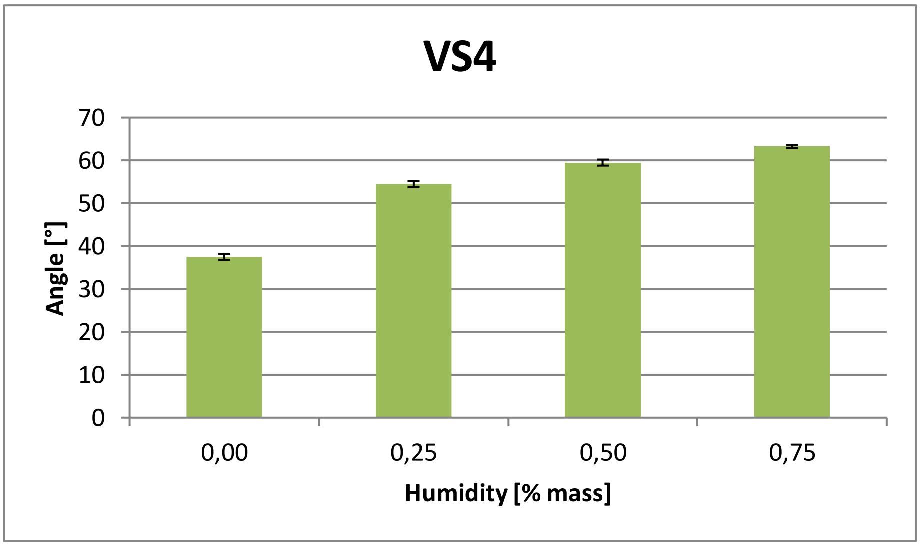


Figure 33: Friction angle results for VS4

As expected, the finer grain size makes the outflow to be higher and the friction angle to be lower than for rock salt and humidity content. For no humidity content, the outflow ratio is 65,2% with a standard deviation of 0,1% and the friction angle is 38° and a standard deviation of 1°. The outflow decreases with the increase of humidity, reaching a 25,7% of outflow, lower result if compared with rock salt, for a dampness content of 0,75% in mass and a friction angle of 63°. This shows that vacuum salts are prone to be more affected by humidity levels than larger grain salts, like rock salt. Even if they contain anticaking agent.

5.3.9 VS5 – Vacuum salt with 8 mg/kg SFC

This sample is vacuum salt with 8 mg/kg of anticaking agent sodium ferrocyanide. It was delivered with a humidity of 0,01% in mass. The sample was also tested for the humidity at delivery.

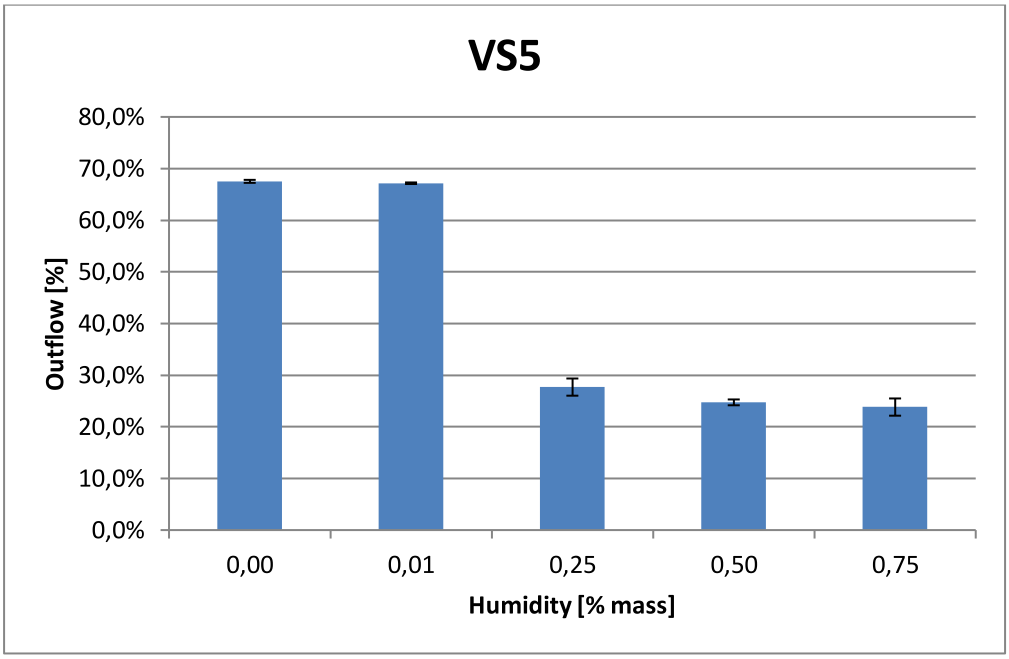


Figure 34: Outflow ratio results for VS5

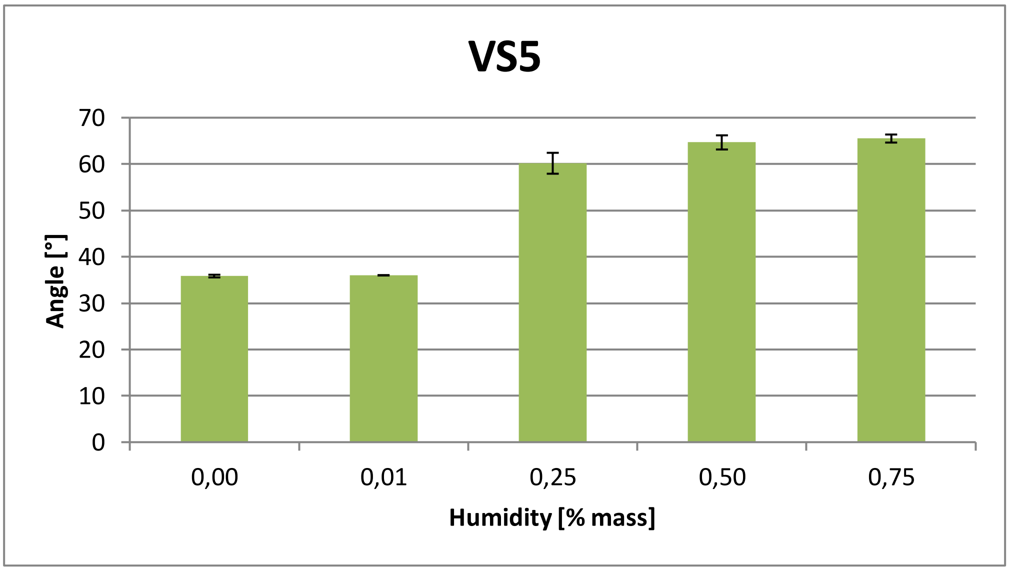


Figure 35: Friction angle results for VS5

The results show an initial outflow of 67,5% with a friction angle of 36°, that decreases rapidly to less than the half with a dampness level of 0,25%, while the friction angle almost doubles.

5.3.10 VS6 – Vacuum salt with 12 mg/kg biodegradable anticaking agent

This sample is a vacuum salt contains a biodegradable anticaking agent produced with remain products from the wine industry, such as acetic acid and iron tartrates in 12 mg/kg. The humidity at which it was delivered was 0,01% in mass. The results obtained are similar to those of salt without caking agent VS1 and VS2, even though with a slightly better performance.

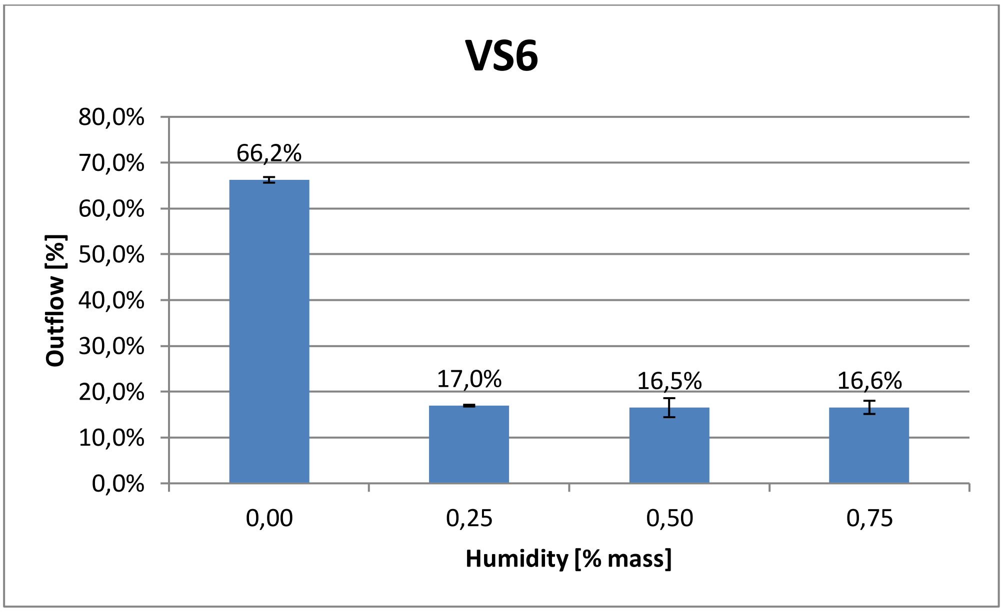


Figure 36: Outflow ratio results for VS6

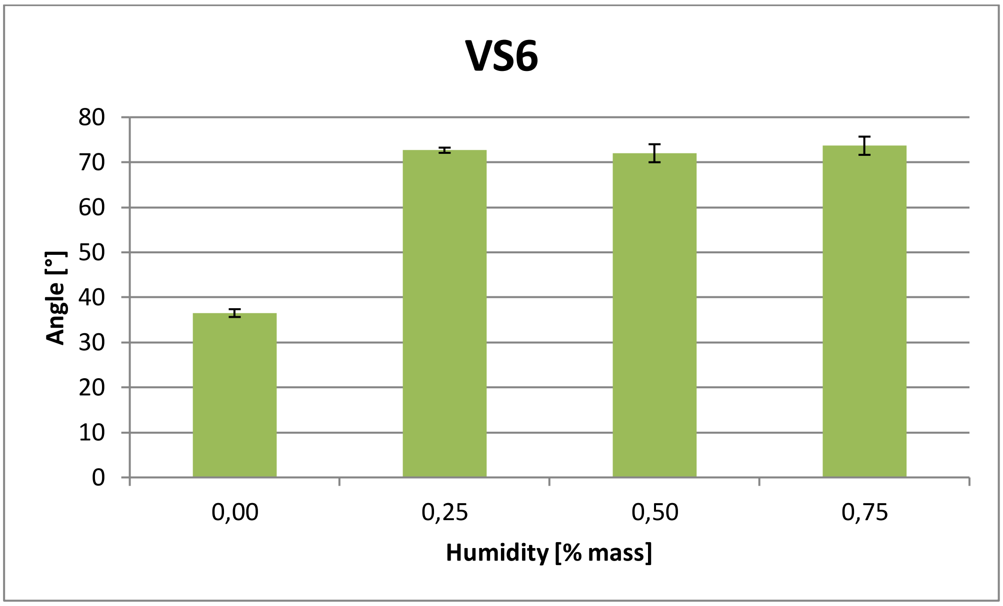


Figure 37: Friciton angle results for VS6

Extra tests on sample VS6 showed no big difference, in the results, even if the sample was not dried in the drying cabinet. This discarded the possibility of the anticaking agent disappearing during the drying process. These results are presented in the following graphs.

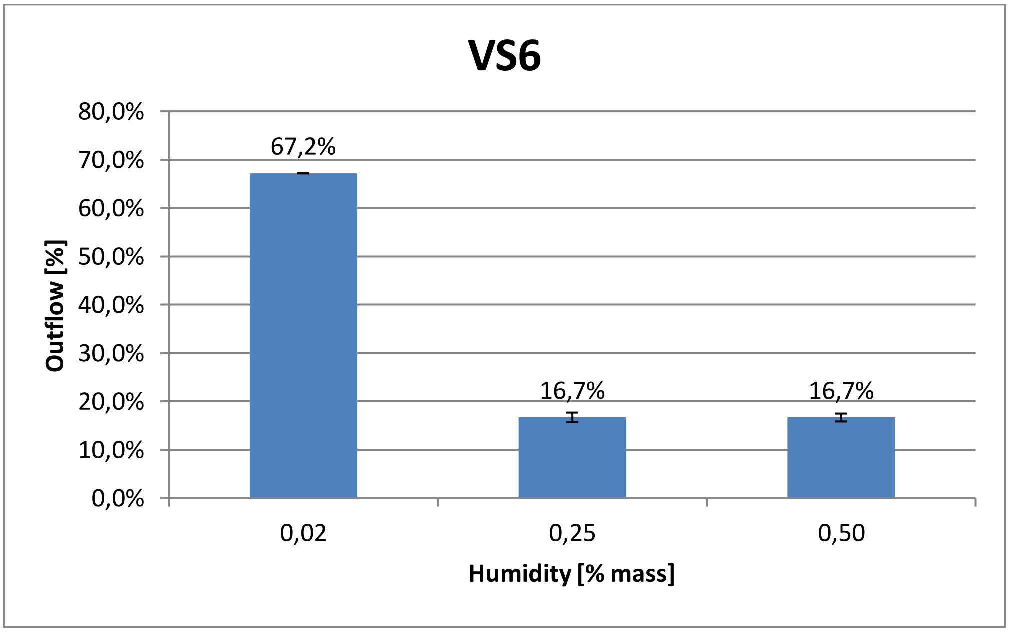


Figure 38: Outflow ratio results for VS6 - not dried samples

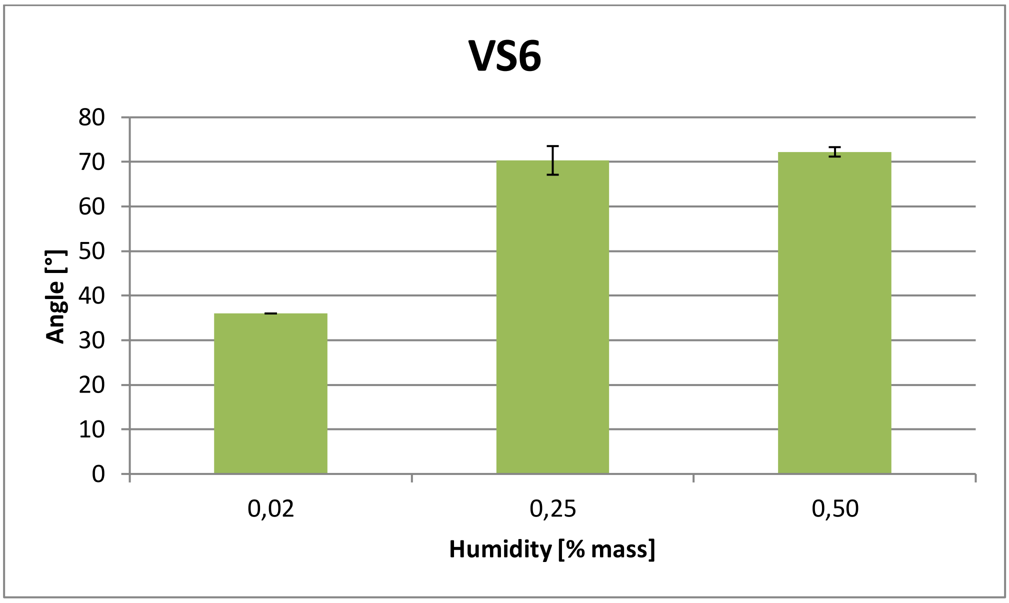


Figure 39: Friction angle results for VS6 - not dried samples

Another factor considered to have had influence on the tests results was the time of the sample after being moistened. To check if it may have had influence it was decided to let the sample in the tray for 2 hours after being moistened and afterwards tested.

The differences of results show that for a low humidity level of 0,25% if the sample is let 2 hours the results vary, and the effect of the anticaking agent is similar to those from samples containing Sodium ferrocyanide in 8 mg/kg concentration. But for a higher humidity of 0,5% the time lapse seemed not to have any influence on the results.

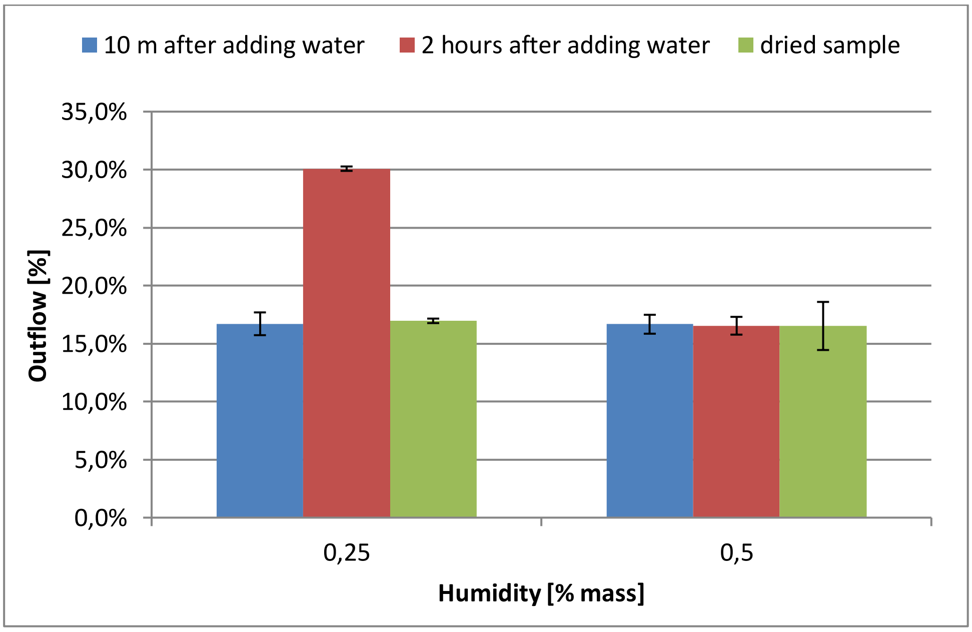


Figure 40: Comparison of average results for VS6 depending on average time of moist samples in the Auslaufbox

The sample tested back in May was observed in July to see if the moisture level of 0,75% of water had caused any caking. It was seen that even though it presented a coarser grain appearance, the formation of lumps had not taken place. It can be seen in the following image.

Figure 41. Sample VS6 one month and a half after being tested.

5.3.11 VS7 – Vacuum salt with 22 mg/kg SFC

This sample is a vacuum salt containing an anticaking agent sodium ferrocyanide in 22 mg/kg. The humidity at which it was delivered was 0,07% in mass. Its concentration of sodium ferrocyanide is higher than the commercial concentrations for vacuum salt, surpassing the maximum 10 mg/kg established by Austrian legislation.

The results yielded show a better performance in terms of pourability, but very irregular, as the standard deviation for outflow ratio with a 0,25% humidity in mass reaches 15,9% and was determined by 5 tests.

The results don't go under 30% of outflow ratio for a 0,75% humidity level, which is a better performance than the rest of Vacuum salt samples.

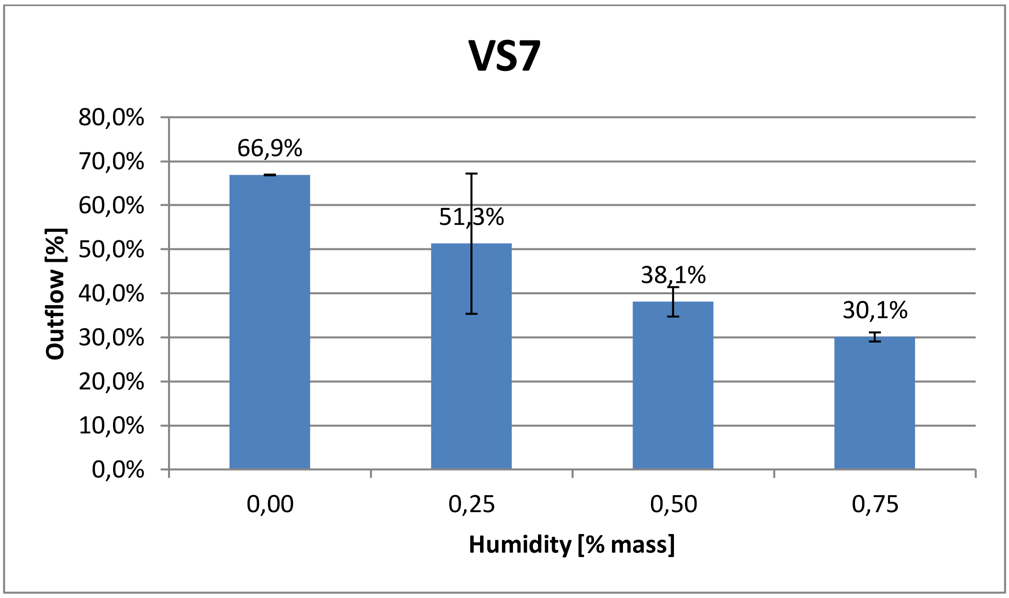


Figure 42: Outflow ratio results for VS7

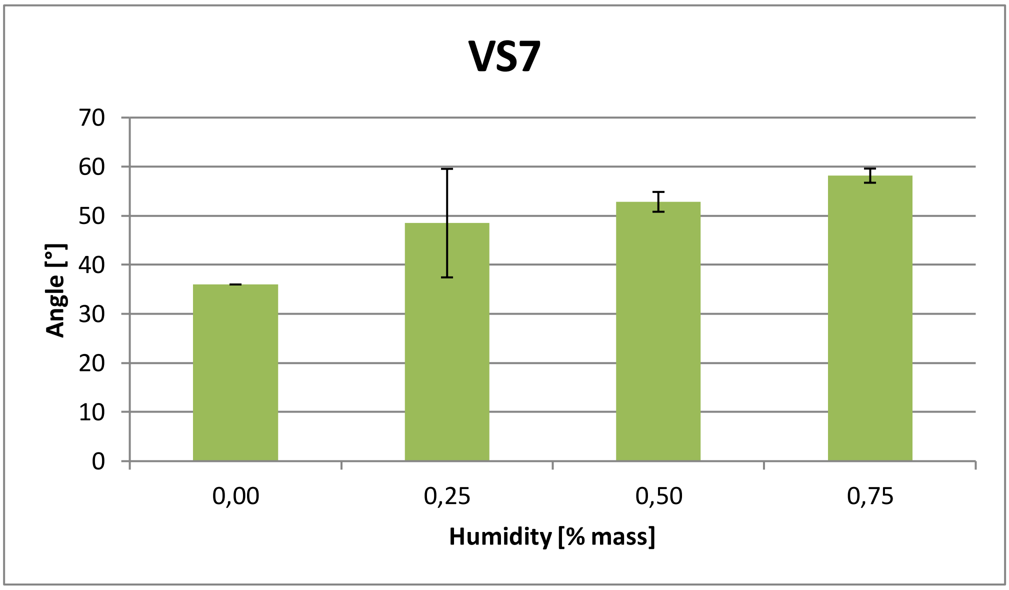


Figure 43: Friction Angle results for VS7

5.3.12 VS8 – Vacuum salt with 95 mg/kg SFC

Sample VS8 is a vacuum salt containing 95 mg/kg of anti-caking agent sodium ferrocyanide. This level is way above the legal level on Vacuum salt for de-icing purposes. Its results show that the outflow ratio and the friction angle have almost the same result for 0,5% humidity and 0,75% humidity. The

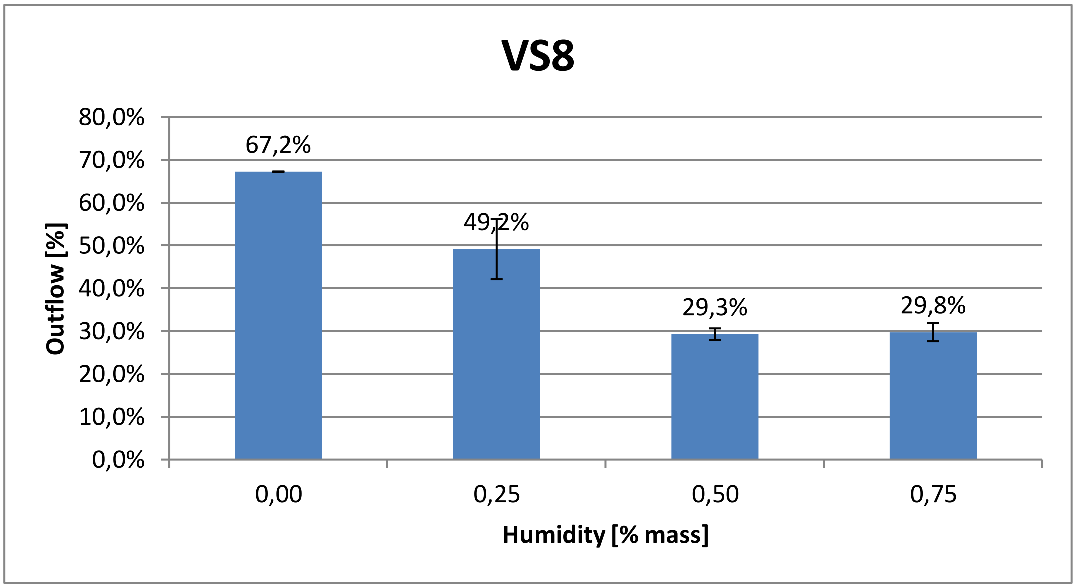


Figure 44: Outflow ratio results for VS8

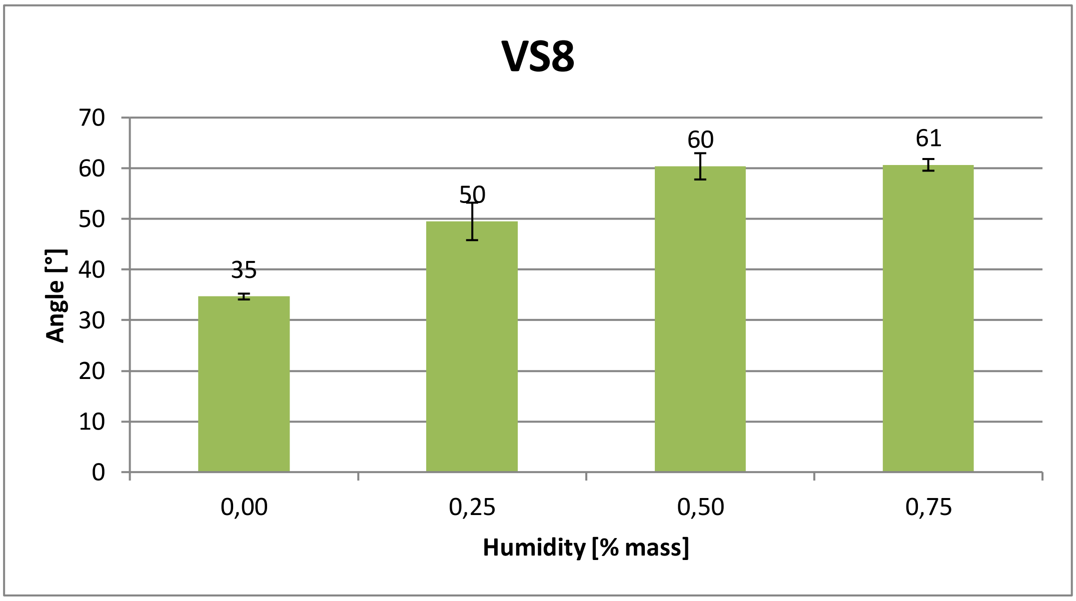


Figure 45: Friction angle results for VS8

5.3.13 Additional overnight tests

The extra tests carried out on different salt samples and different humidity levels yielded different results.

On sample VS3 without any anticaking agent after being left in normal environmental conditions from the laboratory for 19 h the results were no outflow at all, which is different from the results obtained on average for normal tests. For samples VS4 and VS8 the results obtained for the extra tests were slightly worse than the average results, both for the outflow ratio and the friction angle. With differences of 4,3% and 3° VS4 and 2,5% and 4° for sample VS8. This samples contained sodium ferrocyanide anticaking agent on 8 mg/kg and 95 mg/kg respectively.

A totally different behaviour was observed for samples VS7, which had a much better performance than the average normal tests. This may be due to the variability of results of samples containing large amounts of anti-caking agent, also observed on normal tests.

The comparison between the average results for the same humidity with the usual test of the Auslaufbox and the results obtained with the extra test can be seen in the following graphs.

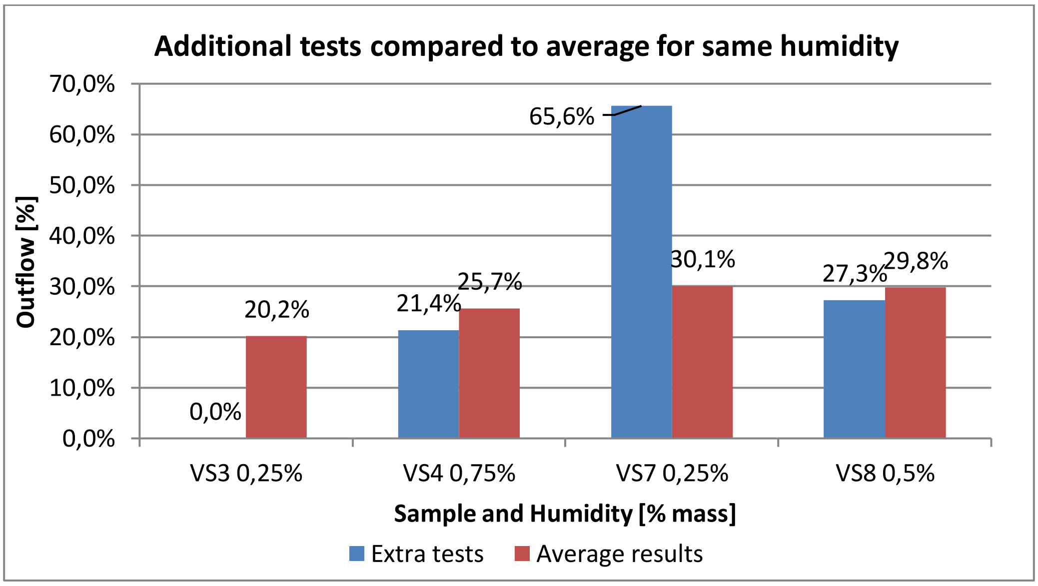


Figure 46: Outflow ratio from additional test with weigh applied compared to main test programme average results

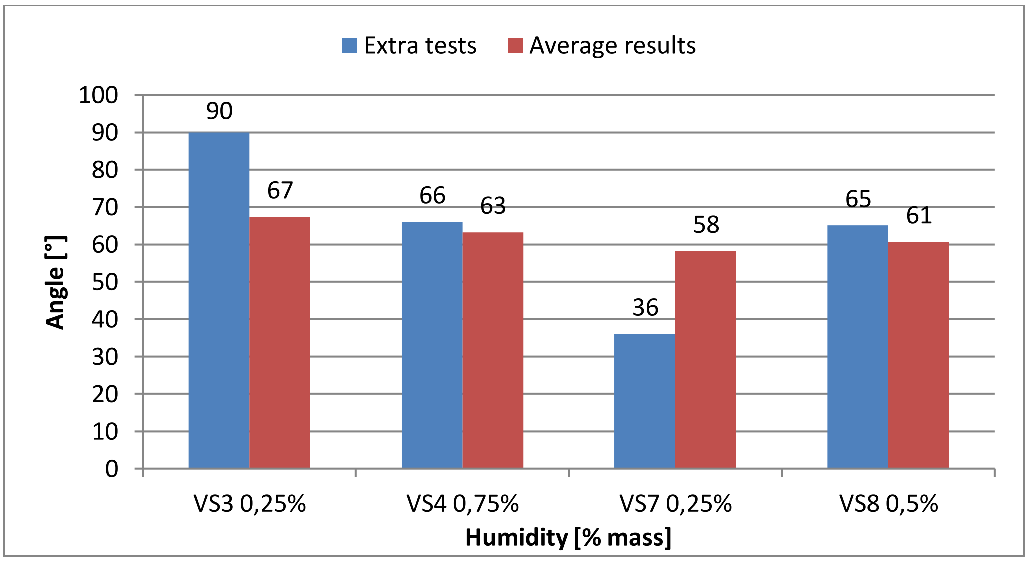


Figure 47: Friction angle from additional test with weigh applied compared to main test programme average results

6 Analysis of the flowability experiments results

The results of the flowability experiments carried out with the 'Auslaufbox nach Sonntag' show the different behaviour of salts depending on if they have or not anti-caking agent. Humidity also plays a big role on the results of the pourability tests, even though there are other factors that have been observed to play a role on the results like grain size, time between moisturizing and test execution and compaction of salt.

Numerical average results for outflow ratio [%] and friction angle are shown in the following tables.

Table 7. Outflow ratio numerical results

Outflow [%]						
Sample	Anti-caking agent [mg/kg]	Grain size [mm]	Humidity level [%]			
			0,00	0,25	0,50	0,75
RS1	0	0/3	62,3%	30,4%	24,1%	14,4%
RS2	0	0/2	60,8%	15,5%	14,2%	14,2%
RS3	70-100	0/3	60,7%	51,1%	42,7%	31,5%
RS4	70-100	0/2	63,3%	39,6%	33,4%	27,5%
VS1	0	fine	62,5%	25,9%	24,9%	17,2%
VS2	0	fine	64,9%	14,5%	15,8%	10,7%
VS3	0	fine	66,2%	20,2%	13,0%	15,8%
VS4	8	fine	65,2%	38,9%	27,5%	25,7%
VS5	8	fine	67,5%	27,7%	24,7%	23,8%
VS6	12	fine	66,2%	17,0%	16,5%	16,6%
VS7	22	fine	66,9%	51,3%	38,1%	30,1%
VS8	95	fine	67,2%	49,2%	29,3%	29,8%

Table 8. Internal Friction angle numerical results

Friction angle [°]						
Sample	Anti-caking agent [mg/kg]	Grain size [mm]	Humidity level [%]			
			0,00	0,25	0,50	0,75
RS1	**0**	**0/3**	41	59	63	74
RS2	**0**	**0/2**	40	67	70	70
RS3	**70-100**	**0/3**	40	47	51	56
RS4	**70-100**	**0/2**	37	55	57	61
VS1	**0**	**fine**	39	62	64	68
VS2	**0**	**fine**	37	74	73	77
VS3	**0**	**fine**	37	67	76	72
VS4	**8**	**fine**	38	55	60	63
VS5	**8**	**fine**	36	60	65	66
VS6	**12**	**fine**	37	73	72	73
VS7	**22**	**fine**	36	49	53	58
VS8	**95**	**fine**	35	50	60	61

As it can be seen from the numerical results, all samples have similar results, for both outflow ratio and friction angle, when the humidity of samples is 0,00%. Rock salts have on average 61,8% of outflow ratio and 39° of friction angle for 0,00% humidity. For Vacuum salts with this humidity level outflow ratio is on average 65,8% and 37° of friction angle.

All the samples have worse flowability performance when in presence of humidity. The effect of the anticaking agent can be observed when the humidity level of samples is increased. This is also visible in the following graphs, divided between rock salt and vacuum salt.

In the following sections, the results obtained from tests with the ‘Auslaufbox nach Sonntag’ are analysed based on plotted graphs.

In section 5.3 graphs show the average obtained from the tests. The standard deviation is plotted as error bars. In each graph, samples are listed with its name, followed by grain size for rock salt and finally the anti-caking agent amount for both kinds of salts.

Linear relationship friction angle and outflow

The results showed there is a linear relationship between the friction angle and the outflow that has been observed in all the samples. It means that both parameters describe the same way flowability, as expected. Yielding always for a 0% outflow a 90° angle. For other outflows the angles may vary, but results clearly form a lineal point cloud. This is seen in the following graph, elaborated with the results obtained from all tests.

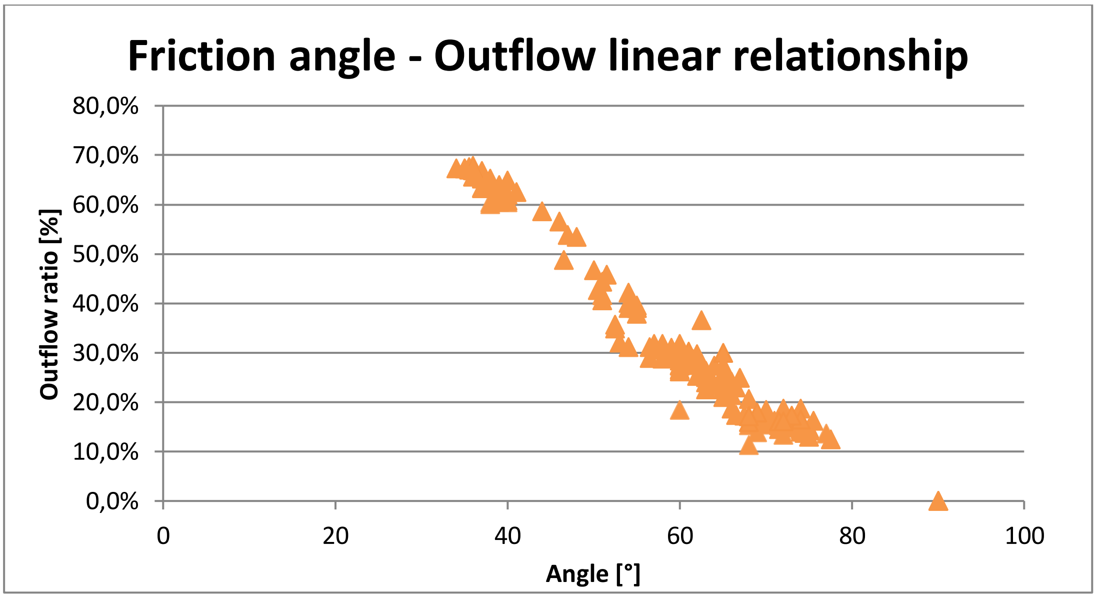


Figure 48: Friction angle - Outflow ratio

On average, for all cases lower friction angle means higher discharge of the Auslaufbox, and this is confirmed by the results obtained. Although there are some little variations, in rock salts for example, due to the different grain sizes of the samples.

For this reason, commenting on outflow ratio and friction angle obtained from the 'Auslaufbox nach Sonntag' test is the same after all.

Rock salt results analysis

The average results and standard deviation from tests for Vacuum salts are presented in the following graphs.

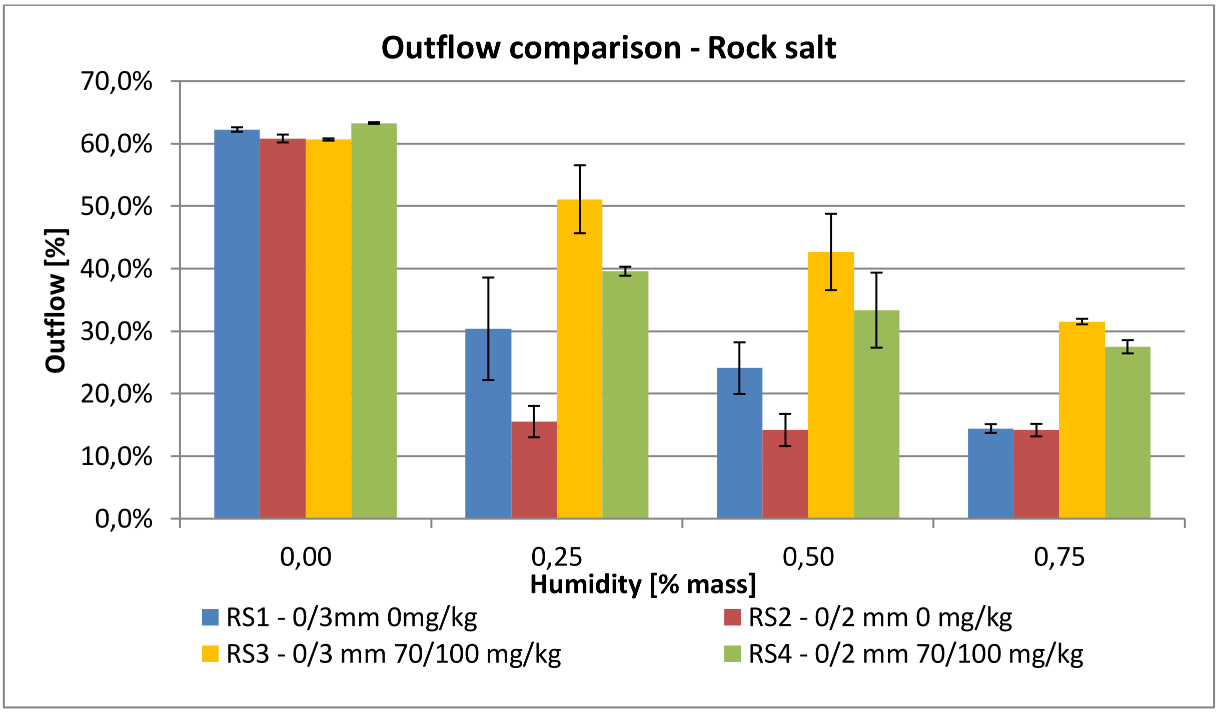


Figure 49: Outflow ratio results from Rock salt

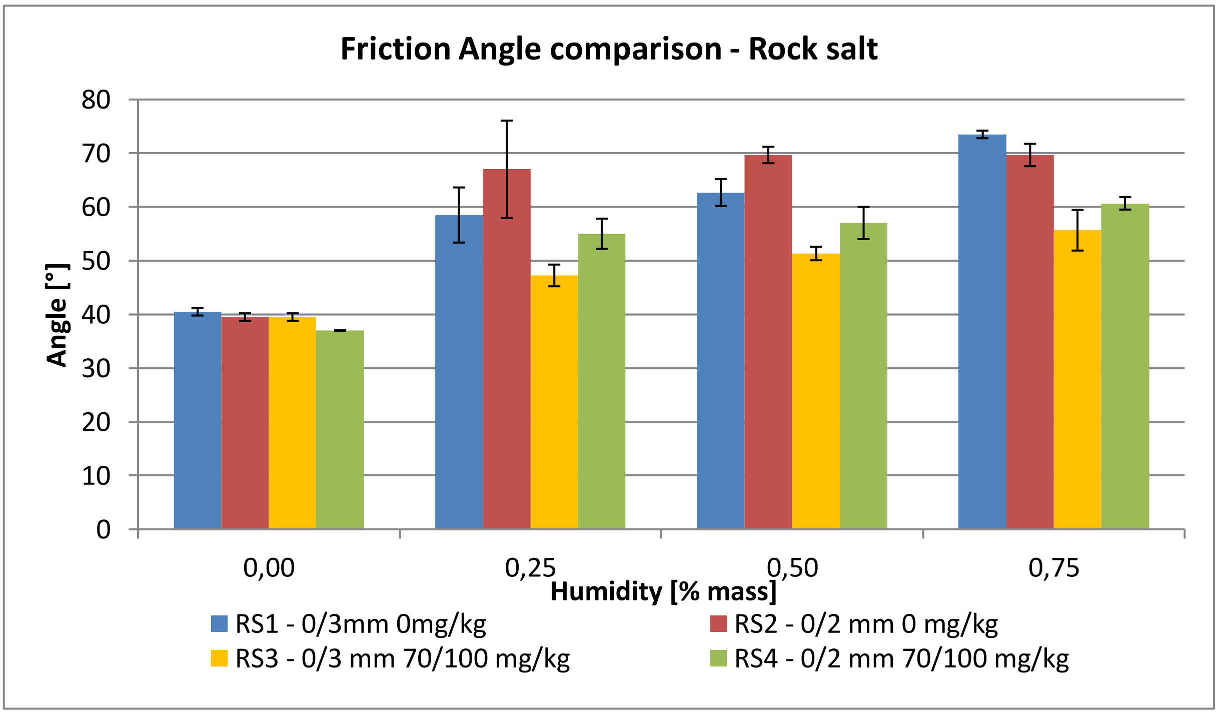


Figure 50: Friction angle results from rock salt

As it can be seen for rock salt, the behavior of salts containing anticaking agent is different from those samples without anticaking agent. Samples containing anti-caking agent (RS3 and RS4) show a better flowability by having a higher outflow rate and inferior angles than samples without anti-caking agent (RS1 and RS2) as the samples are exposed to higher humidity levels.

Grain size affects also the outflow ratio and friction angle results. Grain size salts of 0/2 mm (RS2 and RS4) have lower outflow and higher friction angle than those with grain size 0/3 mm (RS1 and RS3).

It is also clear that the standard deviation increases as water is added to the samples, for both outflow ratio and friction angle and for a humidity of 0,75% it lessens in the case of outflow ratio.

Vacuum salt results analysis

The average results and standard deviation from tests for Vacuum salts are presented in the following graphs.

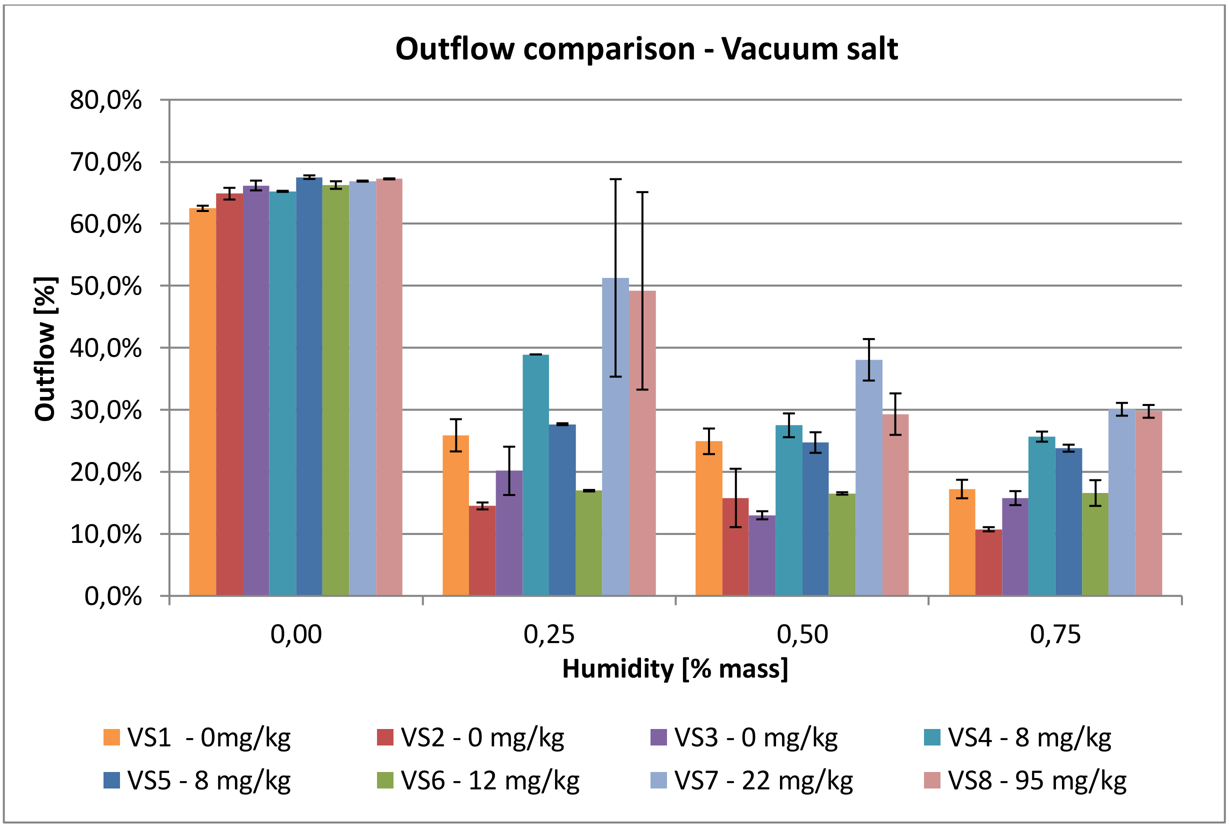


Figure 51: Outflow ratio results comparison for Vacuum salt

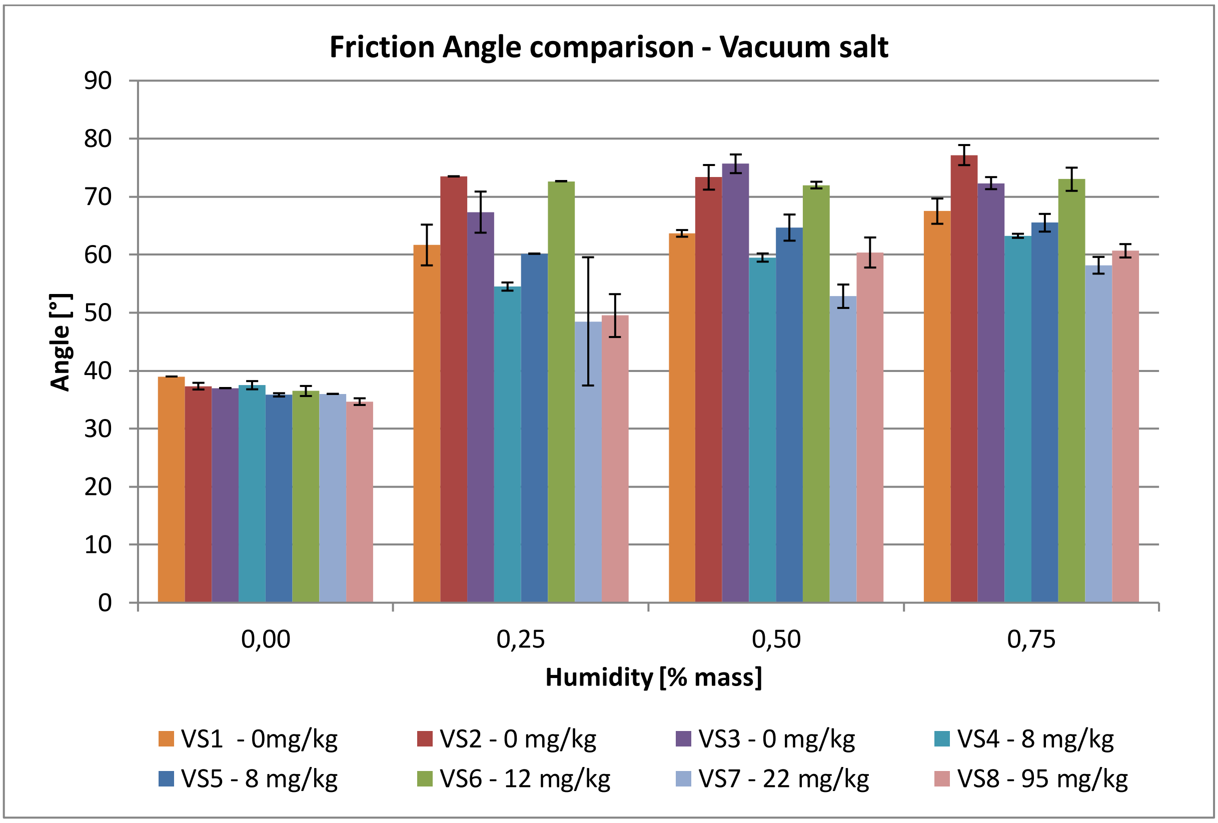


Figure 52: Friction angle comparison for vacuum salt

For Vacuum salt, in general results between different samples do show clearly different behavior depending on the kind of anticaking agent used and its amount. There are also some remarkable differences between salts that are *a priori* the same type and contain the same amount of anti-caking agent. For instance, sample VS1 despite not having anticaking agent has a similar behavior to samples containing ferrocyanide (VS4 and VS5) and even better than the sample containing biodegradable anti-caking agent. It clearly has different results than those of samples VS2 and VS3, that do not contain any agent either. This can be due to the fact that the sample was strongly caked, and had to be sieved and broken to be able to test it with the 'Auslaufbox'.

Sample VS6, containing biodegradable anti-caking agent in a concentration of 12mg/kg, expressed as concentration of iron ion, has a performance similar to sample VS2, that does not contain anti-caking agent, and was not exposed to high humidity levels prior to the tests. Additional tests performed on the sample VS6 without having been dried, showed that the drying process did not affect the sample test.

The results showed, that more than not working as an anticaking agent, this biodegradable agent may work in a different way than sodium FC on a microscopic scale, and that results from tests were affected by the time passed between the adding of moisture and the tests being carried out as explained on page 58.

VS4 and VS5, both containing 8 mg/kg of anticaking agent have similar results, and both better than sample VS6 with Bio anticaking agent. Meanwhile samples VS7 and VS8 (containing the highest anti-caking agents amounts) yielded irregular results, having the highest standard deviations from all samples.

Salts without anticaking agent

Graphs with the average results for samples VS1, VS2, VS3, RS1 and RS2 that do not contain anticaking agent are presented and discussed below.

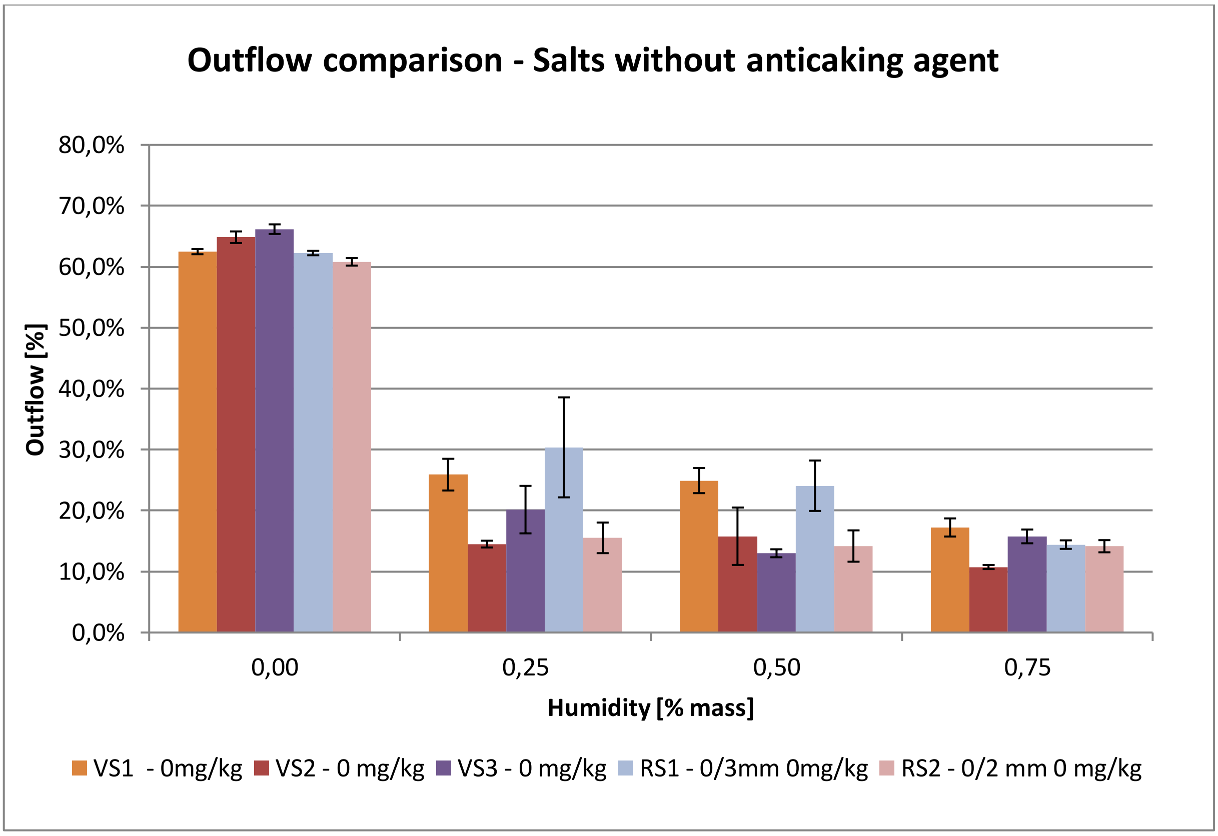


Figure 53: Outflow ratio comparison for salts without anticaking agent

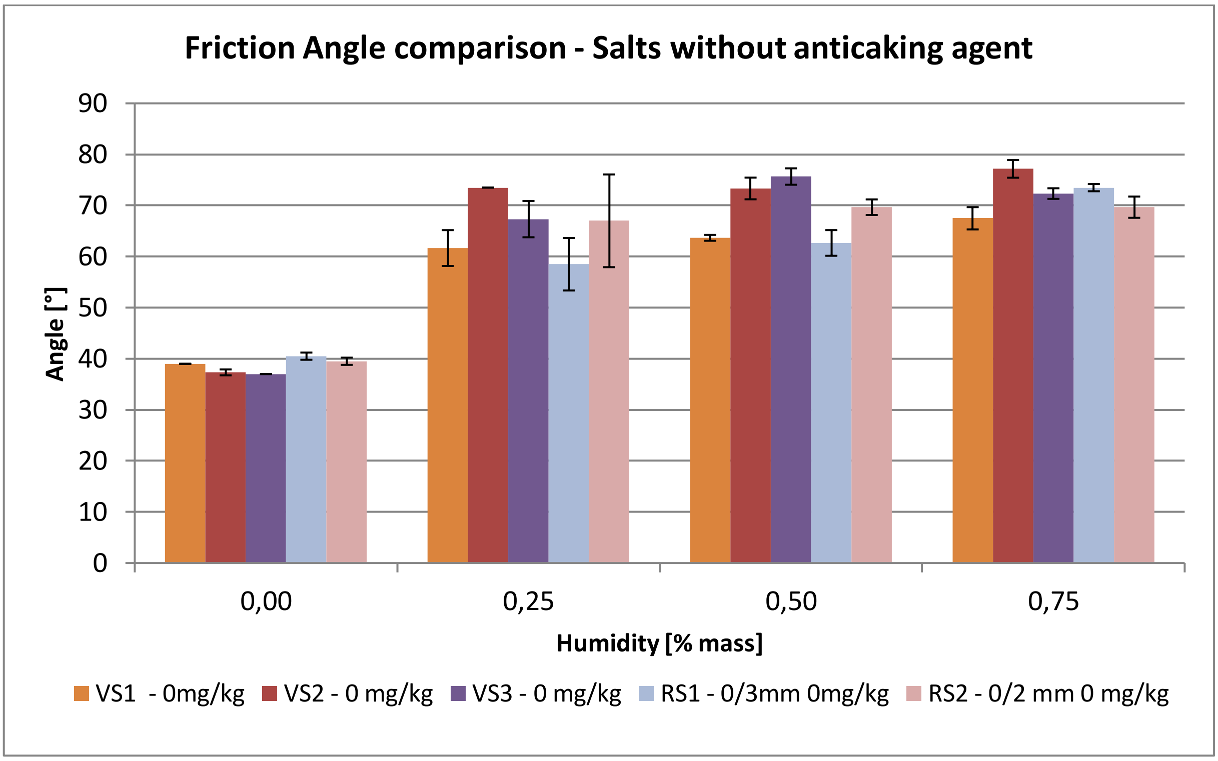


Figure 54: Friction angle results comparison for salts without anticaking agent

As it can be observed for VS without anticaking agent, sample VS1 that was caked and needed to be sieved has a better flowability performance in presence of humidity, yielding in all cases the highest outflow ratio and the lowest friction angle between all VS samples.

VS2 and VS3 have a similar behaviour, even though it is variable which one has better results depending on the humidity level, they both distinguish from sample VS1. These results may be interesting, to see the effects of breaking the crystals bridges of lumps, once they have been formed due to a high dampness of the salt, as it is 1,2% in mass.

If compared with rock salt samples, vacuum salt samples have a similar behaviour to sample RS2, that has a finer grain size. For humidities of 0,25 and 0,50 % sample RS1 shows on average a better performance, although the standard deviation is also greater than for other samples.

It can also be noted that there are practically no differences between salt types when the samples are dry, all results are nearly the same. There are obvious differences for humidities of 0.25% and 0.50%. But these differences diminish as moisture increases, and for 0,75 humidity there are not remarkable differences between rock salt and vacuum salt based on the obtained results.

Standard deviation of results also behaves similarly in both types of salt, and it increases for humidities of 0,25% and 0,50% and decreases for 0,75% humidity in general.

Salts with anticaking agent

Graphs with the average results for samples of VS4, VS5, VS6, VS7, VS8 and RS7 and RS8 that contain anticaking agent in different concentrations are presented and discussed following.

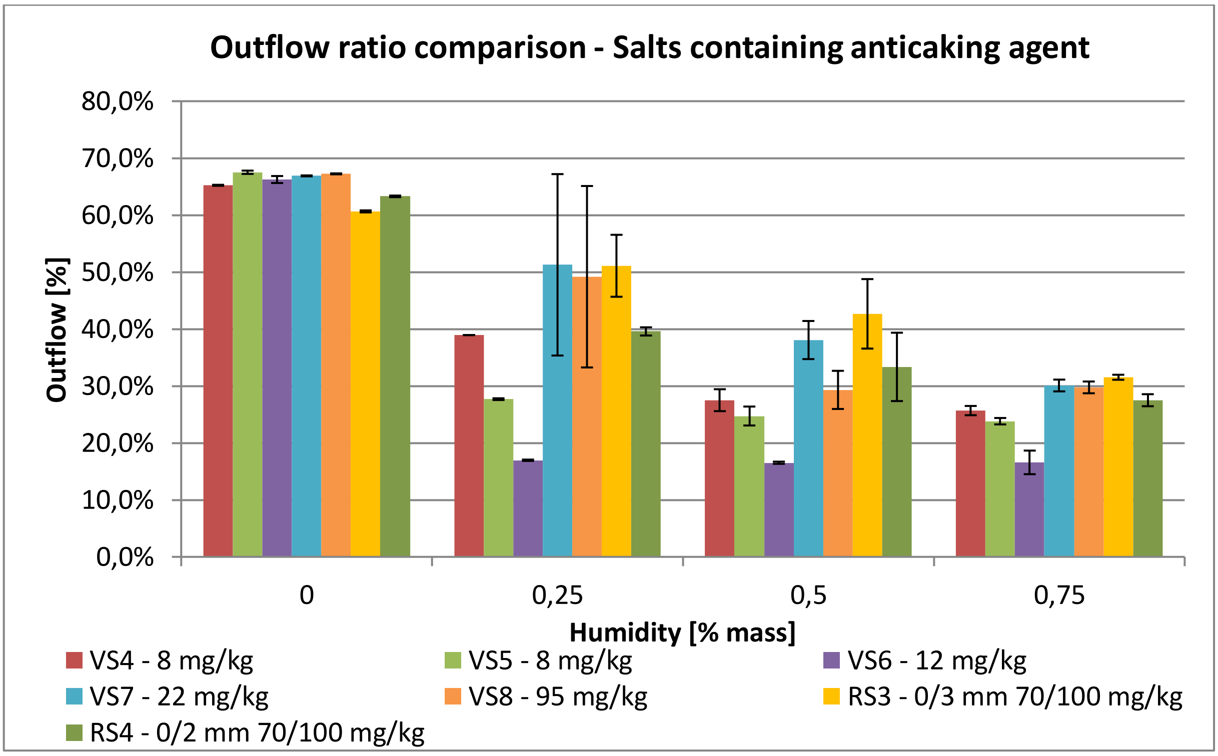


Figure 55: Outflow ratio results comparison for salts containing anticaking agent

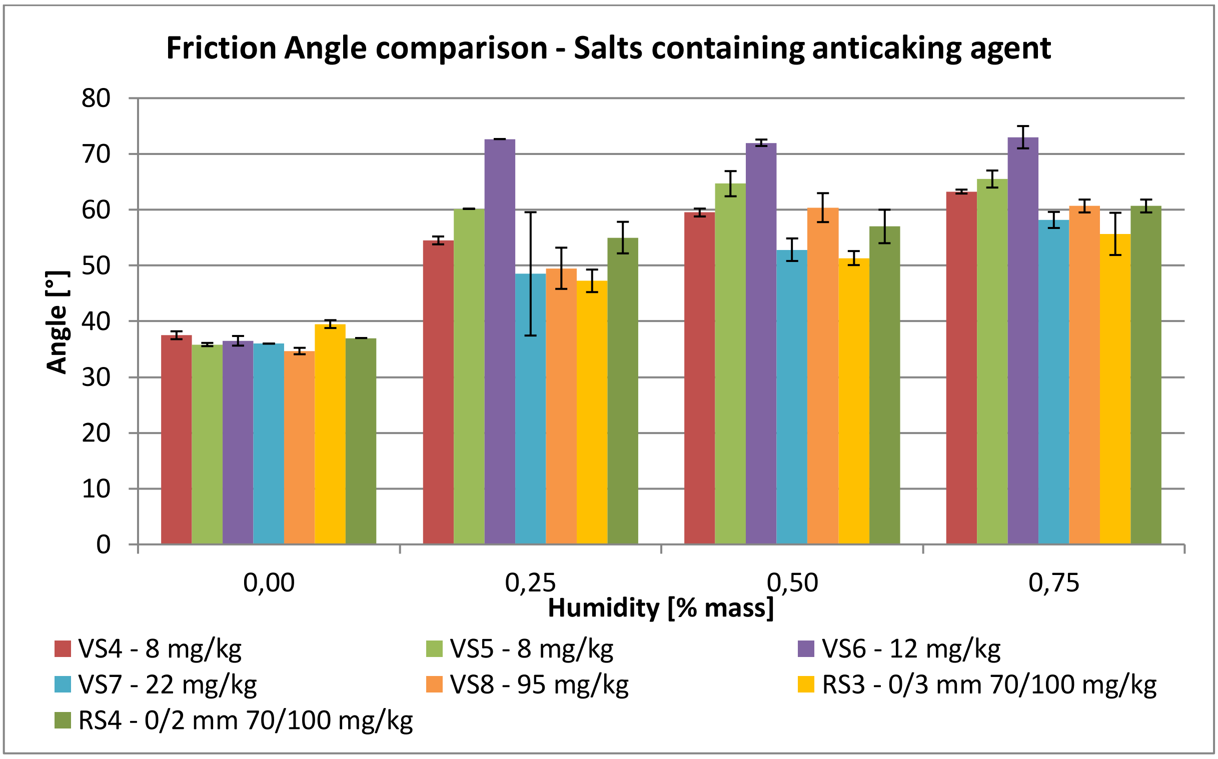


Figure 56: Friction angle result comparison for samples containing anticaking agent

For samples containing anticaking agents, results from VS4 and VS5, show that despite having the same characteristics, same anticaking agent quantity of 8 mg/kg, the discharge of the VS5 is a bit higher for a 0,00% humidity level, and a little bit lower when there is humidity. This may be due to a slightly finer average grain size of sample VS5. This is also observed with the rock salt, since bigger grain size sample RS3 yields a larger outflow ratio than sample RS4. The opposite happens with the friction angle, as they are inversely proportional.

For sample VS6 tests do not yield similar results compared to samples containing anticaking agent. Outflow ratio and friction angle are comparable to results obtained for samples with no anticaking agent. As outflow ratio and friction angle are similar to all other samples for 0,00% humidity, but for a humidity of 0,25% outflow decreases to 17% and angle increases to 73° and they remain in these values for 0,50% and 0,75% humidities. In a similar way, as tested samples with no anticaking agent do. This may be due to the fact that biodegradable anticaking agent may work differently than sodium ferrocyanide at a microscopic scale, so the test results do not show similar flowability behaviour. Nevertheless, this does not mean that it is not an effective agent, as the sample was not observed to cake after a month and in references this biodegradable agent is also confirmed to keep salt lump free [7]. When tested for a longer period in the box it showed it tended to have better results at a humidity of 0,25%.

Samples containing larger quantities than allowed by Austrian regulations VS7 and VS8, show a better flowability performance on average, but it is not proportional to the amount of anticaking agent contained. It should also be noted that VS8 containing 95 mg/kg of anticaking agent sodium FC, has worse results than sample VS7, containing 22 mg/kg of the same agent. They also have noticeably higher standard deviations than other samples, due to the largely variable results during the tests, especially for a humidity of 0,25%.

If rock salt and Vacuum salt are compared, despite the large quantity of anticaking agent they contain 70 to 100 mg/kg, their results are not so far away from samples containing sodium FC in minor concentrations as VS4 and VS5 in 8 mg/kg.

It can also be noted that differences between results from rock salt due to grain size are not so different from those between samples with the same characteristics in vacuum salt and same sodium FC amount. For example, samples VS4 and VS5. For a 0,25% humidity samples VS4 and VS5 had an average outflow of 38,9% and 27,7% respectively while RS3 – 0/3mm had an average outflow of 51,1% and RS4 – 0/2 mm of 39,6%. While differences in grain size for Rock salt could be noticed at first sight, it was not the case for vacuum salt, and both samples were tagged as fine grain.

It should be added that the differences due to the grain size of the rock salt decrease when the humidity increases. And this also occurs in the vacuum salt samples, as can be seen in the graphs above.

Bulk density of salt respect humidity.

Another aspect observed from the data obtained with the 'Auslaufbox' is that bulk density decreased almost linearly for all the samples, with coefficient of determination R^2 ranging 0,8 to 0,9. This can be observed in the following graphs plotting the total mass in the Auslaufbox for all the tests and the humidity ranging 0,00 to 0,75%.

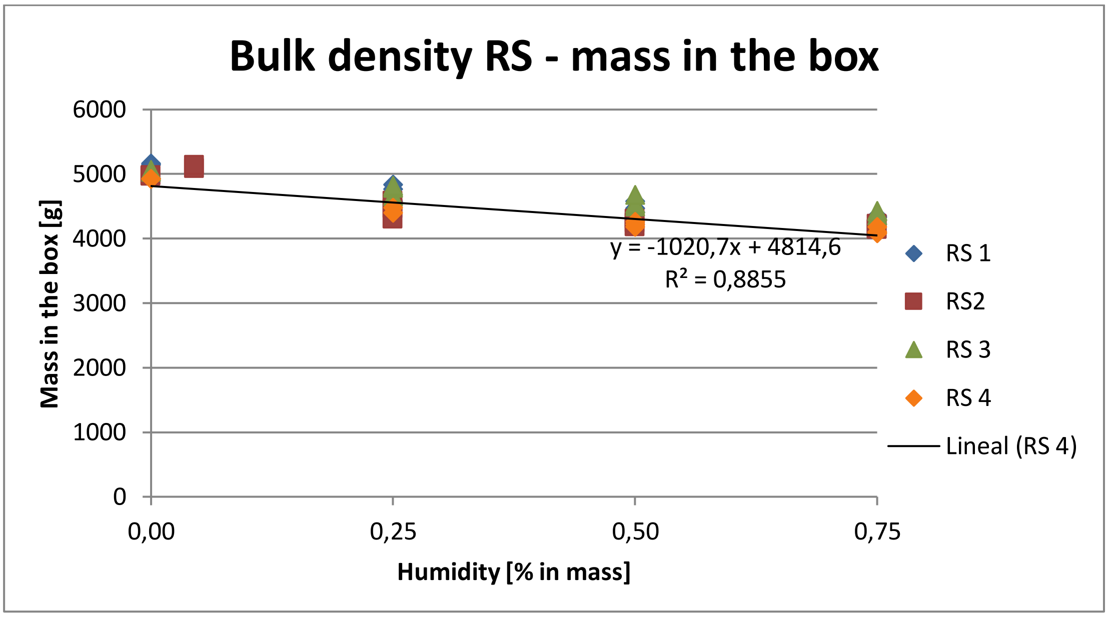


Figure 57: Bulk density results from rock salt

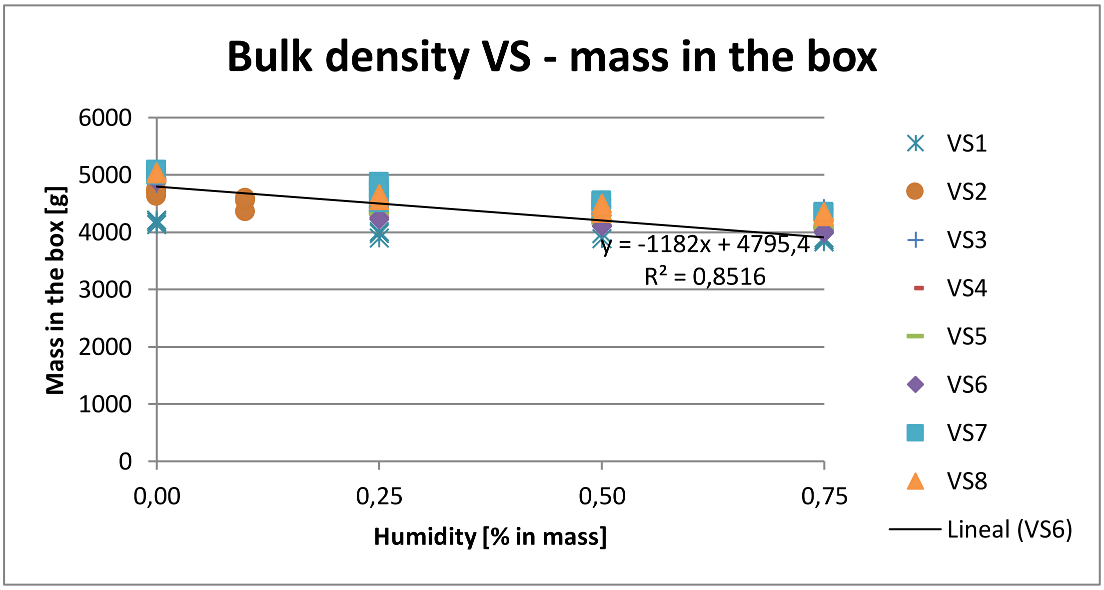


Figure 58: Bulk density results from vacuum salt

Further tests

Following graphs are plotted based on the average outflow ratio of tested samples divided by type, grain size and humidity tested, depending on the anticaking agent quantity of the salt. This is done in order to determine whether an optimum anticaking agent quantity could be found in further tests for VS and RS.

Rock salt

To plot these charts, since the samples of rock salt containing sodium FC do not have an exact amount (it is labelled between 70 and 100 mg/kg sodium FC), the results are plotted for the middle value of 85 mg/kg SFC.

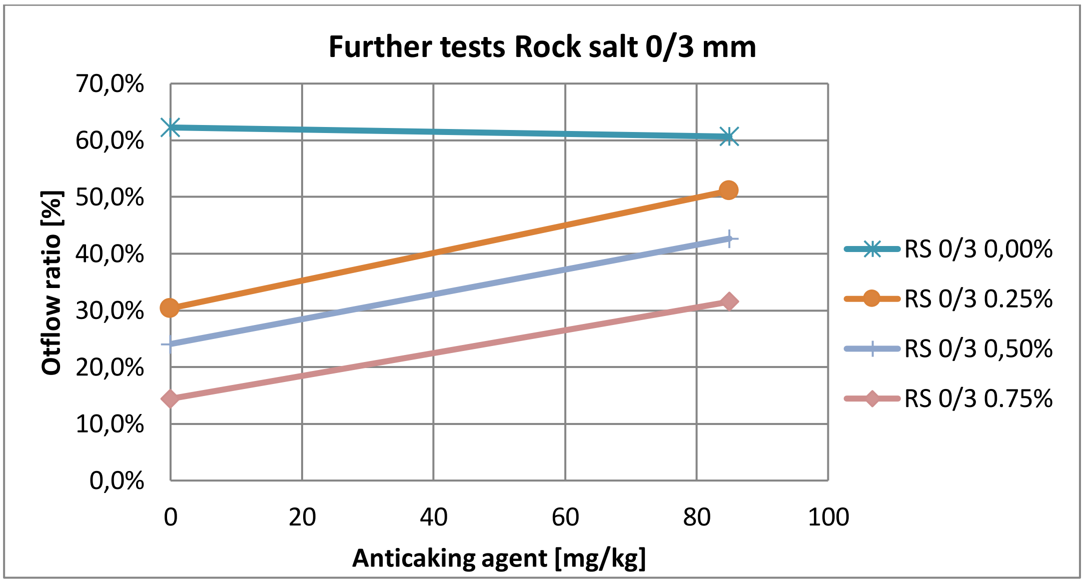


Figure 59: Outflow ratio for RS 0/3 mm depending on the amount of SFC for humidities 0,00 %; 0,25 %; 0,50% and 0,75%

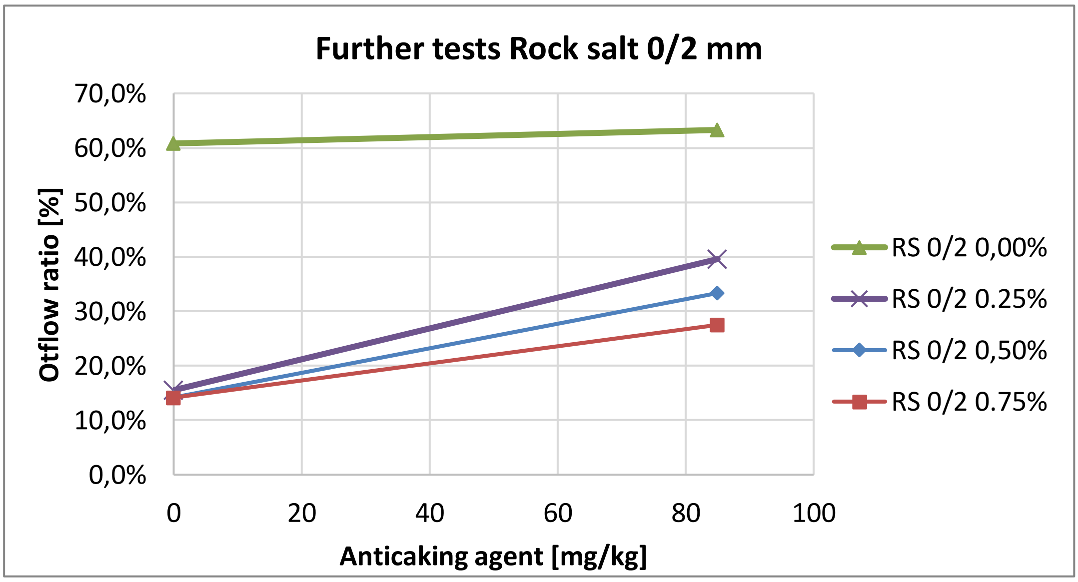


Figure 60: Outflow for RS 0/2 mm depending on the amount of SFC for humidities 0,00 %; 0,25 %; 0,50% and 0,75%

In the case of Rock salt, it can be seen once again the effect of grain size. For a grain size 0/3 mm and no anticaking agent, outflow ratio progressively decreases with the increase of humidity. Nevertheless, for the grain size of 0/2 mm outflow ratio drops from 60,8% to

15,5% for a 0,25% humidity and is maintained approximately at this value for greater humidities of 0,50% and 0,75%.

It is clear from the plots that salts with concentrations of sodium ferrocyanide between 0 and 70 mg/kg should be tested in order to determine a real curve for rock salt for both grain sizes.

It would be also interesting to test samples of salt containing larger amounts of SFC, considering that, according to the Austrian guidelines it is allowed up to 200 mg/kg for rock salt. This could would allow to see what the optimal concentration is and if the level of Austrian regulations is too high. Considering that smaller amounts are better for the environment and they could have the same effect on the pourability of salt.

<u>Vacuum salt</u>

To plot the averages for vacuum salt, the data taken for 0 mg/kg of anticaking agent are obtained only from VS2 and VS3, since VS1 has a marked different behaviour presumably due to strong caking before the test. For 8 mg/kg outflow ratios averages averages are obtained from samples VS4 and VS5. For 22 mg/kg belong to the average results from sample VS7 and for 95 mg/kg from VS8. For 12 mg/kg it is about the sample VS6 with biodegradable anti-caking agent. For this reason, this last sample is plotted outside the lines.

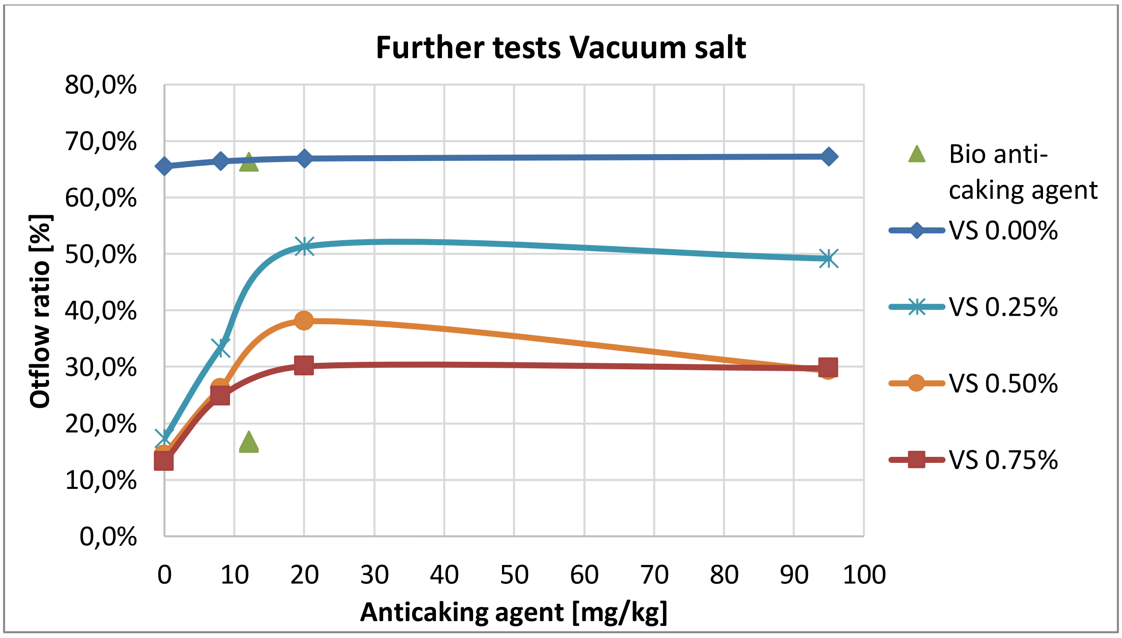


Figure 61: Outflow ratio for VS depending on the amount of anti-caking agent for humidities 0,00 %; 0,25 %; 0,50% and 0,75%

As it can be seen, for all the different anticaking agents with dry conditions, outflow ratios are around 66%. When salt is moistened, the effect of anti-caking agent is perceptible in the outflow ratios for sodium ferrocyanide. In the case of biodegradable agent, it has the same behavior as it contained no agent, based on the outflow ratio average results. Since for all humidities, average outflow ratios are about 17%.

Based on the results, it can be seen that samples containing 22 mg/kg SFC have a clearly better pourability performance than samples containg 8 mg/kg. Taking into account that Austrian guidelines have set the limit for vacuum salt in 10 mg/kg SFC it would be interesting to test if with this amount of anti-caking agent, the problems that arise during vacuum salt storage could be avoided. Thereby check whether an increase in the anti-caking limit would make sense.

7 Summary and recommendations

Winter maintenance must ensure safety in the road network, and in Austria this is of great importance due to long period over the year that it is necessary. Thus, having a large economic and environmental cost, in Austria efforts are made continously in research to optimise every aspect related to winter road maintenance.

This Master thesis is framed in the context of a basic element for winter road maintenance such as de-icing salt and its characteristics related to the anti-caking agent and the moisture it contains.

The results obtained with the 'Auslaufbox nach Sonntag' yield results that are coherent with the anticaking agent content and the humidity levels. Even though the variances between results from different samples are not so diverse.

In general, the addition of water has an immediate influence on the test results, that show how the outflow ratio drops and the friction angle increases for all samples, and this is usually proportional to the anti-caking agent content of the salt.

This test has shown that it is valid for the study of flowability of salt. But it should be noted, that for salts, how the tests are carried out can have a notable influence on the results. The filling of the Auslaufbox was made always with a funnel in order not to compact the sample inside the apparatus. Time between moisture addition and tests was proved to have influence on results. Therefore, it was stablished that samples when humid should be in the Auslaufbox from 5 to 10 minutes. This was not enough for samples containing biodegradable anticaking agent, that according to the outflow ratio and friction angle, failed to confirm the effects of the agent for tests being carried out this way.

Based the results, the biodegradable anti-caking agent does not have a perceptible effect on NaCl, as sodium FC has in general for all samples containing it. Furthermore, the yielded results resemble results from salts without anti-caking agent. It was firstly thought that the drying process may have had influence in the flowability tests, but this was subsequently discarded by additional tests performed on samples VS6.

It is believed that biodegradable agent works probably in a different way than sodium FC at a microscopic level, and therefore the results are different. During the time in the laboratory, it was not observed that samples containing the biodegradable agent would form lumps. In further tests, it would be interesting to assay samples with larger quantities than 12 mg/kg. This would allow to check if salt containing this agent would have tangible results in the 'Auslaufbox nach Sonntag' tests.

The results obtained for amounts of sodium FC 22 mg/kg and 95 mg/kg, greater than allowed by Austrian regulations for vacuum salt (10 mg/kg) confirm that its use is not justified in the case of 95 mg/kg. It yielded variable results and the performance of this samples was not proportional to the greater amounts of sodium FC and did not show big difference from 22 mg/kg to 95 mg/kg. Although It is remarkable that they both showed better flowability on average than the samples containing 8 mg/kg.

For vacuum salt it is seen that the best flowability was achieved by 22 mg/kg of SFC, having results almost as good as rock salt 0/3 mm with 70-100 mg/kg. This leads to think that a limit of anti-caking agent set on 20 mg/kg instead of 10 mg/kg for vacuum salt in the Austrian guidelines could make sense.

Results for rock salt show the effect of sodium FC respect the same sample not containing it. It was only possible to test one samples without anti-caking agent and one sample with sodium FC in a concentration between 70 and 100 mg/kg in two grain sizes 0/2 mm and 0/3 mm. It would be interesting for further tests to have different amounts of anti-caking agent and try to obtain an outflow ratio curve with respect to the concentration of anti-caking agent that allows to determine which would be an optimum concentration for each grain size tested. Thus, one could check whether the limit established by the Austrian regulations for rock salt of 200 mg/kg is an overstated value or not, in order to preserve the fluidity of the salt. So far, it can be concluded that the best pourability performance of all was achieved by rock salt 0/3 mm with 70-100 mg/kg SFC. Which could presumably be improved by a larger amount of anti-caking agent of approximately of 150 mg/kg.

Grain size has also been found to be a factor affecting the results of the tests, being samples with finer grain more susceptible to moisture due to a greater specific surface. Resulting into a worse flowability performance when moistened, despite having a slightly better performance when completely dry. This has been seen in Rock salts and presumably also in Vacuum salts.

It was also found that the bulk density of salt measured with the Auslaufbox data could be related linearly to the humidity in mass of the salt with a specific linear regression with a coefficient of determination R^2 ranging 0,8 to 0,9 for each sample tested. This could have a useful application, which would allow to measure approximately in a quick and simple way the humidity of the salt.

As a conclusion, ‘Auslaufbox’ test has been proved to be a good method to test flowability of salt with practical purposes, as advantages are the easy way of carrying the tests out, and no need for special instrumentation further than the ‘Auslaufbox’, a tray and a scale.

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II. List of figures

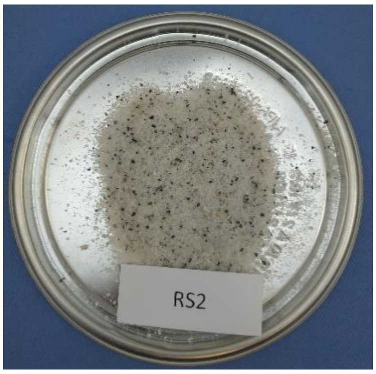
III. List of tables

IV. Photographic Annex

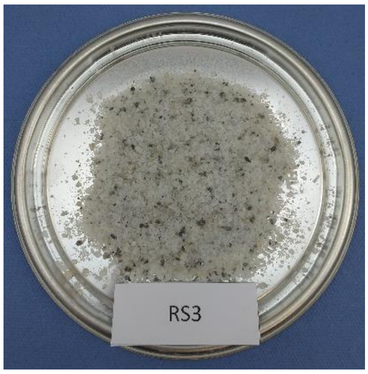
Pictures of the samples:

In the comments, the information of the salt sample is explained as shown below:
Sample name: Grain size, anticaking agent content.

RS1: 0/3 mm, no anticaking



RS2: 0/2 mm, no anticaking



RS3: 0/3 mm, 70-100 mg/kg sodium ferrocyanide

RS4: 0/2 mm, 70-100 mg/kg sodium ferrocyanide

VS1: fine, no anticaking

VS2: fine, no anticaking

VS3: fine, no anticaking

VS4: fine, 8 mg/kg Sodium ferrocyanide

VS5: fine, 8 mg/kg Sodium ferrocyanide

VS6: fine, 12 mg/kg Biodegradable anticaking agent

VS7: fine, 22 mg/kg Sodium ferrocyanide

VS8: fine, 95 mg/kg Sodium ferrocyanide

Storage of samples in the laboratory:

Sample VS7

Samples RS1, RS2, RS3 and RS4

Samples VS2, VS3, VS4, VS6 and VS8

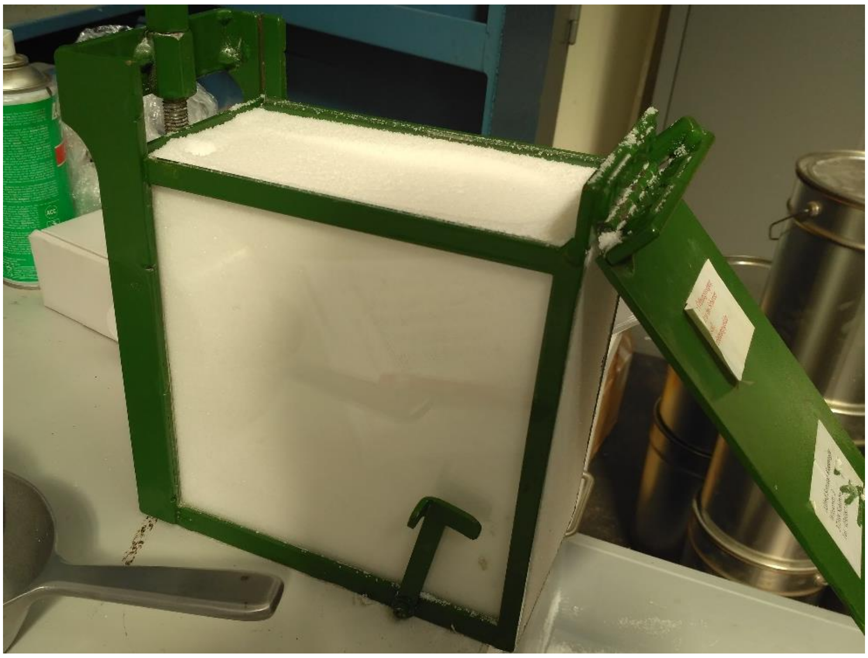
Sample VS1 and VS5

Storage of samples after the tests:

Auslauf box during a test:

Filling of the Auslaufbox:


Auslaufbox when there is 0% outflow ratio:



V. Test results

RS1 – Rock Salt 0/3 mm and no anti-caking agent

<table>
<tr><th>Sample Name</th><th>Humidity (%)</th><th>Total mass(g)</th><th>Discharged mass(g)</th><th>Mass in Box(g):</th><th>Discharge in %:</th><th>Standard dev.</th><th>Average (%)</th><th>Angle (°)</th><th>Standard dev.</th><th>Average (%)</th><th>Time</th></tr>
<tr><td>RS1</td><td>0,00</td><td>5168,6</td><td>3230,8</td><td>1937,8</td><td>62,5%</td><td rowspan="3">0,4%</td><td rowspan="3">62,3%</td><td>41</td><td rowspan="3">1</td><td rowspan="3">41</td><td>10 m</td></tr>
<tr><td>RS 1</td><td>0,00</td><td>5143,2</td><td>3188,9</td><td>1954,3</td><td>62,0%</td><td>40</td><td>10 m</td></tr>
<tr><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td>RS 1</td><td>0,25</td><td>4583,6</td><td>1035,2</td><td>3548,4</td><td>22,6%</td><td rowspan="3">8,2%</td><td rowspan="3">30,4%</td><td>65</td><td rowspan="3">5</td><td rowspan="3">59</td><td>10 m</td></tr>
<tr><td>RS 1</td><td>0,25</td><td>4773,6</td><td>1858,7</td><td>2914,9</td><td>38,9%</td><td>55</td><td>10 m</td></tr>
<tr><td>RS 1</td><td>0,25</td><td>4841,5</td><td>1433,5</td><td>3408</td><td>29,6%</td><td>62</td><td>10 m</td></tr>
<tr><td>RS 1</td><td>0,50</td><td>4411,9</td><td>925,8</td><td>3486,1</td><td>21,0%</td><td rowspan="3">4,1%</td><td rowspan="3">24,1%</td><td>65</td><td rowspan="3">3</td><td rowspan="3">63</td><td>10 m</td></tr>
<tr><td>RS 1</td><td>0,50</td><td>4474,3</td><td>1005,8</td><td>3468,5</td><td>22,5%</td><td>63</td><td>10 m</td></tr>
<tr><td>RS 1</td><td>0,50</td><td>4580,9</td><td>1318,1</td><td>3262,8</td><td>28,8%</td><td>60</td><td>10 m</td></tr>
<tr><td>RS 1</td><td>0,75</td><td>4335,4</td><td>646,6</td><td>3688,8</td><td>14,9%</td><td rowspan="2">0,7%</td><td rowspan="2">14,4%</td><td>73</td><td rowspan="2">1</td><td rowspan="2">74</td><td>10 m</td></tr>
<tr><td>RS 1</td><td>0,75</td><td>4354,9</td><td>606,5</td><td>3748,4</td><td>13,9%</td><td>74</td><td>10 m</td></tr>
</table>

RS2 – Rock Salt 0/2 mm and no anti-caking agent

Sample Name	Humidity (% mass)	Total mass(g)	Discharged mass(g)	Mass in Box(g):	Discharge in %:	Standard dev.	Average (%):	Angle (°):	Standard dev.	Average (°):	Time
RS2	0,00	4972,9	3002,1	1970,8	60,4%	0,6%	60,8%	40	1	40	5m
RS 2	0,00	4974,5	3047,3	1927,2	61,3%			39			5m
RS 2	0,25	4305,1	584,0	3721,1	13,6%	2,5%	15,5%	77	9	67	10m
RS 2	0,25	4464,6	654,7	3809,9	14,7%			74			10m
RS 2	0,25	4585,7	841,6	3744,1	18,4%			60			10m
RS 2	0,50	4181,9	666,6	3515,3	15,9%	2,6%	14,2%	71	2	70	10m
RS 2	0,50	4242,4	476,5	3765,9	11,2%			68			10m
RS 2	0,50	4308,6	662,8	3645,8	15,4%			70			10m
RS 2	0,75	4149,2	573,2	3576,0	13,8%	1,0%	14,2%	69	2	70	10m
RS 2	0,75	4142,9	633,2	3509,7	15,3%			68			10m
RS 2	0,75	4221,8	564,9	3656,9	13,4%			72			10m
RS 2	0,05	5088	3056,0	2032,0	60,1%	0,2%	60,2%	38	0	38	5m
RS 2	0,05	5134	3100,0	2034,0	60,4%			38			5m

RS3 – Rock Salt 0/3 mm and 70-100 mg/kg of SFC

Sample Name	Humidity (% mass)	Total mass(g)	Discharged mass(g)	Mass in Box(g):	Discharge in %:	Standard dev.	Average (%):	Angle (°):	Standard dev.	Average (°):	Time
RS3	0,00	5039	3050	1989	60,5%	0,2%	60,7%	39	1	40	10m
RS 3	0,00	5069	3081	1988	60,8%			40			10m
RS 3	0,25	4664	1987	2677	42,6%	5,4%	51,1%	50,5	2	47	10m
RS 3	0,25	4791	2336	2455	48,8%			46,5			10m
RS 3	0,25	4816	2574	2242	53,4%			48			10m
RS 3	0,50	4503	1605	2898	35,6%	6,1%	42,7%	52,5	1	51	10m
RS 3	0,50	4546	2077	2469	45,7%			51,5			10m
RS 3	0,50	4680	2184	2496	46,7%			50			10m
RS 3	0,75	4366	1376	2990	31,5%	0,4%	31,5%	60	4	56	10m
RS 3	0,75	4434	1418	3016	32,0%			53			10m
RS 3	0,75	4424	1376	3048	31,1%			54			10m

RS4 – Rock Salt 0/2 mm and 70-100 mg/kg of SFC

<table>
<tr><th>Sample Name</th><th>Humidity (%)</th><th>Total mass(g)</th><th>Discharged mass(g)</th><th>Mass in Box(g):</th><th>Discharge in %:</th><th>Standard dev.</th><th>Average (%):</th><th>Angle (°):</th><th>Standard dev.</th><th>Average (°):</th><th>Time</th></tr>
<tr><td>RS4</td><td>0,00</td><td>4934</td><td>3118</td><td>1816</td><td>63,2%</td><td rowspan="2">0,1%</td><td rowspan="2">63,3%</td><td>37</td><td rowspan="2">0</td><td rowspan="2">37</td><td>10m</td></tr>
<tr><td>RS 4</td><td>0,00</td><td>4928</td><td>3124</td><td>1804</td><td>63,4%</td><td>37</td><td>10m</td></tr>
<tr><td>RS 4</td><td>0,25</td><td>4404</td><td>1788</td><td>2616</td><td>40,6%</td><td rowspan="2">0,7%</td><td rowspan="2">39,6%</td><td>51</td><td rowspan="2">3</td><td rowspan="2">55</td><td>10m</td></tr>
<tr><td>RS 4</td><td>0,25</td><td>4482</td><td>1774</td><td>2708</td><td>39,6%</td><td>55</td><td>10m</td></tr>
<tr><td>RS 4</td><td>0,50</td><td>4190</td><td>1676</td><td>2514</td><td>40,0%</td><td rowspan="3">6,0%</td><td rowspan="3">33,4%</td><td>54</td><td rowspan="3">3</td><td rowspan="3">57</td><td>10m</td></tr>
<tr><td>RS 4</td><td>0,50</td><td>4228</td><td>1198</td><td>3030</td><td>28,3%</td><td>60</td><td>10m</td></tr>
<tr><td>RS 4</td><td>0,50</td><td>4262</td><td>1352</td><td>2910</td><td>31,7%</td><td>57</td><td>10m</td></tr>
<tr><td>RS 4</td><td>0,75</td><td>4082</td><td>1082</td><td>3000</td><td>26,5%</td><td rowspan="3">1,1%</td><td rowspan="3">27,5%</td><td>60</td><td rowspan="3">1</td><td rowspan="3">61</td><td>10m</td></tr>
<tr><td>RS 4</td><td>0,75</td><td>4111</td><td>1126</td><td>2985</td><td>27,4%</td><td>60</td><td>10m</td></tr>
<tr><td>RS 4</td><td>0,75</td><td>4187</td><td>1198</td><td>2989</td><td>28,6%</td><td>62</td><td>10m</td></tr>
</table>

VS1 – Vacuum salt with no anticaking agent

Sample Name	Humidity (% mass)	Total mass(g)	Discharged mass(g)	Mass in Box(g):	Discharge in %:	Standard dev.	Average (%):	Angle (°):	Standard dev.	Average (°):	Time
VS1	0,00	4141	2568	1573	62,0%			39			10 m
VS1	0,00	4186	2622	1564	62,6%	0,4%	62,5%	39	0	39	10 m
VS1	0,00	4218	2650	1568	62,8%			39			10 m
VS1	0,25	3904	924	2980	23,7%			65			10 m
VS1	0,25	3982	1006	2976	25,3%	2,6%	25,9%	62	4	62	10 m
VS1	0,25	4010	1153	2857	28,8%			58			10 m
VS1	0,50	3893	914	2979	23,5%			64			10 m
VS1	0,50	3981	956	3025	24,0%	2,1%	24,9%	63	1	64	10 m
VS1	0,50	3980	1086	2894	27,3%			64			10 m
VS1	0,75	3835	664	3171	17,3%			66,5			10 m
VS1	0,75	3872	608	3264	15,7%	1,5%	17,2%	70	2	68	10 m
VS1	0,75	3898	728	3170	18,7%			66			10 m

Sample Name	Humidity (%)	Total mass(g)	Discharged mass(g)	Mass in Box(g):	Discharge in %:	Standard dev.	Average (%):	Angle (°):	Standard dev.	Average (°):	Time
VS1	1,20	3784	649	3135	17,2%	0,1%	17,2%	67,5	2	69	10 m
VS1	1,20	3832	662	3170	17,3%			70			10 m

VS2 – Vacuum salt with no anticaking agent

Sample Name	Humidity (% mass)	Total mass(g)	Discharged mass(g)	Mass in Box(g):	Discharge in %:	Standard dev.	Average (%):	Angle (°):	Standard dev.	Average (°):	Time
VS2	0,00	4694	2994	1700	63,8%	0,9%	64,9%	38	1	37	10m
VS2	0,00	4741	3092	1649	65,2%			37			10m
VS2	0,00	4739	3107	1632	65,6%			37			10m
VS2	0,25	4336	634	3702	14,6%	0,3%	14,5%	75	2	74	10m
VS2	0,25	4368	648	3720	14,8%			72			10m
VS2	0,25	4430	628	3802	14,2%			75			10m
VS2	0,50	4226	618	3608	14,6%	2,3%	15,8%	75	3	73	10m
VS2	0,50	4224	778	3446	18,4%			70			10m
VS2	0,50	4312	618	3694	14,3%			75			10m
VS2	0,75	4080	0	4080	0,0%	9,3%	10,7%	90	11	77	10m
VS2	0,75	4062	690	3372	17,0%			69			10m
VS2	0,75	4065	620	3445	15,3%			72,5			10m
VS2	0,0129%	4626	2905	1721	62,8%	0,6%	63,4%	39	0	39	10m
VS2	0,0129%	4688	2974	1714	63,4%			39			10m
VS2	0,0129%	4682	2992	1690	63,9%			39			10m

Sample Name	Humidity (% mass)	Total mass(g)	Discharged mass(g)	Mass in Box(g):	Discharge in %:	Standard dev.	Average (%):	Angle (°):	Standard dev.	Average (°):	Time
VS2	0,10	4354	744	3610	17,1%	4,7%	19,4%	70	2	70	7m
VS2	0,10	4552	1130	3422	24,8%			67			1h
VS2	0,10	4597	750	3847	16,3%			72,5			10m

VS3 – Vacuum salt with no anticaking agent

Sample Name	Humidity (% mass)	Total mass(g)	Discharged mass(g)	Mass in Box(g):	Discharge in %:	Standard dev.	Average (%):	Angle (°):	Standard dev.	Average (°):	Time
VS3	0,00	4850	3166	1684	65,3%	0,8%	66,2%	37	0	37	10 m
VS3	0,00	4878	3245	1633	66,5%			37			10 m
VS3	0,00	4900	3270	1630	66,7%			37			10 m
VS3	0,25	4536	1082	3454	23,9%	3,9%	20,2%	63,5	4	67	10 m
VS3	0,25	4598	740	3858	16,1%			70,5			10 m
VS3	0,25	4704	966	3738	20,5%			68			10 m
VS3	0,50	4454	552	3902	12,4%	0,7%	13,0%	77,5	2	76	10m
VS3	0,50	4460	576	3884	12,9%			75			10m
VS3	0,50	4572	626	3946	13,7%			74,5			10m
VS3	0,75	4418	706	3712	16,0%	1,1%	15,8%	72	1	72	10m
VS3	0,75	4386	638	3748	14,5%			71,5			10m
VS3	0,75	4412	740	3672	16,8%			73,5			10m
VS4	0,75	4414	0	4414	0,0%			90			1h

Sample Name	Humidity (% mass)	Total mass(g)	Discharged mass(g)	Mass in Box(g):	Discharge in %:	Standard dev.	Average (%):	Angle (°):	Coment	Time
VS3	0,25	4498	0	4498	0,0%			90	Weight applied 1,336 g	19 h

VS4 – Vacuum salt with 8 mg/kg SFC

Sample Name	Humidity (% mass)	Total mass(g)	Discharged mass(g)	Mass in Box(g):	Discharge in %:	Standard dev.	Average (%):	Angle (°):	Standard dev.	Average (°):	Time
VS4	0,00	5082	3312	1770	65,2%	0,1%	65,2%	38	1	38	10 m
VS4	0,00	5054	3300	1754	65,3%			37			10m
VS4	0,25	4307	1677	2630	38,9%	0,0%	38,9%	54	1	55	10m
VS4	0,25	4402	1714	2688	38,9%			55			10m
VS4	0,50	4199	1098	3101	26,1%	1,9%	27,5%	60	1	60	10m
VS4	0,50	4268	1232	3036	28,9%			59			10m
VS4	0,75	4039	1014	3025	25,1%	0,8%	25,7%	63,5	0	63	10m
VS4	0,75	4083	1072	3011	26,3%			63			10m
VS4	0,75	4089	875	3214	21,4%			66	With a load of 1.340g over the salt		20h

VS5 – Vacuum salt with 8 mg/kg SFC

Sample Name	Humidity (% mass)	Total mass(g)	Discharged mass(g)	Mass in Box(g):	Discharge in %:	Standard dev.	Average (%):	Angle (°):	Standard dev.	Average (°):	Time
VS5	0,00	5054	3397	1657	67,2%	0,3%	67,5%	36	0	36	10 m
VS5	0,00	5045	3420	1625	67,8%			36			10 m
VS5	0,00	5028	3397	1631	67,6%			35,5			10 m
VS5	0,25	4341	1326	3015	30,5%	1,7%	27,7%	58	2	60	10 m
VS5	0,25	4324	1206	3118	27,9%			62,5			10 m
VS5	0,25	4337	1192	3145	27,5%			60			10 m
VS5	0,50	4154	1042	3112	25,1%	0,6%	24,7%	65	2	65	10 m
VS5	0,50	4178	1006	3172	24,1%			66			10 m
VS5	0,50	4220	1056	3164	25,0%			63			10 m
VS5	0,75	4086	934	3152	22,9%	1,7%	23,8%	66,5	1	66	10 m
VS5	0,75	4094	1054	3040	25,7%			65			10 m
VS5	0,75	4122	942	3180	22,9%			65			10 m
VS5	0,0088%	5019	3376	1643	67,3%	0,2%	67,2%	36	0	36	
VS5	0,0088%	5068	3398	1670	67,0%			36			

VS6 – Vacuum salt with 12 mg/kg biodegradable anticaking agent

Sample Name	Humidity (%)	Total mass(g)	Discharged mass(g)	Mass in Box(g):	Discharge in %:	Standard dev.	Average (%):	Angle (°):	Standard dev.	Average (°):	Time
VS6 - 12 mg/kg	0,00	4892	3220	1672	65,8%	0,6%	66,2%	37	1	37	10 m
VS6	0,00	4902	3234	1668	66,0%			37			10 m
VS6	0,00	4944	3310	1634	66,9%			35,5			10 m
VS6	0,25	4228	711	3517	16,8%	0,2%	17,0%	73	1	73	10 m
VS6	0,25	4260	732	3528	17,2%			72			10 m
VS6	0,25	4239	716	3523	16,9%			73			10 m
VS6	0,50	4116	768	3348	18,7%	2,1%	16,5%	72	2	72	10 m
VS6	0,50	4188	608	3580	14,5%			74			10 m
VS6	0,50	4100	672	3428	16,4%			70			10 m
VS6	0,75	4012	648	3364	16,2%	1,4%	16,6%	75,5	2	73	10 m
VS6	0,75	3994	745	3249	18,7%			74			10 m
VS6	0,75	4023	616	3407	15,3%			71,5			10 m
VS6	0,75	4004	650	3354	16,2%			71			10 m

Sample Name	Humidity (%)	Total mass(g)	Discharged mass(g)	Mass in Box(g):	Discharge in %:	Standard dev.	Average (%):	Angle (°):	Standard dev.	Average (°):	Time
VS6	0,0094%	4904	3287	1617	67,0%	0,1%	67,1%	36	0	36	10 m
VS6	0,0094%	4920	3306	1614	67,2%			36			10 m
VS6	0,0094%										

VS6 – Vacuum salt with 12 mg/kg biodegradable anticaking agent – Not dried in the cabinet

Sample Name	Humidity (% mass)	Total mass(g)	Discharged mass(g)	Mass in Box(g):	Discharge in %:	Standard dev.	Average (%):	Angle (°):	Standard dev.	Average (°):	Time	Comments
VS6	0,0169	4886	3285	1601	67,2%	0,0%	67,2%	36	0	36	10 m	
VS6	0,0169	4898	3290	1608	67,2%			36			10 m	
VS6	0,25	4318	688	3630	15,9%	1,0%	16,7%	68	3	70	10 m	Tested straight after adding the water
VS6	0,25	4334	772	3562	17,8%			69			10 m	Tested straight after adding the water
VS6	0,25	4322	708	3614	16,4%			74			10 m	Tested straight after adding the water
VS6	0,25	4468	1338	3130	29,9%	0,2%	30,1%	65	3	63	10 m	Tested 2h after adding water
VS6	0,25	4574	1382	3192	30,2%			61			10 m	Tested 2h after adding water
VS6	0,5	4070	702	3368	17,2%	0,8%	16,7%	73	1	72	10 m	Tested straight after adding the water
VS6	0,5	4076	656	3420	16,1%			71,5			10 m	Tested straight after adding the water
VS6	0,5	4180	714	3466	17,1%	0,8%	16,5%	68	3	70	10 m	2h after adding water
VS6	0,5	4238	678	3560	16,0%			72			10 m	2h after adding water

VS7 – Vacuum salt with 22 mg/kg SFC

Sample Name	Humidity (% mass)	Total mass(g)	Discharged mass(g)	Mass in Box(g):	Discharge in %:	Standard dev.	Average (%):	Angle (°):	Standard dev.	Average (°):	Time
VS7	0,00	4998	3340	1658	66,8%	0,1%	66,9%	36	0	36	10m
VS7	0,00	5068	3388	1680	66,9%			36			10m
VS7	0,00	5088	3408	1680	67,0%			36			10m
VS7	0,25	4514	1432	3082	31,7%	15,9%	51,3%	58	11	49	10m
VS7	0,25	4754	2782	1972	58,5%			44			10m
VS7	0,25	4874	3160	1714	64,8%			40			10m
VS7	0,25	4515	1652	2863	36,6%			62,5			10m
VS7	0,25	4825	3122	1703	64,7%			38			10m
VS7	0,50	4420	1670	2750	37,8%	3,3%	38,1%	55	2	53	10m
VS7	0,50	4554	1588	2966	34,9%			52,5			10m
VS7	0,50	4530	1882	2648	41,5%			51			10m
VS7	0,75	4349	1259	3090	28,9%	1,0%	30,1%	56,5	1	58	10m
VS7	0,75	4350	1320	3030	30,3%			59			10m
VS7	0,75	4340	1344	2996	31,0%			59			10m

Sample Name	Humidity (% mass)	Total mass(g)	Discharged mass(g)	Mass in Box(g):	Discharge in %:	Standard dev.	Average (%):	Angle (°):	Comment	Time
VS7	0,25	4986	3272	1714	65,6%			36	Weight applied 1,336 g	23h

VS8 – Vacuum salt with 95 mg/kg SFC

Sample Name	Humidity (% mass)	Total mass(g)	Discharged mass(g)	Mass in Box(g):	Discharge in %:	Standard dev.	Average (%):	Angle (°):	Standard dev.	Average (°):	Time
VS8	0,00	5048	3393	1655	67,2%	0,1%	67,2%	35	1	35	10:12
VS8	0,00	5026	3378	1648	67,2%			35			10:24
VS8	0,00	5032	3387	1645	67,3%			34			10:40
VS8	0,25	4562	2454	2108	53,8%	7,1%	49,2%	47	4	50	10:55
VS8	0,25	4668	1966	2702	42,1%			54			11:07
VS8	0,25	4636	2052	2584	44,3%			51			11:22
VS8	0,25	4660	2636	2024	56,6%			46			11:32
VS8	0,50	4340	1247	3093	28,7%	1,3%	29,3%	61,5	3	60	13:43
VS8	0,50	4366	1284	3082	29,4%			62			13:55
VS8	0,50	4358	1220	3138	28,0%			61,5			14:11
VS8	0,50	4506	1402	3104	31,1%			56,5			15:43
VS8	0,75	4286	1178	3108	27,5%	2,1%	29,8%	62	1	61	14:41
VS8	0,75	4342	1376	2966	31,7%			60			15:02
VS8	0,75	4354	1310	3044	30,1%			60			15:15

Sample Name	Humidity (% mass)	Total mass(g)	Discharged mass(g)	Mass in Box(g):	Discharge in %:	Standard dev.	Average (%):	Angle (°):	Comment	Time
VS8	0,50	4452	1214	3238	27,3%			65	1,336 kg applied	18h