Sodium Borohydride

A Study of the Effect of Operational Conditions on the Material Characteristics of Salt-like Substances During Bulk Storage

ME54010-20: Literature Research
Report number: 2023.MME.8834.

Casper E. van Eijk

Delft University of Technology

TU Delft

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by

Casper E. van Eijk

Student Name	Student Number
Van Eijk, Casper E.	4599756

Supervisor: Dr. Ir. D. L. Schott
Supervisor: Ir. M. C. van Benten
Faculty: Faculty of Mechanical Engineering, Delft

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Summary

Throughout this literature research report, the influence of operational conditions during bulk storage on the mechanical characteristics of various inorganic, crystalline, day-to-day salts has been investigated, in order to make a prediction on the effect of operational conditions on the mechanical characteristics of sodium borohydride. Firstly, an analysis and selection of the various mechanical characteristics has been conducted. Secondly, an analysis of the chemical properties helped to select a list of materials that are chemically similar to sodium borohydride. Thirdly, an effort was made to compare these materials to sodium borohydride using the selected mechanical characteristics. Using the particle sizes, bulk densities and angles of repose, sodium borohydride could to a certain extent be linked to calcium chloride, magnesium sulfate, potassium chloride, potassium carbonate and silicon dioxide. Lastly, an analysis on the effect of operational conditions on the mechanical characteristics of the materials has been conducted. No definitive conclusions could be drawn about the effect of the operational conditions on the mechanical characteristics of sodium borohydride.

Contents

1
Introduction

Nowadays, almost everyone is aware of global warming and the implications that accompany this effect. It is the responsibility of humans to try to minimize this effect in any way possible. One of the main contributors to this global warming effect is the colossal amount of energy that is consumed every day. The consumption of fossil fuels especially, is detrimental to the environment [1]. Because of this, it is imperative to try to decrease the amount of energy that is consumed and to try to find new, greener ways to fulfil the remaining energy demand. Climate change targets have highlighted the importance of finding solutions to these challenges and one of the solutions to keeping up with the high demand of energy sustainably is to make use of hydrogen produced power, a renewable energy source that only emits water [2]. Hydrogen is a clean fuel, especially when the electrolysis of water to create the hydrogen is done with renewable electricity. On top of that, the technology that uses hydrogen fuel to work is often a lot more efficient. However, hydrogen gas is a difficult gas to handle. It needs to be highly compressed to be transported in gas form, or it needs to be kept at an extremely low temperature to be transported in liquid form [3, 4]. It becomes apparent that finding other ways of storing hydrogen are necessary.

1.1. Purpose of Research

The need for other ways of storing hydrogen, in order to provide enough renewable energy solutions to keep up with the energy demand, creates possibilities for new subjects to study. One of these subjects is the storage of hydrogen using sodium borohydride ($NaBH_4$) as the storage medium. According to Mohring and Wu [3], the hydrogen on demand system that has been developed and patented by Millennium Cell proves to be an intelligent way of using the sodium borohydride as an easily controllable storage medium for practical hydrogen generation possibly in applications for backup power or fuel cell vehicles. However, a significant amount of work still needs to be done in order to bring this technology to commercialization [3]. A particular area that still needs a significant amount of research is the behaviour of sodium borohydride during bulk storage, with emphasis on the behaviour of the material throughout a range of different operational conditions.

The purpose of this literature research is to investigate the behaviour of sodium borohydride during bulk storage while subjected to various operational conditions. Due to the limited data available on the behaviour of the bulk stored sodium borohydride, this literature research tries to accomplish this by comparing sodium borohydride to various other materials that bear a close resemblance to sodium borohydride and possess relatively similar mechanical characteristics.

1.2. Research Questions

In order to successfully accomplish the purpose of this research, the following research question will have to be answered:

> What is the effect of different operational conditions during bulk storage on the mechanical characteristics of materials similar to sodium borohydride?

This research question has been divided into the following sub-questions that will outline this report:

1. Which mechanical characteristics are of importance in classifying bulk materials?
2. Which materials bear a resemblance to sodium borohydride?
3. To what extent can these materials be compared to sodium borohydride?
4. What is the effect of bulk storage operational conditions on the mechanical characteristics?
5. What is the expected behaviour of sodium borohydride to the bulk storage operational conditions?

1.3. Methodology and Scope of Research

In order to answer these sub-questions, and subsequently answer the research question, a certain step-by-step methodology has been followed. Firstly, a selection of the relevant mechanical characteristics has been made. With respect to these mechanical characteristics, the aim is on the bulk material characteristics, describing the mechanical characteristics of the bulk material in interaction with itself. The particle material characteristics, describing the material characteristics of one particle, have been omitted because this research deals with bulk material, rather than individual particles. The equipment material characteristics and the interaction characteristics have been omitted because in this research, the presence of handling equipment has been neglected.

Secondly, a list of twenty materials bearing a resemblance to sodium borohydride has been constructed. For the selection of these materials, the focus has been on inorganic, crystalline, salt-like materials. Additionally, it has been decided that these materials need to have a day-to-day application. This is decided because it is expected that a lot of information is available on the production and storage of these materials. After the selection of the relevant materials, selected mechanical characteristics have been listed.

Thirdly, a comparison has been made between sodium borohydride and the other materials. And lastly, an analysis has been conducted on the effect of the operational conditions on the mechanical characteristics of the twenty materials. Information has only been found on this effect for three of the twenty materials, so to illustrate the information gap present in the available literature, a detailed search strategy has been included in Appendix A.

1.4. Outline of Research

This literature research is guided by the aforementioned sub-questions. As a result, the following chapters have been created.

- Chapter one includes the purpose and research questions, as well as the methodology, scope, and outline of this research report.
- Chapter two includes definitions of the various classes of mechanical characteristics and a selection of the relevant classes, as well as definitions of the relevant mechanical characteristics in these classes.
- Chapter three includes an introduction to sodium borohydride, as well as the selection process of the twenty materials and a list of the mechanical characteristics of these materials.
- Chapter four includes a comparison between sodium borohydride and the twenty materials based on their mechanical characteristics.
- Chapter five includes an analysis of the operational conditions that become relevant when storing bulk material. It includes definitions of the operational conditions, as well as the influence of these operational conditions on the relevant mechanical characteristics of the resembling materials. On top of that, a prediction is made on the influence of the operational conditions on the mechanical characteristics of sodium borohydride.
- In chapter six, the conclusions of this literature research have been summarized, and the research questions have been answered.

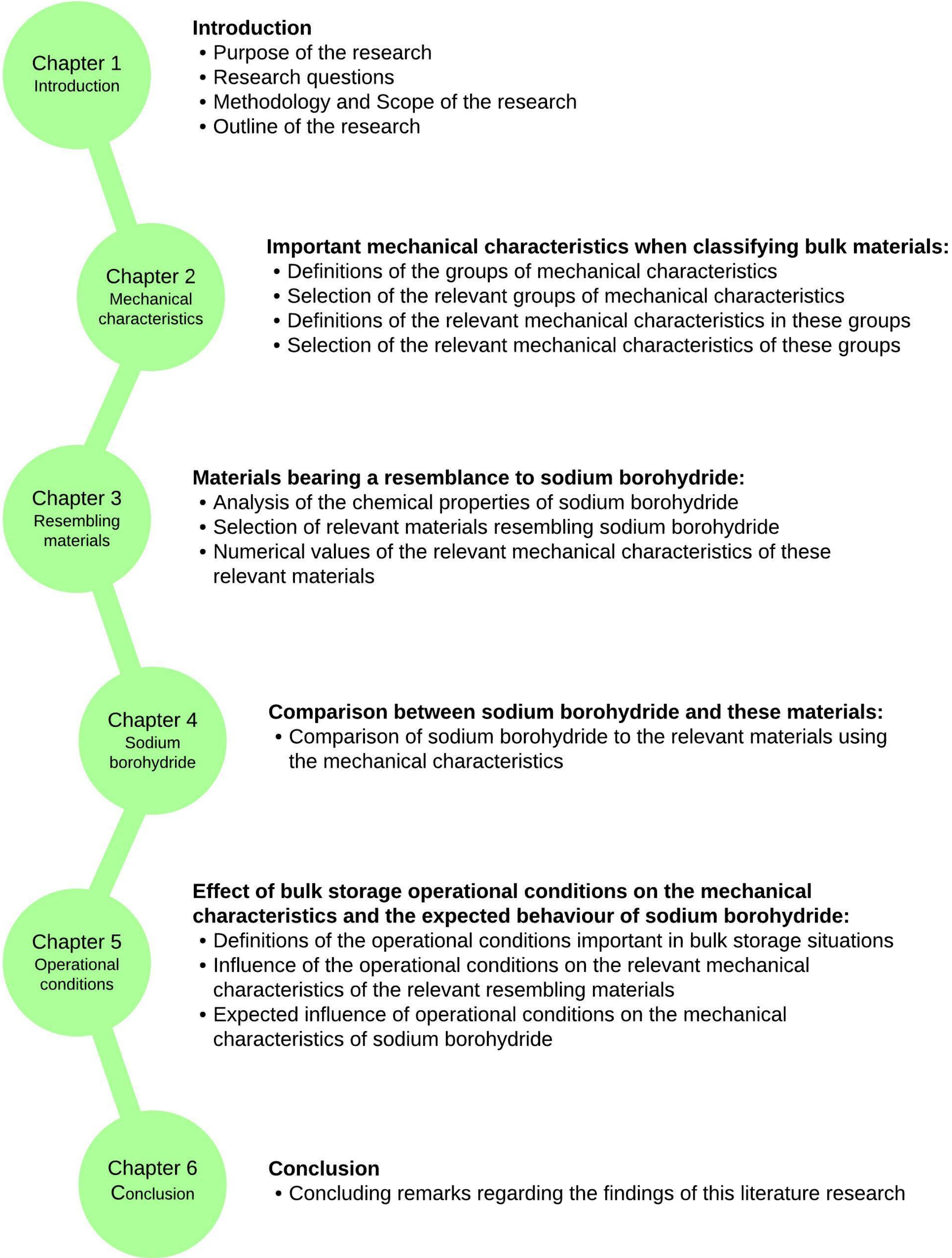

Figure 1.1: Outline of this report.

2
Mechanical Characteristics

In this chapter, an introduction is given into the mechanical characteristics that are widely used to describe and define materials and their behaviour. First, the different groups of mechanical characteristics are introduced and defined, and the relevant groups are selected for further analysis. Next, the mechanical characteristics in the selected groups are defined, and the relevant characteristics are determined.

2.1. Classes of Mechanical Characteristics

Mechanical characteristics describe the characteristics and behaviour of certain materials and their surroundings. The mechanical characteristics can be divided into four different classes. The final summary of these classes and its characteristics can be found in Figure 2.1 [5][6][7].

Mechanical characteristics

Class 1: *Particle material characteristics,* describing the material characteristics of one particle.
Class 2: *Bulk material characteristics,* describing the material characteristics of a bulk of material interacting with itself.
Class 3: *Equipment material characteristics,* describing the material characteristics of the handling equipment that is in contact with the material.
Class 4: *Interaction characteristics,* describing the characteristics of the interaction between handling equipment and bulk material.

Class 1: Particle Material Characteristics

The first and most straightforward class of mechanical characteristics is the class of particle material characteristics. Particle material characteristics describe the material characteristics of a single particle. Common particle material characteristics are particle shape and size, density, elasticity, and Poisson's ratio, internal damping, surface roughness and surface hardness.

Class 2: Bulk Material Characteristics

Bulk material characteristics describe the mechanical characteristics and the material behaviour of a bulk of multiple particles interacting with one another. Common bulk material characteristics are the particle size and shape distributions, the bulk density and elasticity, the compressive and tensile strength, the internal friction, the shear angle, the angle of repose, cohesion and flowability of the bulk.

Class 3: Equipment Material Characteristics

The third class consists of the mechanical characteristics of the handling equipment itself. Common equipment material characteristics are the shape and size of the equipment, the displacements, velocities, and accelerations of the equipment, the surface roughness of the equipment and mechanical properties such as the shear modulus, Poisson's ratio and the hardness of the equipment material.

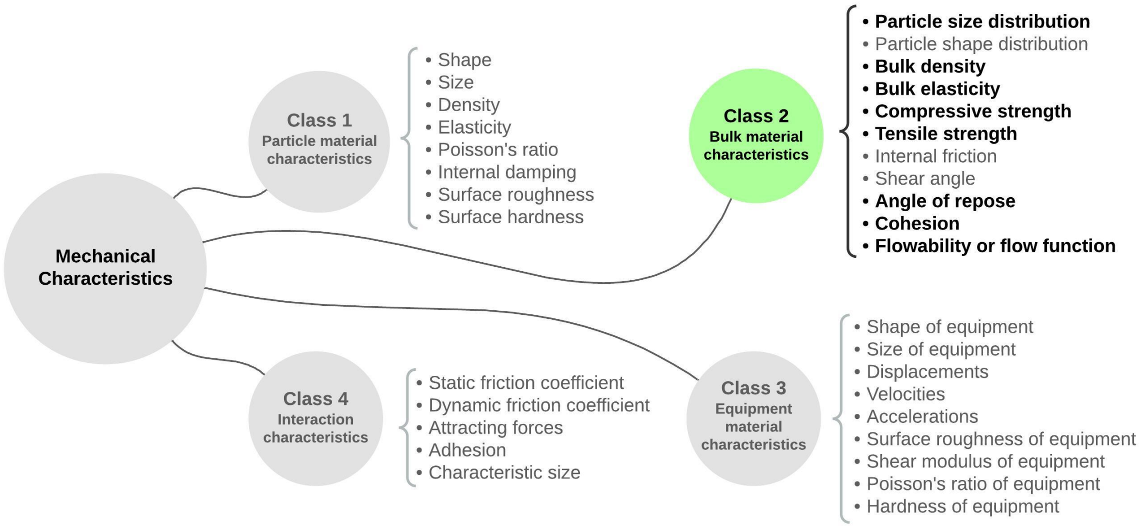

Figure 2.1: Overview of the mechanical characteristics.

Class 4: Interaction Characteristics

The interaction characteristics describe the mechanical characteristics of the interaction between the handling equipment and the material itself. Common interaction characteristics are the coefficients of static and dynamic friction between the particles and the equipment, the adhesion between particles and equipment and the characteristic size of the equipment with respect to the particle size.

Selection of Relevant Class of Mechanical Characteristics

Not all of these four classes of mechanical characteristics are of particular interest to this literature assignment. Class 1 has been omitted from this research because it concerns itself with the material characteristics of individual particles, rather than those of bulk material. Class 3 and 4 have been omitted due to the fact that this research does not include the influence of the equipment on the bulk material. Class 2 on the other hand is of great interest to this research, because this class concerns the bulk material characteristics.

2.2. Definitions of Bulk Material Characteristics

As was discussed before, the bulk material characteristics are the most important mechanical characteristics to this literature research. This is due to their applicability to bulk material, and its behaviour during bulk storage. Within the class of bulk material characteristics, a distinction can be made between eleven individual characteristics. An introduction and definition to these eleven characteristics has been provided.

Particle Size Distribution

A lot of different types of granular materials exist in an array of different applications and varieties. Bulks of these granular materials almost always consist of groups of particles with different particle sizes existing within this bulk in a sort of distribution, also called the particle size distribution [8]. When using the particle size or particle size distribution, it is important to take note of the way in which this size is determined. Vastly different ways of selecting a relevant measurement length can be distinguished throughout the literature [9, 10].

When the size-range of the available particles in a bulk is known, the particle size distribution can be determined. This describes the relative amounts of the different-sized particles within a bulk. The distribution of the particle sizes can have a large effect on the properties of a bulk material [11]. When deviations between the particle sizes in a bulk are very small, the particles are monodisperse, but more often than not, the particle sizes vary significantly, and the size distribution is called polydisperse [10].

When dealing with a polydisperse material, it is important to keep in mind that the difference in particle size can have an effect on determining the behaviour and the bulk characteristics of that material [8].

Particle Shape Distribution

Similarly to particle size, particles within a bulk can also have different particle shapes. Particle shape can have an influence on properties like flowability and packing of particles in a bulk [9]. Three different scales can be identified in which particle shape becomes important, the macroscale which is related to the general 3D form of the particles, the mesoscale which is related to the general roundness and angularity of the contour of the particle and the microscale which is related to the smoothness and porosity of the particles [10]. Again, it is important to note that different ways of determining the particle shape distribution exist. When using the particle shape or particle shape distribution, it is important to distinguish the way in which this is measured [9, 10].

Bulk Density

The bulk density is an important characteristic for dry particulate materials, determining the relation between the mass and the volume of a bulk of particles. Because the interstitial space between the particles is included in the bulk density, the packing of the particles is also of importance. A distinction can be made between two types of packing; loose packing, a method of packing particles by dropping these particles from a standardized height through a standardized funnel, or tight packing, a method of packing the particles by tapping the bulk of material with a given mass [10]. The packing of the bulk material has an influence on the interstitial space, or voids, of the bulk. The ratio of the void volume to the total volume of the bulk determines the porosity [12]. Because the bulk density measures the density of a bulk of material while taking into account the density of the solid material and the amount of porosity, it is important to note that the bulk density affects the bulk strength [11].

Bulk Elasticity and Plasticity

A bulk of material can experience elastic or plastic deformation when subjected to certain loads and stresses. When the material exhibits elastic behaviour, the strain will return to the original point when the stress is released. However, when it exhibits plastic behaviour, a part of the strain will remain after the stress is released. For bulk solids, elastic deformation is often negligible because it does not play a particularly significant role in bulk solid applications. This is due to the fact that the particles will often start moving when the bulk is subjected to stress. This means that the bulk has plastically deformed instead of elastically. Plastic deformations of bulk solids can contribute to changes in material characteristics of the bulk. Plastic deformation of bulk solid material generally refers to the moving of particles with respect to each other, rather than the deformation of individual particles. Common changes in bulk material characteristics due to plastic deformation are for example a change in bulk volume or a change in bulk density. This in turn can have an effect on the stiffness of the bulk of material. What happens with the bulk density and the bulk volume depends on the consolidation of the bulk before it was plastically deformed, but also on the load that is exerted on the bulk that set in motion the plastic deformation [12].

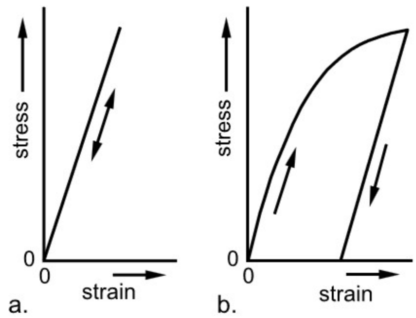

Figure 2.2: Stress-strain relationship for elastic (a) and plastic (b) deformation of solids [12].

Compressive and Tensile Strength of Bulk

The compressive strength of a material describes the tendency of the material to withstand a crushing or compressive force before the material deforms plastically. For bulk solids, the unconfined yield strength is often used to give a measure of the compressive strength of the bulk. The unconfined yield strength is determined by first compressing the bulk material with a given consolidation stress. Next, the boundaries surrounding the bulk are removed. And finally, the bulk is compressed using a new stress. The stress causing failure is referred to as the unconfined yield strength. However, throughout this work, the term compressive strength is used. The tensile strength of the material describes the tendency of the material to withstand a pulling or tensile force before the material deforms plastically [12].

Internal Friction between Particles

The internal friction between particles can be expressed using the angle of internal friction. It determines the directions of sliding of powder particles in relation to the principle planes of these powder particles [13]. The angle of internal friction is a measure of the force that is required to cause particles to move or slide on each other. This angle is influenced by material characteristics such a particle surface friction, particle shape, particle hardness, particle size and particle size distribution [14]. An important distinction must be made between the angle of internal friction and the effective angle of internal friction. The angle of internal friction is defined as the local slope of the yield locus against the σ-axis. It is often sufficient to construct the angle of internal friction using the linearized yield locus. In this case, the angle of internal friction is determined by the slope of this linearized yield locus with the σ-axis. The effective angle of internal friction is the slope of the effective yield locus and defines the ratio of the minor principal stress to the major principal stress at a steady state flow. The angle of internal stress usually decreases with an increasing consolidation stress [12]. The angle of internal friction is measured using a specialized shearing box in which the bulk material particles slide on each other [15]. The higher the angle of internal friction, the more difficult it is for the powder or bulk solid material to move [16].

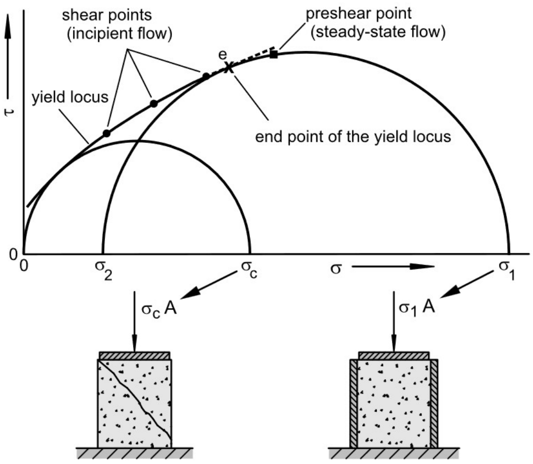

Figure 2.3: Yield locus and Mohr stress circles defining the unconfined yield strength and the consolidation stress [12].

Cohesion of Material

Cohesion is a term that describes the phenomenon of particles in a bulk solid sticking together. It can also be expressed as the resistance of the bulk solid to shear at zero compressive normal load [13]. The cohesion value can be found when looking at the intersection point of the yield locus with the shear axis at the point where the normal stress is equal to zero [12]. A couple of sources of cohesion that can be identified include liquid bridges when moisture is present, van der Waals forces, electrostatic forces and magnetic forces. The first two are the most prevalent forces that cause cohesion between particles. An increase in cohesion can be caused by an increase in moisture content or a decrease of the particle size [14]. For many powders, the value of cohesion can be approximated by taking twice the tensile strength of the bulk material. Additionally, more cohesive bridging is present in a material with a high bulkiness and therefore a high porosity [17]. As was said before, an increase in cohesion can be caused by an increase in moisture content, however, above a critical moisture content, the forces of cohesion start to get disrupted. This is due to the water content between the particles. This water also prohibits the production of new bonds [18].

Flowability or Flow Function

Flowability can be defined as the ability of bulk solid material to flow. It is greatly affected by mechanical characteristics like particle size and moisture content, as well as operational conditions like storage time, temperature, pressure and relative humidity. These material characteristics can cause caking or bridging of the material, which in turn influences the flowability. Flowability itself is not a natural material property, therefore it can be best described using the flow function, also called the failure function of the material [14]. Different consolidation stresses yield different unconfined yield strengths. The instantaneous flow function is a plot of these variables

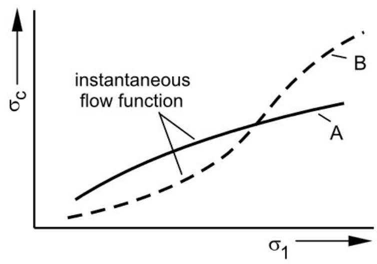

Figure 2.4: Unconfined yield strength dependent on consolidation stress [12].

Angle of Repose and Shear Angle

The angle of repose describes the slope of a heap of bulk solid material. Various ways of defining an angle of repose can be distinguished. The three main ways are the poured angle of repose, the drained angle of repose and the dynamic angle of repose.

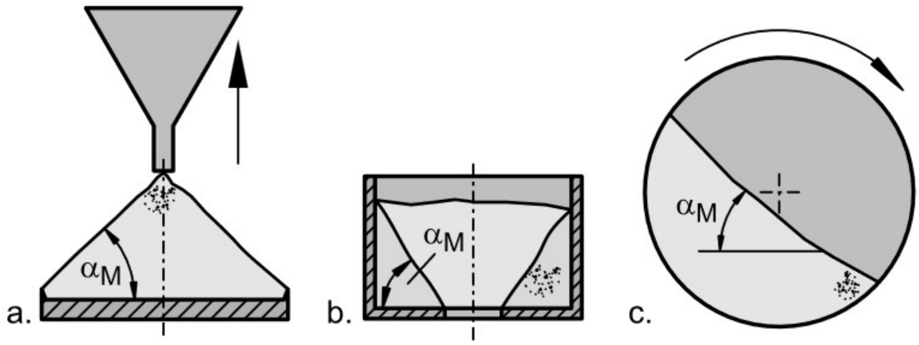

Figure 2.5: Poured angle of repose (a), drained angle of repose (b), dynamic angle of repose (c) [12].

The poured angle of repose is determined by pouring the bulk solid material through a funnel onto a flat surface with a protruding outer edge. This funnel can either be held fixed at a constant height above the flat surface, or moved upwards at a constant height above the top of the heap. This way of determining the angle of repose can only deal with materials at a low compaction grade. This type of compaction is usually present at the surface of a pile of material. It is also important to note that dynamic effects caused by dropping the material from a certain height might influence the results of the angle of repose test, and therefore this angle of repose is not applicable in situations in which greater stresses or time consolidation play a role [12]. When a big angle of repose is measured, it usually signifies that the particles stick together due to cohesion, causing a difficult flow, whereas a small angle of repose signifies a material that flows relatively easy. Usually, the angle of repose increases with an increasing moisture content [14]. Additionally, the angle of repose is influenced by the friction between the particles as well as the cohesion between the particles [19].

The drained angle of repose also describes the slope of a stationary heap of bulk solid material, however the way of determining this drained angle of repose is different compared to the angle of repose. This angle is determined by positioning the bulk solid material in a flat-bottomed box. Subsequently, a central outlet is used to discharge parts of the bulk solid material. The drained angle of repose is then determined by the slope of the material that remains within the container. Because the material is first positioned in a box and later drained through the central outlet, this material is able to consolidate somewhat. Therefore, the stresses in the material that cause this consolidation influence the drained angle of repose. The larger these stresses, the steeper the drained angle of repose shall be [12].

The dynamic angle of repose describes the slope of a dynamically rotating heap of bulk solid material. It is determined by slowly rotating a partly filled cylinder horizontally around its axis and measuring the slope of the material inside the cylinder [12]. The dynamic angle of repose can help to observe the behaviour of the powder when subjected to dynamic surroundings and can indicate some degree of cohesion within the material [13], because cohesive materials do not flow downward continuously, but tend to fall down like avalanches which makes the process of determining the angle of repose much more difficult. The dynamic angle of repose is dependent on the revolving speed, the state of fluidization, the ratio between the particle size and the cylinder size and possible segregation and agglomeration of the material [12].

The shear angle is somewhat of a synonym of the angle of repose, and these two terms are often used interchangeably [7]. Another term that is used to describe the angle of repose is the avalanche angle [20].

2.3. Selection of Relevant Bulk Material Characteristics

A selection is made of the relevant bulk material characteristics listed in Figure 2.1.

Class 2: Bulk material characteristics

- v **Particle size distribution:** Although the particle size of particles in a bulk of material can vary significantly, the size of the particles can be determined easily using a sieve. Therefore, it is expected that sufficient data is available on the particle size distributions of the materials that have been selected.
- x **Particle shape distribution:** The particle shape distribution has been omitted from this research because a preliminary scan of the available literature showed that not sufficient data was available.
- v **Bulk density:** The dry bulk density is often well documented for granular bulk materials, and therefore it is incorporated in this research.
- v **Bulk elasticity and plasticity:** The bulk elasticity and plasticity are important measures of the behaviour of a material when it is subjected to stress. Therefore, it has to be incorporated in this research.
- v **Compressive strength:** The compressive strength is an important measure of how well a bulk material can withstand compressive stresses before deforming plastically. Therefore, it must be incorporated in this research.
- v **Tensile strength:** The tensile strength is an important measure of how well a bulk material can withstand tensile stresses before deforming plastically. Therefore, it must be incorporated in this research.
- x **Internal friction:** The importance of incorporating the internal friction of a bulk material is believed to be covered by the incorporation of the angle of repose and the flowability into this research. Therefore, the internal friction is omitted from this research.
- v **Cohesion:** Cohesion of a bulk material is highly influenced by the presence of humidity, as well as consolidation. Therefore, cohesion is incorporated in this research.
- v **Flowability or flow function:** The flowability of a bulk solid material is dependent on various mechanical characteristics as well as operational conditions. Therefore, it is incorporated in this research.
- v **Angle of repose and shear angle:** The angle of repose is easy to measure using one of the discussed methods. Therefore, it is expected that sufficient data is available. Therefore, the angle of repose is incorporated in this research.

A further selection has been made of these mechanical characteristics. For the materials selected in section 3.2, numerical values have been assigned to the particle size distribution, bulk density and angle of repose. These have been used to compare the materials with one another in chapter 4. These three mechanical characteristics have been selected because the particle size, bulk density and angle of repose are often measured in laboratory setups, which means that sufficient data can be found, and the dryness of the samples is often known. The other mechanical characteristics selected in section 2.3 have been used to investigate their behaviour when subjected to various operational conditions in chapter 5.

3

Resembling Materials

In this chapter, the chemical properties of sodium borohydride have been investigated and documented. On top of that, a list of twenty inorganic, crystalline, salt-like, day-to-day materials has been created. Last but not least, an overview of the numerical values of the mechanical characteristics discussed in chapter 2 of these materials has been created.

3.1. Analysis of the Chemical Properties of Sodium Borohydride

Sodium borohydride is an inorganic sodium salt. It appears as white to greyish crystals or crystalline powder [21]. In order to find materials with a close resemblance to sodium borohydride, it is important to look for inorganic, crystalline salts.

According to Encyclopaedia Britannica, the field of chemistry defines a salt as the product of a reaction between an acid and a base. The positive ion, or cation, of the base, and the negative ion, or anion, of the acid react together and neutralize each other to form an electrically neutral salt and another product. When a salt is dissolved or in a molten state, most salts will fully disintegrate into their respective positively charged cations and their negatively charged anions again. These are also called electrolytes [22]. The positively charged ions, or cations, are ions that lose electrons in the neutralization reaction. Most metals tend to want to lose their valence electrons, and therefore most metals are cations. The negatively charged ions, or anions, are ions that gain electrons in the neutralization reaction. Most non-metals tend to accept the valence electrons, and therefore they can be classified as anions [23].

Within the category of salts, a distinction can be made between organic salts and inorganic salts. An inorganic salt, is a salt that does not contain carbon-hydrogen bonds, and logically, an organic salt is a salt that does contain carbon-hydrogen bonds [24]. A crystal or crystalline powder is signified as a solid material in which the atoms of that material are arranged in a recurring pattern. The smallest individual part of that recurring pattern is called the unit cell [25].

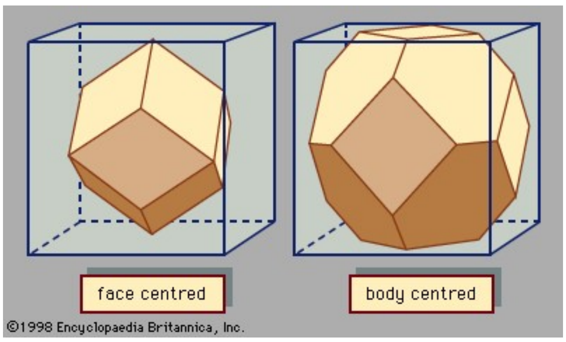

Figure 3.1: Unit cells for face-centred and body-centred cubic lattices [25].

3.2. Comparable Inorganic, Crystalline, Salt-like Materials

In order to find materials that bear a resemblance to sodium borohydride, and make sure that the materials that are being researched in this assignment microscopically resemble one another, it is important to look for inorganic, crystalline salts. However, most salts have a crystalline structure [26], therefore, to further limit the scope of this research, only materials that have a relative day-to-day use have been researched. This was done due to the fact that presumably, ample research has been done regarding these materials, and the production methods and storage conditions of these materials might be well known and well documented. The twenty selected materials and sodium borohydride have been listed in Table 3.1 below, accompanied by their day-to-day use.

	Material			
Name	**Synonym**	**Code**	**Application**	
Sodium Borohydride	-	NaBH4	Make other chemicals, treat waste water, store hydrogen	[21]
Aluminium Sulfate	-	Al2(SO4)3	Water purification, pH regulation, coagulating agent, deodorant	[27]
Ammonium Nitrate	-	NH4NO3	Fertilizer, explosive in mining industry	[28]
Calcium Carbonate	Limestone/chalk	CaCO3	Construction, paper, plastics, paint	[29]
Calcium Chloride	-	CaCl2	Drying agent, road de-icing, food preservation	[30]
Calcium Sulfate	Gypsum	CaSO4	Building materials, dentistry, cast	[31]
Copper(II) Sulfate	-	CuSO4	Pesticide, germicide, feed additive, soil additive	[32]
Iron(II) Sulfate	-	FeSO4	Water or sewage treatment, fertilizer ingredient	[33]
Magnesium Chloride	-	MgCL2	Medicine, cathartic, alloys,	[34]
Magnesium Sulfate	Epsom Salt	MgSO4	Laxative, fertilizers, bath salts	[35]
Potassium Carbonate	-	K2CO3	Catalyst, fertilizer, flame retardant	[36]
Potassium Chloride	-	KCl	Fertilizer, water softener, production of potassium metal	[37]
Potassium Nitrate	Indian Saltpetre	KNO3	Fertilizer, gunpowder, food preservative, fireworks, toothpaste	[38]
Silicon Dioxide	Sand	SiO2	Sand	[39]
Sodium Bicarbonate	Baking Soda	NaHCO3	Baking agent, antacids, fire extinguisher	[40]
Sodium Carbonate	Washing Soda	Na2CO3	Glass, soap, paper	[41]
Sodium Chloride	Table salt	NaCl	Cooking, preserving food, de-icing agent	[42]
Sodium Metabisulfite	-	Na2S2O5	Food preservative/antioxidant and laboratory agent	[43]
Sodium Nitrate	Chile Saltpeter	NaNO3	Solid propellants, explosives, fertilizers, food preservative	[44]
Sodium Sulfate	-	Na2SO4	Electrolyte replenisher	[45]
Zinc Sulfate	-	ZnSO4	production of rayon, feed supplement, fertilizer ingredient	[46]

Table 3.1: List of the selected materials.

3.3. Mechanical Characteristics of these Materials

An analysis of the mechanical characteristics of the materials discussed in section 3.2 has been conducted. Numerical values have been listed in Table 3.2 for the particle size, the bulk density and the angle of repose. These values have been used in chapter 4 to compare the materials with sodium borohydride.

Material	**Particle size [micron]**			**Bulk density [kg/m^3]**			**Angle of repose [$^\circ$]**		
	Min	**Max**		**Min**	**Max**		**Min**	**Max**	
Sodium Borohydride	1000	4000	[47]	510	530	[47]	34	36	[47]
Aluminium Sulfate	100	2500	[48]		1041	[49]		32	[50][51]
Ammonium Nitrate	100	1000	[52]		785	[49]	30	44	[50][51]
Calcium Carbonate		50	[53]		705	[49]	38	45	[50][54]
Calcium Chloride	500	5000	[55]	817	1057	[49][56]	28	32	[56]
Calcium Sulfate	26	124	[57]		721	[49]	30	42	[50]
Copper(II) Sulfate	150	2000	[58]		833	[49]		31	[50]
Iron(II) Sulfate	220	1000	[59]		1282	[49]			
Magnesium Chloride		500	[60]		192	[49]			
Magnesium Sulfate	200	7000	[61]		833	[49]	30	40	[62]
Potassium Carbonate	100	2500	[63]		1185	[49]	30	40	[62]
Potassium Chloride	500	2000	[64]		961	[49]	30	40	[62]
Potassium Nitrate	50	250	[65]		800	[66]	30	40	[62]
Silicon Dioxide	62	500	[67]		1586	[49]	34	35	[50][54]
Sodium Bicarbonate	65	70	[68]	801	2200	[49][69]	35	42	[50][70]
Sodium Carbonate	75	600	[71]	950	1150	[72]		38	[73]
Sodium Chloride	1	1000	[74]	1282	1330	[49]		25	[50]
Sodium Metabisulfite				1000	1200	[75]			
Sodium Nitrate	519	1223	[76]		1346	[49]		24	[50]
Sodium Sulfate	30	450	[77]		1362	[49]	30	40	[62]
Zinc Sulfate				1281	1442	[78]			

Table 3.2: Particle size range, bulk density and angle of repose of the selected materials.

4

Sodium Borohydride

In the previous chapter, the various materials have been listed, including their particle size, bulk density and angle of repose. In this chapter, these mechanical characteristics have been used to compare the various materials with one another and with sodium borohydride.

4.1. Comparison between Materials and Sodium Borohydride

Particle Size vs. Bulk Density

The ranges of the particle sizes and the bulk densities of the various materials have been plotted in Figure 4.1. Two things can be concluded from this plot. Firstly, the bulk density of sodium borohydride is not similar to the bulk densities of the other materials. This means that when primarily looking at the bulk density, the behaviour of sodium borohydride can not be approximated by any of the selected materials. Secondly, there are a couple of materials that possess a roughly similar particle size range, like calcium chloride and a bit like magnesium sulfate.

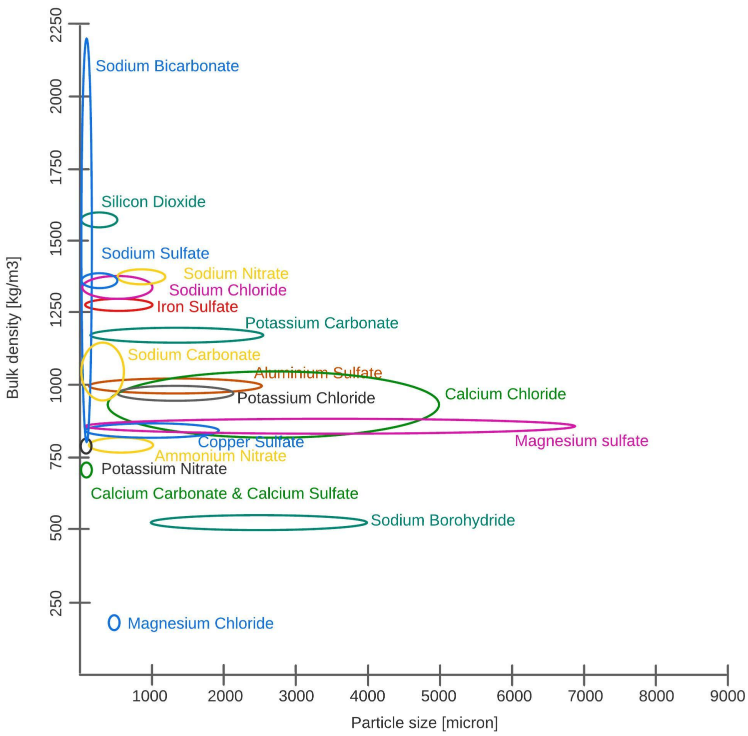

Figure 4.1: A plot of the particle sizes vs. the bulk densities.

Particle Size vs. Angle of Repose

The ranges of the particle sizes and the angles of repose of the various materials have been plotted in Figure 4.2. As discussed in section 4.1, the particle size of sodium borohydride is a bit similar to that of calcium chloride and magnesium sulfate. On top of that, the plot of sodium borohydride has some overlap with potassium carbonate and potassium chloride. Additionally, the angle of repose of silicon dioxide and sodium borohydride are quite similar.

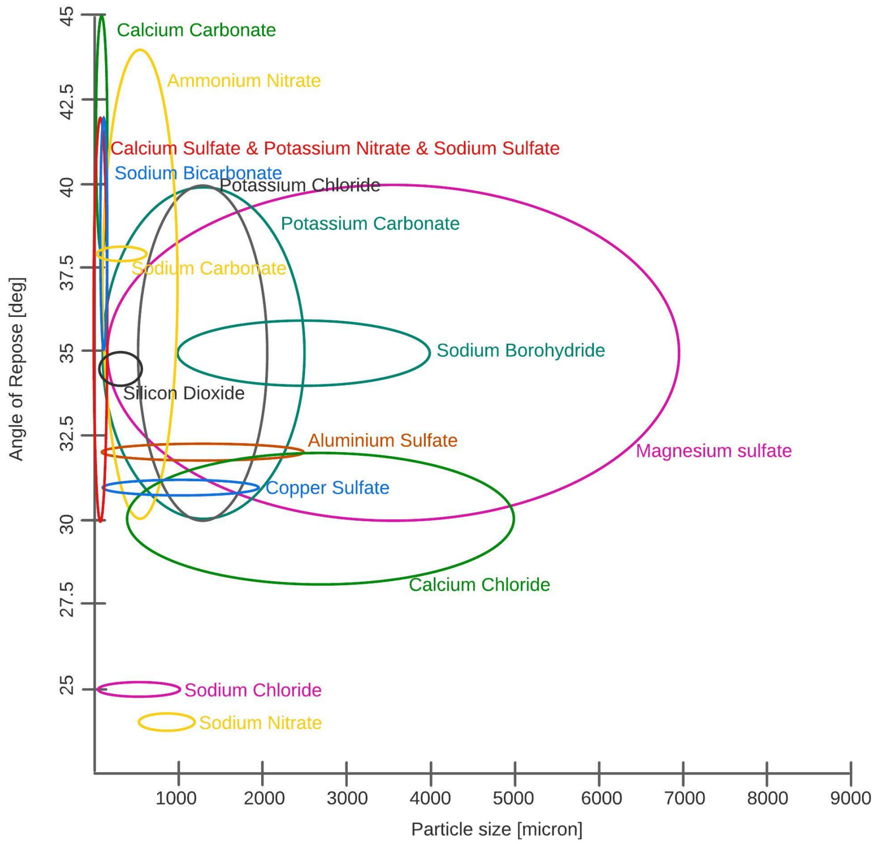

Figure 4.2: A plot of the particle sizes vs. angles of repose.

Angle of Repose vs Bulk Density

The ranges of the angles of repose and the bulk densities of the various materials have been plotted in Figure 4.3. As was concluded in section 4.1, the bulk density of sodium borohydride is nowhere near any of the other materials. However, as concluded in Figure 4.1, the angle of repose of sodium borohydride does overlap somewhat with a number of the materials.

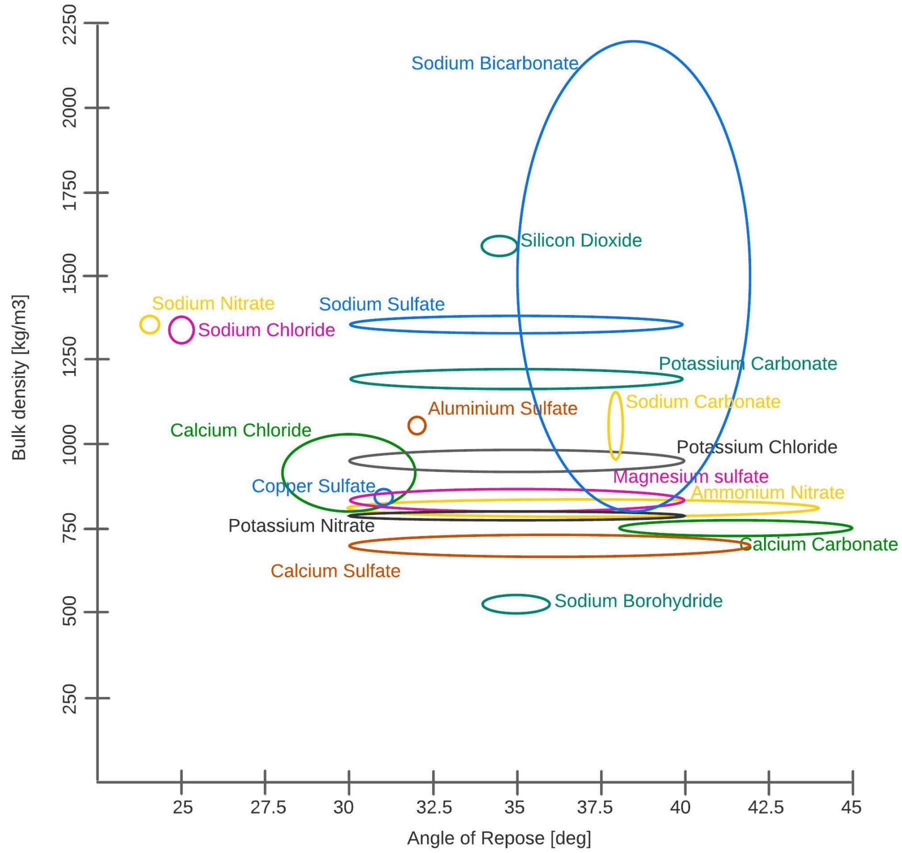

Figure 4.3: A plot of the angles of repose vs. the bulk densities.

4.2. Conclusions

Based on the above plots, a couple of things can be concluded. Some of the analysed materials bear a small resemblance to sodium borohydride, and these have been further discussed in the sections below.

Calcium Chloride

Calcium chloride bears a close resemblance to sodium borohydride when looking at the particle size distributions. The ranges of the particle sizes are quite similar, as can be seen in Figure 4.2. The bulk density of calcium chloride, on the other hand, is about twice as high as the bulk density of sodium borohydride, and the angle of repose differs approximately 5 degrees.

Magnesium Sulfate

The plots of magnesium sulfate and sodium chloride overlap in Figure 4.2, although the ranges of magnesium sulfate are quite a lot larger than that of sodium borohydride. However, the bulk density difference between magnesium sulfate and sodium borohydride is quite significant.

Potassium Chloride

Potassium chloride bears a resemblance to sodium borohydride when looking at the particle size distribution. In Figure 4.2 the plots overlap somewhat, signifying that their angles of repose and their particle size are alike. The bulk density however is almost twice as high for potassium chloride compared to sodium borohydride.

Potassium Carbonate

Potassium carbonate bears a resemblance to sodium borohydride when looking at the particle size distribution. In Figure 4.2 the plots overlap somewhat, signifying that their angles of repose and their particle size are alike. The bulk density however is more than twice as high for potassium carbonate compared to sodium borohydride.

Silicon Dioxide

Silicon dioxide resembles sodium borohydride when looking at the angle of repose. Their angles of repose are very similar. However, The bulk density of silicon dioxide is more than three times as high as that of sodium borohydride.

Conclusion

The results of the analysis of the mechanical characteristics of these materials show that although the materials that have been studied all consist of inorganic, crystalline salts, Their respective mechanical characteristics can still differ quite a lot. This means that none of the materials studied in this research sufficiently resemble sodium borohydride.

5
Operational Conditions

Throughout the world, silos are used to store all kinds of bulk solid materials. Because of their height, silos can hold the same amount of material on a smaller surface area when compared to other means of storage. On top of that, because silos are enclosed, the material is somewhat protected from outside influences. Last but not least, by keeping the material in one place, it is easier to control and register the operational conditions influencing the storage conditions of the material [79]. However, the design of the silo is of great importance to the effectiveness of that silo, since a poorly designed silo might lead to periods of shut-down. This might be caused by product degradation, segregation, or a blockage of the flow. And these problems might in turn be caused because the material inside the silo is susceptible to the operational conditions in and around the silo [12]. In this chapter, the operational conditions relevant in bulk storage situations are introduced. Additionally, the influence of the operational conditions on the mechanical characteristics of the materials is discussed.

5.1. Definitions of the Operational Conditions

Operational conditions describe influences on the mechanical characteristics that are independent of the material or the handling equipment. These originate with the operational conditions to which the material and equipment are subjected during handling and storage. A couple of different operational conditions important for the analysis of the storage of a material can be identified. These operational conditions are temperature, relative humidity, and stress history, which can be subdivided into handling types and time consolidation [5][6][7]. These operational conditions can influence the material and its behaviour during bulk handling and storage greatly, and they can also influence the various material characteristics that have been discussed in section 2.1.

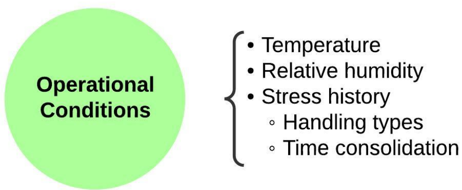

Figure 5.1: Overview of the operational conditions.

Temperature

Temperature is an obvious operational condition influencing the storage conditions of bulk materials. Throughout the day, temperatures can vary quite significantly. This temperature range is subject to conditions like the season, the number of sunlight hours, the cloudiness, the elevation of the location where the silo is placed, the humidity, and the wind speed [80]. The temperature at the location of storage can have a significant influence on the flowability of the material that is being stored. The moisture present in the material might freeze, impacting the flowability. It is also possible that the material undergoes a change in crystallinity due to temperature changes. On top of that, other physical properties can also change due to temperature changes, causing a difference in flow and behaviour of the material [12][14].

Relative Humidity

Relative humidity (RH) describes a ratio of the atmospheric moisture relative to the saturated atmospheric moisture level. The saturated atmospheric moisture level is dependent on temperature, therefore, the relative humidity is dependent on both the temperature and the moisture content [81]. The relative humidity inside a silo, as well as in the interstitial spaces of the material, can also affect the bulk material properties. Many materials take up moisture when subjected to humid conditions, which in turn can change the mechanical characteristics of the material [14].

Stress History

The stress history describes phenomenons like consolidation stress, exerted on the bulk leading to possible adhesion, as well as other changes in mechanical characteristics [12]. These stresses can be caused by a variety of factors, such as the ways in which the material is handled, as well as the time that the material has been stored. The bulk of material weighs on itself, causing a stress. Vibrations due to loading and unloading might also impact the stresses exerted on the bulk material. As a result of this stress, a larger number of contact points between the particles can be distinguished, which in turn can lead to an increased interparticle adhesion [14].

5.2. Influence of the Operational Conditions

It can be expected that the mechanical characteristics of every individual material react differently to the influence of these operational conditions. In Table 5.1, the effects of bulk storage operational conditions on the mechanical characteristics of certain materials as described in literature is presented. As can be seen in Table 5.1, only significant data has been reported on sodium chloride, calcium carbonate and sodium bicarbonate and therefore the other materials that have been identified in chapter 3 have been omitted.

In this table, the colours green, red, yellow, and gray have been used to signify an effect of that specific operational condition on that specific mechanical characteristic of that specific material. Green signifies that when the operational condition increases, the mechanical characteristic increases as well. Red means that when the operational condition increases, the mechanical characteristic decreases. Yellow means that an increase in the operational condition might first increase the mechanical characteristic and later decrease it, or the other way around. Gray means that increasing or decreasing the operational condition does not have an effect on the mechanical characteristic of the material, but it rather stays relatively constant. In sections 5.2.1, 5.2.2 and 5.2.3, these findings have been described in further detail.

Material	PS	BD	BE	CS	TS	AoR	C	F
Temperature								
Sodium Chloride								
Calcium Carbonate				[82]	[82]		[82]	
Sodium Bicarbonate	[83]			[82]	[82]		[82]	
Relative Humidity								
Sodium Chloride	[84]	[20]	[85]	[85][86][87]	[86][87][88][89][90]	[14][20]	[84]	[14][84][20][87][90]
Calcium Carbonate				[82]				[91]
Sodium Bicarbonate	[83]			[82]	[86]		[83][86]	
Stress								
Sodium Chloride		[20]		[85][87]	[87][90][92]	[20]	[93]	[20][87][90]
Calcium Carbonate		[94]		[82]			[82]	
Sodium Bicarbonate	[95]			[82]			[82]	

Table 5.1: An overview of the influence of the operational conditions on the mechanical characteristics.
PS: Particle size, BD: Bulk density, BE: Bulk elasticity, CS: Compressive strength,
TS: Tensile strength, AoR: Angle of repose, C: Cohesion, F: Flowability.

Sodium Chloride

Temperature

- No relevant information could be found on the influence of temperature on the various mechanical characteristics of Sodium Chloride.

Relative Humidity

- **Particle size distribution:** Relative humidity clearly does have an effect on the mechanical characteristics of sodium chloride. Increasing humidity causes particles to take up moisture. Especially smaller crystals tend to absorb moisture during storage due to the relatively higher surface area compared to volume. With a drop in humidity, evaporation causes recrystallizing of the particles. Sometimes the bonds between these particles are so strong, that the barrier between the particles becomes invalid, creating new, bigger particles [84].
- **Bulk density:** An increased relative humidity has a predictable effect on the bulk density of sodium chloride, given the fact that the moisture content of sodium chloride increases when subjected to a higher relative humidity. This can be explained because the moisture takes up the interstitial spaces, whereas before there used to be air in these spaces [20].
- **Bulk elasticity and plasticity** An increased relative humidity leads to an increased moisture content in sodium chloride, which in turn has a positive effect on the crystal plasticity [85].
- **Compressive strength:** The increase in moisture content due to a higher relative humidity causes sodium chloride to have a higher compressive strength, due to an increase in interparticle forces caused by the hydrogen bridges [85, 86, 87].
- **Tensile strength:** Similar to the compressive strength, the tensile strength is increased with a higher relative humidity due to a higher moisture content, and in turn due to an increase in interparticle forces caused by the liquid bridges [86, 87, 88, 89, 90].
- **Angle of repose:** Sodium chloride stored at lower relative humidity showed a lower angle of repose than material stored at higher relative humidity [14, 20].
- **Cohesion:** An increase in relative humidity causes an increase in moisture content, which in turn causes more liquid bridges and an increase in interparticle cohesion [84].
- **Flowability:** An increase in relative humidity during storage decreases the flowability of sodium chloride due to an increased interparticle attraction. On top of that, an increased relative humidity causes an increased angle of repose, which in turn signifies a decreasing flowability. In the same way, an increased level of cohesion between the particles shows a decrease in flowability [14, 84, 20, 87, 90].

Stress History

- **Bulk density:** As a result of compaction, the bulk density of sodium chloride increases over time. This is due to the fact that the material 'settles' and the voids in between the particles become smaller. Therefore, the total volume used to determine the bulk density becomes smaller and the bulk density increases [20].
- **Compressive strength:** An increase in the compressive or crushing strength of sodium chloride has been documented. This is a time dependent increase, where the compressive strength increases with an increasing storage time. It must be said however that this increase in strength is only caused when humidity is present. The compaction forces of the time consolidation make sure the particles are sufficiently close to one another in order for the humidity to cause the formation of liquid bridging between the particles [85, 87].
- **Tensile strength:** For the tensile strength, it has been documented that an increased packing density causes an increase in tensile strength of sodium chloride. As shown before, this increased density can be caused by an increase in humidity, but also by an increase in compaction. Time consolidation causes this compaction in storage, making for an increased tensile strength of the material [87, 90, 92].
- **Angle of repose:** An increasing storage time has been shown to increase the angle of repose of sodium chloride. This is due to the compaction of the material caused by its own weight. This compaction of the material increases over time [20].
- **Cohesion:** Initially, crystalline materials like sodium chloride do not show caking or cohesive effects due to time consolidation. This might however happen when the relative humidity around the sodium chloride exceeds a certain level. At that point, the surface of the particle might dissolve.

When these particles are subjected to stress, possibly due to consolidation, these layers of dissolved material can touch and the material can form strong connections upon drying. Cohesion between the particles has occurred [93].
- **Flowability:** Material flow is impacted by the compaction of the material. This compaction is caused by time consolidation, where the material weighs on itself. This decreases the flowability of the material [20, 87, 90].

Calcium Carbonate

Temperature
- **Compressive strength:** No significant change in the strength of the calcium carbonate material has been observed when exposed to an increase in temperature [82].
- **Tensile strength:** The tensile strength of calcium carbonate agglomerates tends to increase with a higher drying temperature [82].
- **Cohesion:** Given that moisture is present in the initial sample of calcium carbonate, an increase in temperature causes an increase in the formation of agglomerates, or in other words, an increase in cohesion between the particles. This is because a higher drying temperature would cause the moisture to evaporate faster and the material to dry faster. This in turn would mean that the crystals would be less exposed to shearing during this drying. Therefore, the chance for the agglomerates to break again would decrease, meaning an increase in the cohesion between the particles [82].

Relative Humidity
- **Compressive strength:** The strength of the calcium carbonate agglomerates increased with a decreasing moisture content. An increasing relative humidity causes an increase in moisture content in calcium carbonate and therefore a decrease in agglomerate strength [82].
- **Flowability:** For small particles (1 micron), moisture causes agglomeration. In turn, the material forms tiny agglomerated balls that have an increasing effect on the flowability of the material. For larger particles (15 micron), moisture caused the flowability to decrease [91].

Stress History
- **Bulk density:** For calcium carbonate, consolidation stress has a large effect on the bulk density. An increase in consolidation stress causes an increase in the material bulk density [94].
- **Compressive strength:** The strength of calcium carbonate agglomerates is independent of the consolidation stress caused by its own weight weighing down on it, however, agitation stress caused an increase in strength of the calcium carbonate agglomerates [82].
- **Cohesion:** An increase in agitation stress causes the cohesion between the calcium carbonate particles to increase. This is due to the enhancement of the compression and consolidation processes surrounding the calcium carbonate particles, which in turn leads to stronger agglomerates [82].

Sodium Bicarbonate

Temperature
- **Particle size distribution:** The size distribution after drying the samples using microwave irradiation stayed the same, however it is important to note that sodium bicarbonate degrades into sodium carbonate when subjected to high temperatures. This is due to the evaporation of the structural water inside the material. By removing that from the material, the sodium bicarbonate decomposes into sodium carbonate [83].
- **Compressive strength:** An increase in temperature leads to a decrease of sodium bicarbonate agglomerate strength. Although the material shows an increase in the amount of agglomerates, they are of brittle nature and their compressive strength decreases with an increasing temperature. However, the overall crush strength of the material increases due to stronger interparticle connections because of the recrystallization of the material at the higher drying temperatures [82].
- **Tensile strength:** An increasing drying temperature might cause the formation of agglomerates with an increased tensile strength [82].

- **Cohesion:** An increase in temperature causes an increase in the formation of agglomerates. In other words, an increase in temperature increases the amount of cohesion between the particles of sodium bicarbonate [82].

Relative Humidity

- **Particle size distribution:** The average particle size and the particle size distribution showed no significant changes due to a decrease in moisture content. It may be expected that an increase in relative humidity, and consequently moisture content, of the sodium bicarbonate particles shall not influence the particle size distribution [83].
- **Compressive strength:** At a higher temperature, the solubility of sodium bicarbonate increases. If the relative humidity is high, more moisture shall be taken into the material sample, in turn leading to stronger connections between the individual crystals [82].
- **Tensile strength:** The tensile strength of sodium bicarbonate first increases with an increasing relative humidity, but at an intermediate relative humidity, a maximum in tensile strength can be identified, after which the tensile strength starts to decrease again with an increasing relative humidity [86].
- **Cohesion:** An increase in relative humidity causes an increase in moisture content, which in turn causes an increase in the cohesiveness of sodium bicarbonate [83, 86].

Stress History

- **Particle size distribution:** The stresses caused by an increasing compaction stress cause a so-called brittle fracture, which causes the particles to degrade and become smaller. So, an increasing compaction stress causes a decreasing particle size [95]
- **Compressive strength:** An increase in the agitation speed caused a decrease in agglomerate strength, however, the height of the material bed did not have an effect on the strength of the sodium bicarbonate agglomerates [82].
- **Cohesion:** A decrease in material bed height caused an increase in the amount of agglomerates that were formed. In other words, a decrease in consolidation stress caused an increase in the cohesiveness between the sodium bicarbonate particles [82].

5.3. Conclusions

In section 4.1, five materials have been identified that somewhat resemble sodium borohydride. These were calcium chloride, magnesium sulfate, potassium chloride, potassium carbonate and silicon dioxide. In section 5.2, the influence of the operational conditions has been documented on sodium chloride, calcium carbonate and sodium bicarbonate. None of these three materials can be matched to sodium borohydride using the mechanical characteristics. The result of this literature research shows that although the materials that have been studied all consist of inorganic, crystalline salts, the effect that the operational conditions have on the mechanical characteristics of the materials can still vary significantly. Given the limited amount of data available in the scientific literature concerning itself with the effect of operational conditions on the mechanical characteristics of certain materials, it is proposed to increase the effort in obtaining information on the effect of operational conditions by using an experimental setup in a laboratory, rather than commencing another literature research. It is proposed to look further into the relation between sodium borohydride and materials like calcium chloride, magnesium sulfate, potassium chloride, potassium carbonate and silicon dioxide.

6 Conclusion

Throughout this literature research report, the pursuit of knowledge about the operational conditions influencing the mechanical characteristics of various inorganic, crystalline salts has been highlighted. The purpose of this research was to make a prediction on the effect of these operational conditions on the behaviour of sodium borohydride, by comparing sodium borohydride to these other inorganic, crystalline salts.

A breakdown of the well-known mechanical characteristics surrounding the classification of material behaviour has provided a sturdy framework in which this literature research has been conducted. The selected bulk material characteristics; particle size distribution; bulk density; bulk elasticity and plasticity; compressive strength; tensile strength; angle of repose; cohesion and flowability have been introduced in order to provide the reader with a definition to uphold throughout the text.

An analysis of the chemical properties of sodium borohydride helped to guide this research towards a list of relevant, inorganic, crystalline salts. On top of that, another selection criterium used to select these materials was the need to have a significant day-to-day use, with the hope that this would mean an abundance of information of the production and storage of these materials would be available. An analysis of the particle sizes, bulk densities and angles of repose of these materials was conducted in order to construct a framework. This framework could later be used to compare sodium borohydride to the other materials.

An effort was made to compare sodium borohydride to the other materials. Using the particle sizes, bulk densities and angles of repose, sodium borohydride could be linked somewhat to calcium chloride, magnesium sulfate, potassium chloride, potassium carbonate and silicon dioxide. It is however proposed to commence with additional laboratory research to try to match these materials to one another more thoroughly.

The three main operational conditions; temperature; relative humidity and stress history, have been introduced. Only significant data has been reported on sodium chloride, calcium carbonate and sodium bicarbonate. Although the influence of the various operational conditions does have an effect on the selected mechanical characteristics of these materials, no definitive conclusions could be drawn about the effect of the operational conditions on the mechanical characteristics.

For further research, it is proposed to invest more time in the effort to match various materials to sodium borohydride. This could be done on the basis of mechanical characteristics as is done in this research, however, this could also be done using the chemical properties of the materials to match the materials, rather than the mechanical characteristics. Furthermore, it is proposed to commence with laboratory research to try to investigate the effect of the operational conditions on sodium borohydride directly, by subjecting a bulk of sodium borohydride in storage to the various operational conditions while measuring the various mechanical characteristics.

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A
Additional Information

A.1. Search Strategy

An abundance of papers and book excerpts have been analysed in an attempt to gather information on the effect of operational conditions on the mechanical characteristics of various materials. On top of the sources named in Table 5.1, the following papers have been analysed in depth. It was initially expected that these papers would be helpful for the completion of this research, however, these papers did not provide any helpful insights into the subject. An analysis of the search strategy used throughout this report is provided below, to illustrate the effort that has been put in finding relevant source material.

Researched sources: [96] [97] [98] [99] [100] [101] [102] [103] [104] [105] [106] [107] [108] [109] [110] [111] [112] [113] [114] [115] [116] [117] [118] [119] [120] [121] [122] [123] [124] [125] [126] [127] [128] [129] [130] [131] [132] [133] [134] [135] [136] [137] [138] [139] [140] [141] [142] [143] [144] [145] [146] [147] [148] [149] [150] [151] [152]

Throughout this literature research, the helpful tips available on the TU Delft library website have been used to structure the searching process [153]. A search plan was created and a list of important terms and their synonyms was constructed. Search queries that were used throughout this research looked as follows:

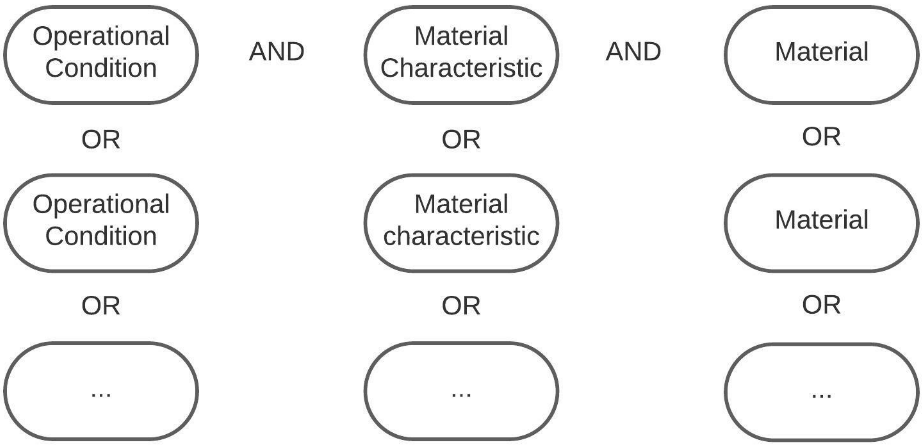

Figure A.1: Search queries.

An example of this is given below:

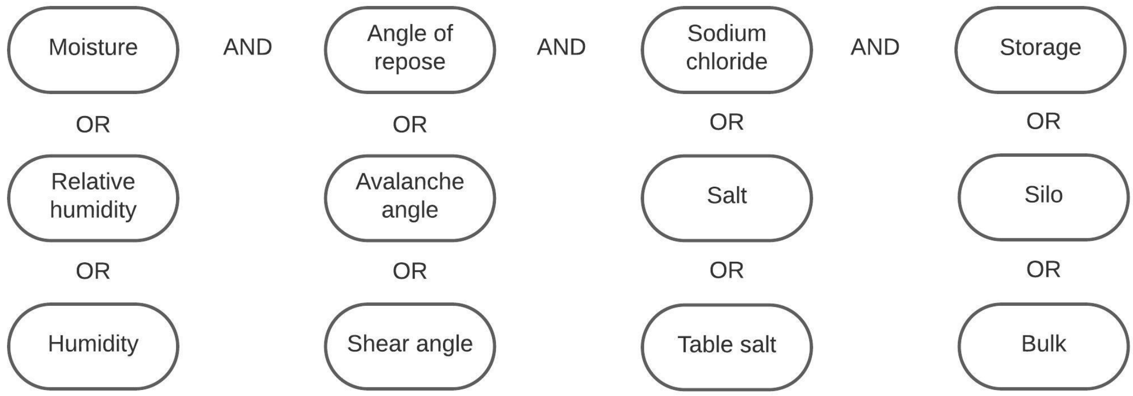

Figure A.2: Example of used search queries.