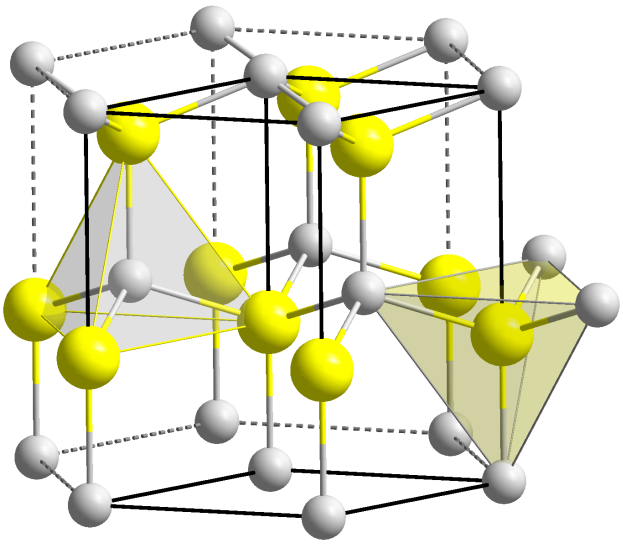

Material analysis of recycled zinc from incinerated waste ash

Analysis of recycled zinc from fly ash of a waste to energy plant with the help of powder X-ray diffraction

Master's thesis in Sustainable Energy Systems

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Department of Architecture and Civil Engineering

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Cover: Crystal structure of zinc oxide where the yellow atoms represent zinc and the gray represent oxygen. The picture is from[1], public domain.

Abstract

With the EU increasing costs for landfill and incentivising recycling it has become more and more common to try to recycle as much as possible from waste products such as fly ash. This is especially the case for waste-to-energy (WtE) plants that have fly ash with high contents of valuable metal ions. A WtE plant company in southern Sweden has started a project regarding the recycling of zinc (Zn) from fly ash. Recently together with other partners a production chain was designed and built to recycle Zn from the fly ash was designed. The principle is based on two main reactions, the first is mixing fly ash from the electrostatic precipitators with acidic water from the open scrubber in the flue gas treatment system. This is to dissolve metal ions in the fly ash into the water via leaching. The remaining solid is then filtered and reentered into the furnace. The second step is meant to produce the solid Zn in the form of zinc hydroxide ($Zn(OH)_2$) via precipitation. By mixing the acidic water from the first reactor with sodium hydroxide (NaOH) the pH increases, which theoretically should promote the formation of $Zn(OH)_2$. However, investigations on the Zn product have shown that the extracted form of Zn is not purely $Zn(OH)_2$ even if the facility operates under optimal conditions. The focus of this project has therefore been to investigate what compounds the extracted product is made out of and if parameters such as pH for the precipitation reaction and the liquid to solid ratio in the first leaching step have effects on the composition. The main method that has been used during this thesis is powder X-ray diffraction (P-XRD), more exactly Bragg-Brentano analysis which is heavily based on Bragg's law. It turned out that the original wet Zn product (about 30% DS) consisted of mostly amorphous material which can't be detected via P-XRD. The only compound identified was gypsum. By drying the sample the amorphous part of the sample decreased and a more crystalline structure appeared. In the dried sample sodium chloride (NaCl) could also be identified in the sample together with gypsum. When drying the samples at $105°C$ the gypsum was converted into anhydrite but there were still no zinc compounds found in the sample. By drying the sample at $250°C$ a much clearer diffractogram could be extracted that contained the missing Zn, however it was not possible to differentiate between the different Zn compounds such as $Zn(OH)_2$, ZnO and different metallic zinc oxides such as zinc iron oxide since they all form similar diffractogram patterns. The total composition analysis of the zinc product showed that about 40% of the product was zinc and the rest was other elements such as sodium and chlorine. Chlorine is an element that easily forms corrosive compounds and is therefore unwanted in the final product. To test if the chlorine is easily removable an extra cleaning step was tested to try to remove chlorine from the sample. The diffractogram showed no presence of sodium chloride and the liquid contained high amounts of chlorine. To further investigate what different zinc compounds that were present in the product another analysis method must be used. It could also be beneficial to investigate if the amorphous zinc compounds are the same as the crystalline ones. The fact that the Zn is not in the form of $Zn(OH)_2$ might not be a big problem since compounds such as ZnO also can be treated and converted into pure Zn. The fact that the Cl easily can be removed is very good for the further treatment of the Zn product.

Contents

1 Introduction

In today's society there are few subjects that are as well studied as electricity and heat production. One of the major technologies when it comes to both heat and electricity production is thermal power plants. In fact more than 60% of the electricity and 80% of the total energy produced in 2020 was produced by thermal power plants [2]. Thermal power plants are plants that convert fuel into heat to produce superheated steam that can then be used for electricity and heat production. This is generally done via combustion of fuel in a furnace/reactor that is surrounded by pipes with flowing water. The heat from the combustion heats up and evaporates the water, the steam is then separated and heated up additionally with the excess heat from the flue gases. The steam can be passed through a turbine to produce electricity and heat can be produced with the residual heat[3].

The standard fuel that has been and still is the primary fuel for thermal power plants is fossil fuels [4]. As the environmental impact of these processes gets more and more important other technologies and fuels than fossil fuels are increasing usage. One such technology is the solid waste thermal power plant or so-called waste-to-energy (WtE) plants which use municipal or industrial solid waste as fuel. There are several advantages when it comes to using waste as fuel, e.g. there is less need for treatment and disposal of the waste and the fuel is also very cheap because there will always be a continuous production of waste that has to be treated/taken care of. There are however a lot of regulations and factors to take into consideration regarding municipal waste (MSW) combustion such as what types of emissions and in what amount are allowed. The European Union also has a lot of regulations regarding the disposal of waste and landfill which makes MSW combustion attractive but an advanced flue gas treatment system is required [5], the European Union also subsides the use of MSW as fuel to make it more competitive with other fuels. MSW has a comparatively low heating value compared to fossil fuels and biofuel which makes it harder to produce high-temperature steam [6]. This can in turn affect the electrical efficiency of the plant. This does not decrease the total efficiency of the plant however, instead a larger share of district heating can be produced. Because of this municipal solid waste power plants are more common in regions where there is a large heating demand. In difference to most other fuels, municipal waste comes in very different sizes and different properties such as moisture content which makes it very different from other commonly used fuels. This can make it harder to design a fluidized bed boiler for incinerating the waste which is the most common type of boiler. Instead a type of reactor that allows for longer residence time and can take a more diverse weight of fuel to make sure complete combustion occurs.

One of the major challenges for a lot of thermal power plants is the treatment of rest products,

most commonly ash. In a power plant there are different types of ash produced depending on what types of fuels are burned. Often two types of ash are produced, so-called bottom ash and fly ash. Bottom ash is made up of heavier chunks of metal, inorganic minerals and uncombusted fuel pieces, and fly ash contains lighter elements and metals [7]. The fly ash is light enough to get carried away by the airflow through the reactor and follows the flue gas through the flue gas treatment system until filtered out. In Figure 1 a simple schematic over a waste to energy power plant and the ash production can be seen. In the picture the bottom ash is extracted from the bottom of the furnace and extracted to the right, and the fly ash is extracted at several points during the flue gas treatment process as indicated by the gray arrows in the middle.

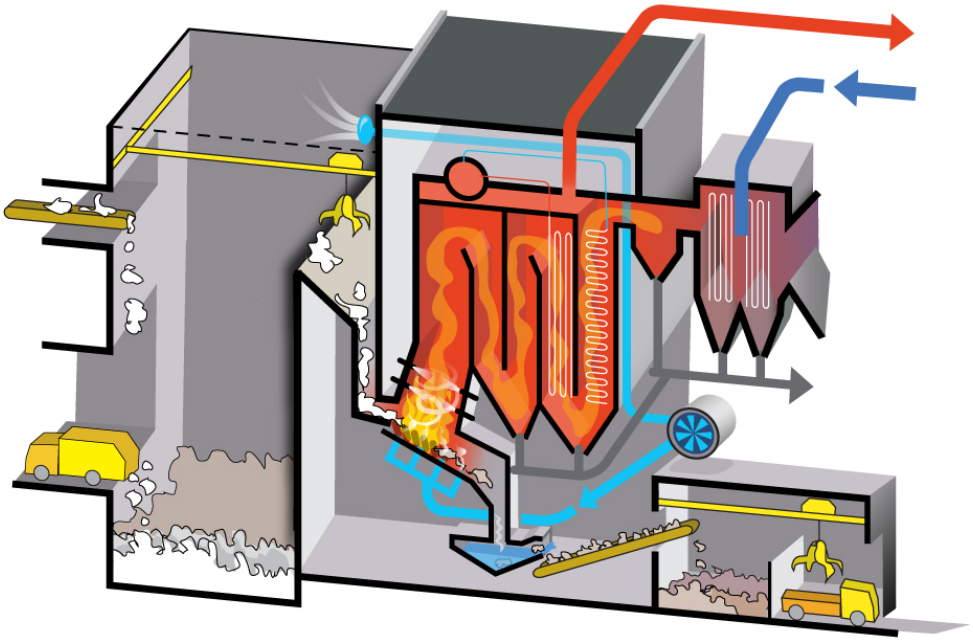

Figure 1: fly ash generation and collection in pulverized coal-fired power plant, with permission from [8]

The ash has to be disposed of in some way and in Sweden bottom ash is used for mostly landfill cover construction [9]. When it comes to the handling of fly ash there are not as many options to go for and it is most commonly landfilled. But as the cost for landfill is increasing and the incentive for recycling is increasing a lot of research on ash recycling is being made [5]. This is especially the

case for WtE plants since the rest product from these plants can contain a lot of valuable metals due to the composition of the fuel. Studies on a WtE plant in southern Sweden show that the fly ash contains a lot of metals such as Zn, magnesium (Mg) and (Na) [10] [11]. When comparing this with the list of endangered elements put out by the American Chemical Society there is a clear incentive to recycle the materials in fly ash from waste to energy plants [12], a picture over the whole list of endangered elements by ACS can be seen in picture 2.

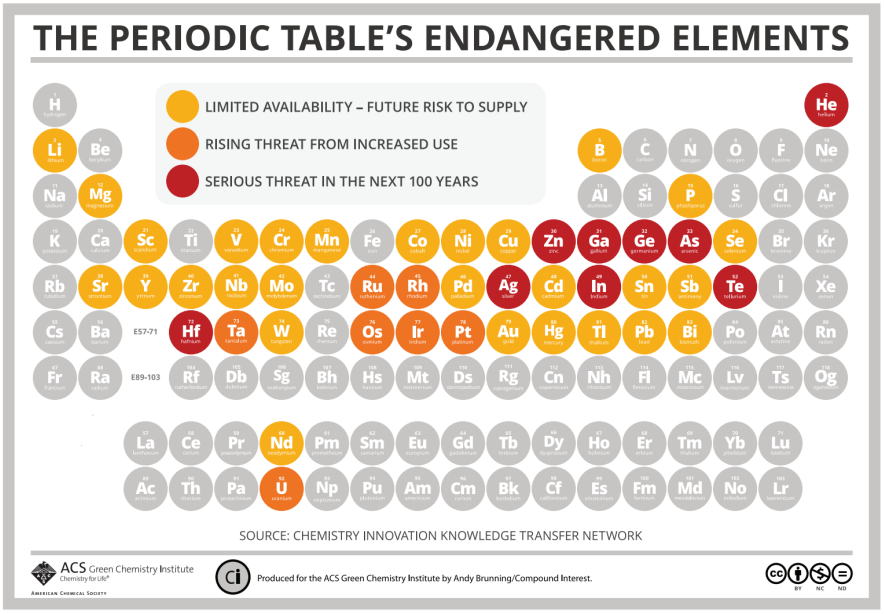

Figure 2: ACS periodic table of endangered elements

This project was done in collaboration with a waste and recycling company that runs a waste to energy plant that incinerates both municipal and industrial waste in southern Sweden. That together with partners, a production chain has been designed and built to recycle Zn from the fly ash. The purpose of the Zn recycling facility is to extract zinc from the fly ash and precipitate it as zinc hydroxide.

1.1 Aim and goal

The project will focus on the Zn product extracted from the precipitation process in the zinc recovery plant. The main goal of this thesis was to investigate if and how major operating parameters of the

zinc recovery facility i.e. liquid to solid ratio during ash leaching and pH for precipitation affect the composition of the final product. Some extra experiments were conducted such as testing the effect of the drying temperature of the product, i.e. how this affects the moisture content of the sample and if it has some implications on the composition of the product. Since the goal of the recycling process is to produce a product that can be used to produce high purity. Elements such as Cl have a major impact on the ability to retreat the product since Cl often creates corrosion compounds that are unwanted in reactors [13]. Because of this it was also investigated how further cleaning can impact the final product's composition in regards to unwanted elements such as chlorine.

1.2 Formulation of research questions

The questions that will be analyzed in the report are:

- How does the liquid to solid ratio in the leaching stage and pH for precipitation affect the composition of the zinc product extracted from the zinc recycling plant, is it possible to optimize these in a way to increase the purity of the product?
- What different types of compounds can be found in the Zn product and do these change with the operating parameters mentioned before?
- Can a second washing or filtration reduce the amount of Cl present in the product?
- What effect does the drying temperature have on the produced product and how long does it take for the sample to dry in different temperatures?
- Can an assessment be made on what sources for Zn in municipal and solid waste and what implications do that have for future usage of Zn?

1.3 Limitations

The work will be done in cooperation with a WtE plant in southern Sweden and it is the Zn product extracted from the Zn recycling facility that will be analyzed. Operating parameters that have previously been suggested to have a big impact on the extracted product will only be investigated. The main method for analyzing the samples will be powder X-ray diffraction, other complementing analysis methods will be mentioned but not used in this report. Since the project only will be done laboratory scale the ethical and environmental impact is very small or negligible. If discovered that a second cleaning step is beneficial and that a high drying temperature is needed the main process

might become more energy intensive. It is important to note that the Zn recycling plant is part of a bigger process and that it uses products from the bigger processes, it is therefore hard to adjust the operating parameters significantly even if it could yield clearer results.

2 Theory

The theory section will be divided into two main sections. The first section will be covering the basics of how the waste to energy plant operates. The following sub-section will then go a little bit deeper into the chemistry behind the Zn recycling section. The second section will go in depth into the physics and chemistry behind crystal structures and then also how these crystal structures can be analyzed with the help of X-ray diffraction.

2.1 The WtE process

Since the Zn recycling plant is coupled together with the rest of the plant and uses products from the process a short rundown of the plant has to be made. In this sub-section all the information is taken from the same source unless stated otherwise [14]. The plant is a medium-sized plant and gathered a total of 1266100 tons of waste during 2021, this is the total amount of waste gathered and not all of it was used for combustion. During the same year a total of 1441000 MWh of district heating and 200000 MWh of electricity was produced. The facility consists of four grate-fired boilers that run at about 1000 °C which is then used to produce saturated steam. The hot flue gases then passes through a number of superheaters that heats up the produced steam to a pressure of about 40 bars and a temperature of about 400 °C. Most of the heavy parts after incineration such as unburnt fuel, metal chunks and slag stay on the grate and are taken out at the end as bottom ash. Most of the volatile elements (such as Zn- and Al compounds) are carried away with the airflow in the flue gas. A lot of the elements in the flue gas are considered hazardous such as NO_x-formations and even mercury (Hg) compounds and it must therefore be taken care of before the flue gas leaves the facility [15], this is the reason for the need of an extensive flue gas treatment system. First the flue gas passes through an electrostatic precipitator which uses potential in the charges of the particles in the flue gas. By having a plate with a charge that the flue gas passes by, the particles in the flue gas with the opposite charge are attracted to the plate by the electrostatic force [16]. The accumulated particles on the plates can then be dropped by shaking the plate or similar. This procedure can collect about 99.5% of particulate matter in the flue gas. The ash that drops down can be collected and is called fly ash. The flue gas is then passed through an open scrubber where it is sprayed on the flue gas. This allows compounds such as hydrochloric acid and heavier metals that were left from the electrostatic filter to precipitate into the water. This removes the compounds from the flue gas but it creates water that needs to be treated instead. Lastly sulfur is separated from the flue gas with the help of NaOH.

2.2 The Zn recovery process

In Figure 3 a schematic over the Zn recycling process can be seen. The section numbered one in the picture is the electrostatic precipitators and the fly ash is collected at the bottom. The next section is the open scrubber, the acidic waste water is extracted and mixed with the fly ash in the first step in the Zn recovery section, which will be presented in the following sub-section

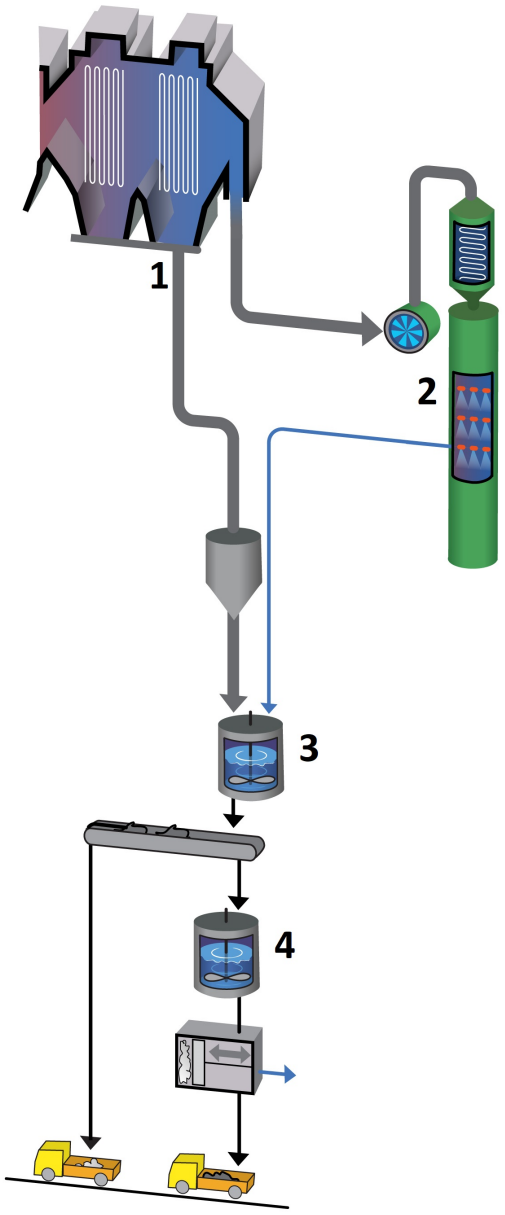

Figure 3: Schematic over the Zn recovery facility, with permission from [8]

Studies made on the fly ash from the electrostatic precipitator show that there is a lot of Zn present in the ash and that it was possible to recover much of it[17]. This led to a project regarding the

recycling of Zn full-scale starting where a Zn recovery plant was constructed where the goal was to produce a zinc hydroxide as a product. The plant mainly consists of two reactors where the first reactor is a continuous reactor that mixes the fly ash from the electrostatic precipitator with the acidic washing water from the open scrubber. The slurry is then filtered and the extracted solid is reentered into the furnace. the water from the open scrubber does contain small amounts of Hg, and therefore it has to pass through a mercury ion exchanger first. The water from the first reactor is then passed on to a second reactor where it is mixed the basic water from sulfur cleaning containing. The process can be seen in picture 3 where the first reactor in the zinc recovery process is marked with a 3. The last reactor is where precipitation of the $Zn(OH)_2$ happens.

The main purpose of the first reaction in the zinc recovery process is to make the Zn in the fly ash dissolve into the water and form Zn-ions [8]. By adding the fly ash to an acidic solution with a high concentration of H^+ the positive Zn-ions dissolve into the water. The most common type of zinc ion is Zn^{2+}. By dissolving the Zn and other metals into the water it is possible to remove unwanted compounds that do not dissolve via filtration. The extracted solid from the filtration is removed and reentered into the furnace [8]. Since the Zn is ionized state and solved in the water the Zn is not extracted from the solution in the filtration. To precipitate the Zn $NaOH$ (lye) is mixed with the acidic water to increase the pH of the water. When $NaOH$ is mixed with water it creates hydroxide ions (OH^-) and sodium ions (Na^+). By mixing the acidic Zn water with the lye wastewater the hydrogen ions and hydroxide ions create water as shown in equation 1.

$$H^+ + OH^- \rightarrow H_2O \tag{1}$$

By mixing specific ratios of the ash leachate and the $NaOH$ a desired pH can be achieved which allows for the creation of $Zn(OH)_2$. studies show that Zn can be precipitated as several different types of hydroxides dependent on what pH the precipitation occurs at [18]. The same study mapped what type of Zn was present at specific pH levels. The study showed that the ideal pH for precipitation of zinc hydroxide was 10, the reaction can be seen in equation 2.

$$Zn^{2+} + 2OH^- \rightarrow Zn(OH)_2 \tag{2}$$

Finally the achieved slurry is filtered via a filter press and washed, the extracted product should be a concentrated solid $Zn(OH)_2$. Since there are more compounds present in the solution than just Zn there are other reactions that also have to be taken into account such as the formation of other hydroxides e.g lead hydroxide [19] and the formation of zinc chlorides [20]. The reaction of

lead hydroxide and ZnCl can be seen below in reactions 3 and 4 respectively

$$Pb^{2+} + 2OH^- \rightarrow Pb(OH)_2 \tag{3}$$

$$Zn^{2+} + Cl^- \rightarrow ZnCl_2 \tag{4}$$

2.3 Crystal structures

A crystal structure is a type of solid in which the atoms are arranged in a highly ordered and repetitive pattern. This regular arrangement of atoms leads to a characteristic geometric shape of the crystal, such as a cube, octahedron, or tetrahedron [21]. The regularity of the crystal structure also results in its characteristic cleavage and fracture patterns, which are predictable based on the orientation of the crystal lattice. In crystallography, a unit cell is the basic repeating unit of a crystal lattice. It is the smallest unit that, when repeated in all three dimensions, generates the entire crystal structure. The dimensions of the unit cell are defined by its lattice parameters, which describe the length of the edges and the angles between them. The lattice parameters are typically represented using three values: a, b, and c, which represent the lengths of the edges of the unit cell, and three angles: α, β and γ which represent the angles between these edges. Together, these six parameters fully define the crystal structure. There are several different types of unit cells, depending on the geometry of the crystal lattice. The most common types are cubic followed by face-centered cubic and also body-centered cubic. But unit cells of crystals do not only come in cubic shapes they can be orthogonal, hexagonal and much more. Each of these unit cells has a different set of lattice parameters, which determine the symmetry and overall shape of the crystal structure. So there might be several cubic crystals but since all atoms have different bond strength no cubic crystal structure has the same lattice parameters as another. This is what gives each solid its characteristics and properties. Crystals can be further categorized based on the type of atomic bonds that hold them together. Ionic crystals, such as table salt, are held together by electrostatic forces between positively and negatively charged ions. Covalent crystals, such as diamonds, are held together by strong covalent bonds between atoms. Metallic crystals, such as copper, are held together by metallic bonds, which are a type of delocalized electron bonding.

Not all solids form clear crystal patterns, some solids form irregular patterns where the atoms and molecules are randomly organized. These solids are so-called amorphous solids. Because of the irregular structure it becomes very difficult to determine the overall structure of amorphous

solids using techniques such as X-ray diffraction. Figure 4 shows a comparison of a crystalline and amorphous solid and XRD is discussed in detail in the following chapter.

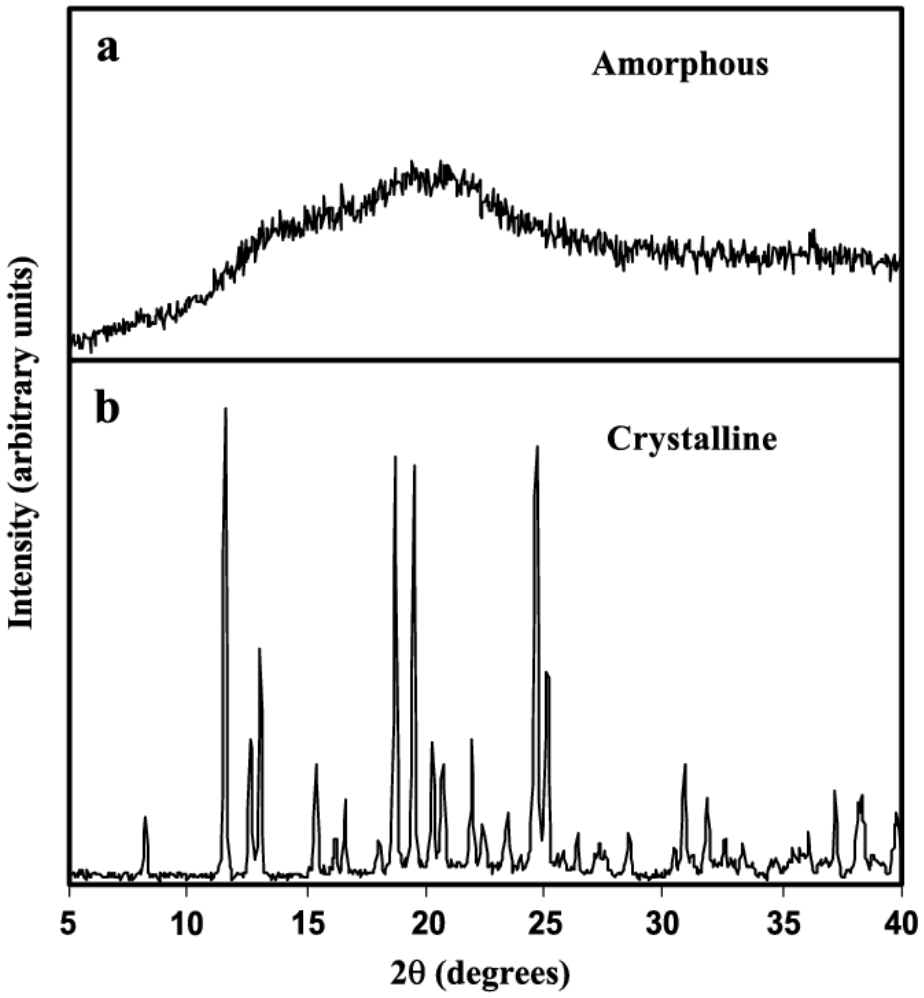

Figure 4: amorphous versus crystalline materilas

2.4 Powder X-ray diffraction

Powder X-ray diffraction (PXRD) is a technique commonly used to study different crystal structures. The basic principle of P-XRD is that X-rays are diffracted by the atoms in a crystal in a way that produces a characteristic diffraction pattern, which can be used to determine the crystal structure of the material. A sample is illuminated by an X-ray source, the source sends out X-rays with a

specified wavelength, amplitude and frequency. The amplitude can be seen as the height of the peak of the wave but also the depth of the trough, amplitude is often denoted A. The wavelength is commonly referred to as λ and is the distance between two peaks of the wave. The frequency is the time between two peaks on the wave, meaning it is the time it takes for the wave to travel exactly one wavelength. The relation between frequency and wavelength can be written as in Eq5 where c denotes the speed of light, f is the frequency and λ is the wavelength.

$$c = \lambda f \tag{5}$$

In Figure 5 a representation of the different parameters can be seen.

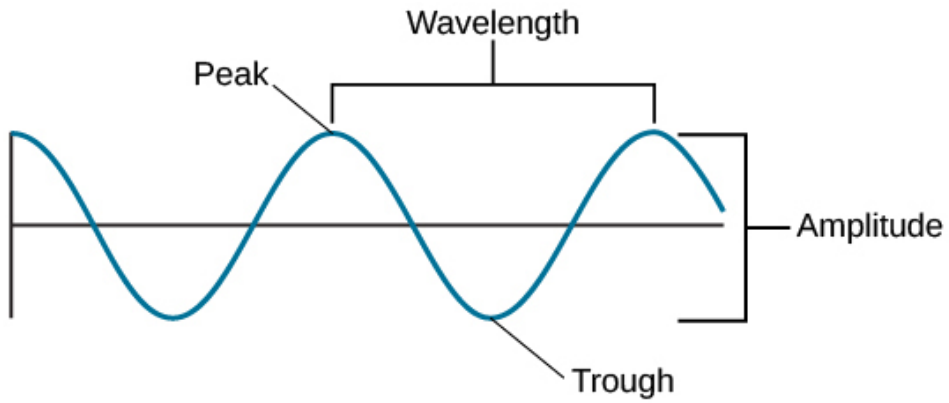

Figure 5: Parameters in an X-ray

When the sample is illuminated by X-rays they are reflected and the X-rays scatter which means they spread out in all directions when bouncing off the surface of the sample[22]. The scattered X-rays can interfere with each other in so-called interference. Since the waves have the same frequency and therefore also wavelength they can interfere with each other in such a way that they amplify each other in so-called constructive interference. This is when the peaks of one wave line up with the peaks of another and they get added together in so-called superposition and form a wave with higher amplitude but the same frequency. The same phenomena can happen the other way around where the peak of one way lines up with the trough of another and via superposition the two waves eliminate each other. This is illustrated in Figure 6 which show two waves with constructive interference to the left and two waves with destructive interference to the right.

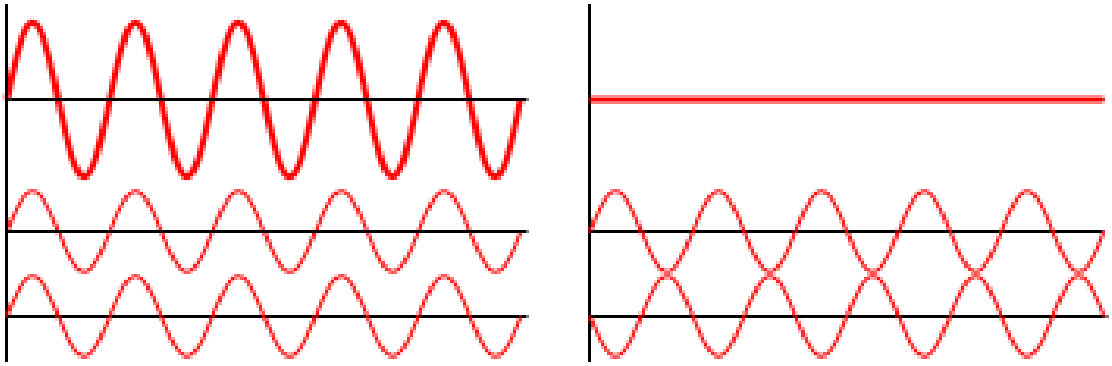

Figure 6: constructive and destructive interference

It is important to note that photons can not only be seen as waves but also particles [23]. In P-XRD there is a detector on the other side of the sample that counts how many photons hit the detector. If there is constructive interference there are a lot of photons hitting the detector and there is a so-called spike. Doing a powder X-ray diffraction analysis over time yields a plot where the amount of particles detected at a certain point is correlated to the angle of the source and the detector, a so-called diffractogram. A simple diffractogram over $Zn(OH)_2$ can be seen in Figure 7.

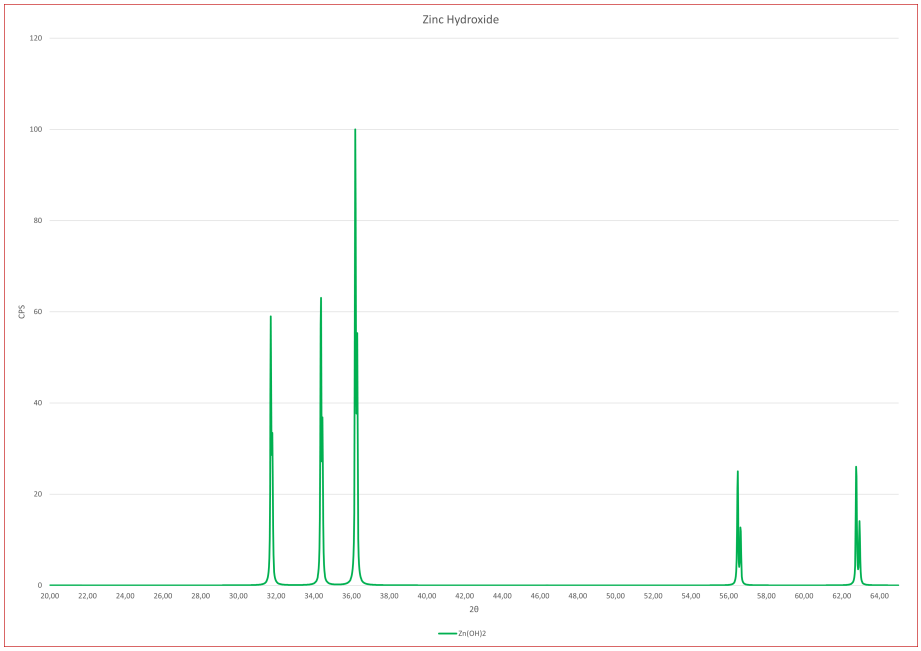

Figure 7: Diffractogram of $Zn(OH)_2$, from [24]

2.4.1 The Bragg-Brentano analysis

Bragg-Brentano analysis, also known as the Bragg-Brentano geometry or the Bragg-Brentano diffractometer, is a common method used in powder X-ray diffraction (P-XRD) experiments to determine the crystal structure of materials. This method is named after the British physicist Sir William Henry Bragg and the Italian physicist Amedeo Avogadro Brentano, who developed the geometry of the diffractometer in the early 1900s.

The Bragg-Brentano analysis involves mounting the powdered sample on a fixed horizontal plane and aligning it perpendicular to the X-ray beam. The X-ray beam is then directed onto the sample at a specific angle (known as the incident angle, Θ), and the diffracted X-rays are collected on a detector placed on the opposite side of the sample. The detector is placed on the other side of the sample so that the reflecting X-rays from the sample hits the detector. The generator can change the incident angle by moving in a circular motion, the detector can then also match the incident angle at all times to perform a so-called coupled 2Θ scan.

The Bragg-Brentano analysis is based on the Bragg equation 6, which states that the X-rays will be diffracted at a specific angle if the distance between the atoms in the crystal lattice matches the wavelength of the X-rays. By measuring the angle of incidence and the diffracted angle, the Bragg-Brentano geometry allows for the determination of the crystal lattice spacing, which can be used to determine the crystal structure of the material. A figure showing the principle of Bragg's law can be seen in Figure 8.

$$n\lambda = 2dsin\Theta \tag{6}$$

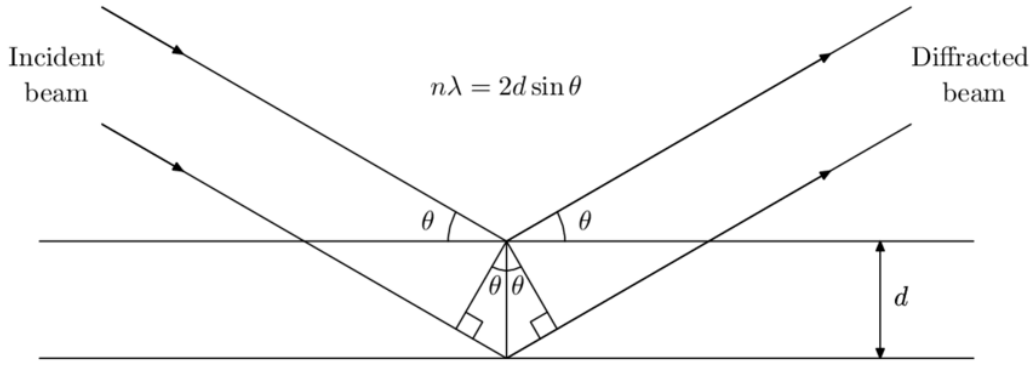

Figure 8: visualization of Bragg's law from [25]

Because of the simplicity of Bragg's law the Bragg Brentano analysis is very efficient at gathering large amounts of data in a short time. However, this method has limitations, as it is susceptible to the effects of preferred orientation, which can result in inaccurate diffraction patterns. It also becomes harder to predict the intensity of the diffractograms when using Bragg-Brentano analysis when analyzing multicomponent crystals because of other crystal structures blocking each other. Therefore in this study the analysis method will be used to identify the presence of a sample and not to quantify the share of that sample in the product [26].

The main use for Bragg-Brentano analysis and most other powder X-ray diffraction methods is that the derived diffractogram can be compared to already existing diffractograms of known compounds in databases. By matching the peaks on the achieved diffractogram to different compounds in the database one can effectively derive which compound/compounds the sample consists of.

2.5 Ion chromatography

Ion chromatography is a way to analyze the concentrations of specific ions in a liquid. The mobile phase or the so-called eluent is pumped through an injection valve with a small pump[27]. An exact amount of the sample is injected into the eluent through the injection valve. The ions in the sample are then carried away to a separation column with the eluent. In the separation column the sample ions react with fixed ions of opposite charge, negative if positive ions are to be analyzed or the other way around. The interaction between the ions makes the sample ions slow down in the eluent and then pass through a suppressor. The suppressor both increases the sensitivity of the sample and reduces the background of the eluent. The eluent then passes through a detector and the data can be evaluated and compared to known patterns in a database.

3 Methods

Total composition analysis of selected elements in the zinc samples was done using total digestion followed by inductively coupled plasma optical emission spectrometry according to the ISO standard [28]. This test was not included in this project and will not be further explained but the results were used. All samples that were gathered before and after the experiments were analyzed via P-XRD, more information regarding the operating parameters can be seen in section 3.2.

3.1 Experimental methods

3.1.1 Drying temperatures affect on the material composition of the zinc product

To test how much water was present in the original samples and what affect this had on the diffractograms, and also the possibility to decrease moisture content the samples were dried in different temperatures. Before drying the samples were weighed so the amount of water evaporated could be calculated. The different drying temperatures that were tested were $50°C$, $105°C$ and $250°C$ for 24 hours respectively, a test was also made where the samples were left to dry under the fume hood to simulate room temperature drying which was left for drying for 48 hours.

3.1.2 Removal of chlorine by washing

Since earlier P-XRD tests indicated the presence of sodium chloride the possibility of adding a third cleaning step to the cleaning process was investigated. The purpose of this step was to remove chloride from the product. The product was mixed with milli-Q water and stirred until a fine slurry was made, the exact weight of the sample and amount of milli-Q water used for the dilution process can be seen in Table 1. The liquid with the dissolved sample was then filtered through a simple filtration setup with a $1{,}6\mu m$ filter. The residue from the filtration was left to dry while the filtrate was analyzed via ion chromatography (IC). The conductivity was measured on the filtrate and the liquid was then diluted to have a conductivity at around $300\mu S$. The dilution factor can be seen in Table 1. Two base samples containing only milli-Q water were also prepared to calibrate and reset the machine after usage. To ensure that the chlorine was removed in the experimental cleaning step the sample was not only analyzed via XRD, instead the filtrate from the process was analyzed with ion chromatography as well as the sample was analyzed with XRD. This was done to ensure that the chlorine was removed from the sample as intended if there were no NaCl crystals left in the sample, otherwise there could always be a possibility that the chlorine formed other compounds than NaCl instead.

Table 1: Data regarding filtration and sample preparation for IC

	Sample 1 (25/1)	Sample 5 (10/3)
Mass sample [g]	20.70	24.02
Mili-Q water [ml]	180	180
dilution factor	32	40

3.2 Analytical methods

3.2.1 Powder X-ray diffraction

The powder X-ray diffraction analysis that was performed was all Bragg-Brentano analyses, the equipment used was D8 discover Bruker which can be seen in picture 9. The generator used was a copper generator with a voltage set to 40 kV and a current set to 40 mA. The primary optic used was a motorized divergence slit with a fixed sample illumination with a length of 10mm. The slits used was nickel filter with 2.5° soller slit. The detector was an Eiger 2R 500K with a region of interest set to 100 times 300 pixels, the analysis was made in 1D mode. the sample was to have a rotation speed of 10rpm and the AirScatter was set to fixed with 2mm. The scan was a Coupled two theta with a starting angle of 20° and a finish angle of 65°. The increment was set to 0.02° with a time per step set to 2 seconds. The sample holders used were hollow puck holders. All the different diffractograms for the different compounds in this study were taken from [24]. In 9 a picture over the D8 Discover Bruker can be seen.

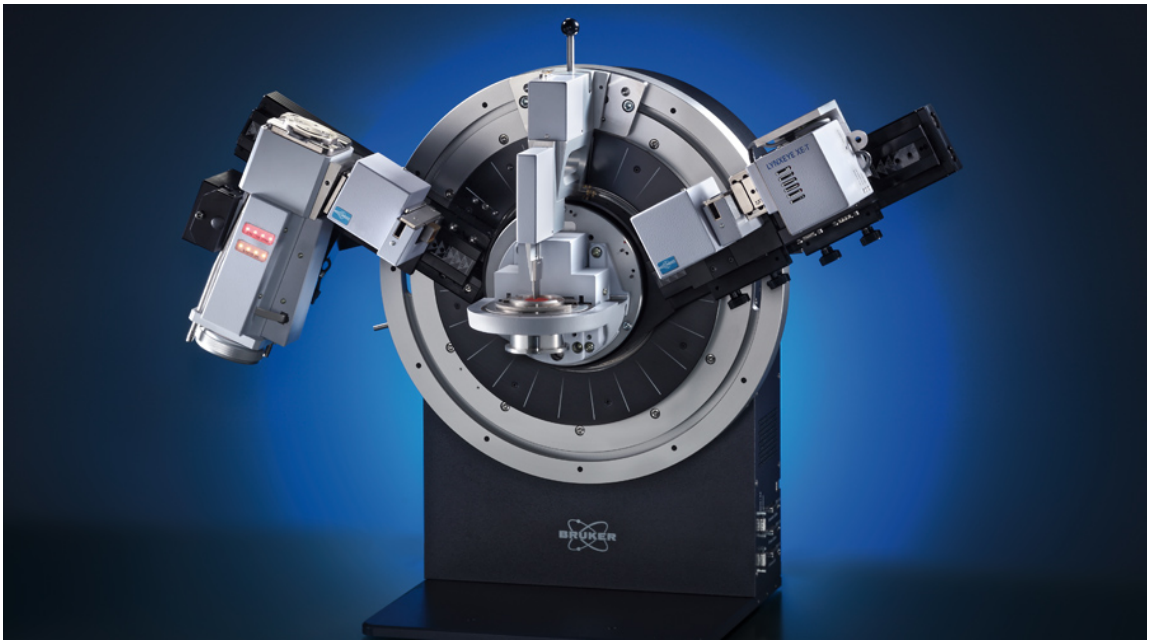

Figure 9: D8 discover Bruker

3.2.2 Ion chromatography

For the chromatography to yield optimal results the conductivity of the liquid sample should be around 300µS. This means that filtrate might have to be diluted, when diluting it is important to use as pure water as possible since chlorine is often present in both tap water and even in some deionized water. Before the liquid was analyzed it was filtered through a 0,45µm filter to make sure no harmful particles from the sample could enter the detector.

The liquid extracted from the filtration was gathered and the conductivity was measured. The sample was prepared for ion chromatography by first diluting it with milli-q water so the conductivity was around 300µS. Two samples with only milli-q water was prepared, one to use before the samples and one after. The first is to calibrate the machine and the latter is to rinse the system.

4 Zinc in municipal and industrial solid waste

All the information in this section has been taken from the U.S department of Health and human services article on zinc toxic profile [29]. Zn is a naturally occurring element that has a wide variety of uses such as construction, electronics, pharmaceuticals, etc. Zinc is also an important nutrient for humans (even if in small amounts) which means that it occurs in several different human wastes as well or so-called municipal waste. Municipal waste refers to the waste generated by households, institutions, and commercial establishments within a municipality. Some of the sources for zinc in both municipal and industrial waste are presented below.

Consumer Products: The main source for Zn in consumer products is different types of electronics such as batteries, remotes, toys and even in small amounts in computers and mobiles. But also different types of household items such as washing machines, dryers, and refrigerators among others. Some consumer products may also contain some amounts of ZnO such as sunscreen and skin care products.

Industrial Waste: One of the major uses for Zn in industries is galvanizing which is the process of applying a thin coat of Zn on metal or steel as a protective coating. This is also one of the major sources of Zn waste in the industrial sector, products such as nails are often galvanized. Other industrial products containing zinc can be paint and the production of ZnO for various commercial uses.

Food Waste: Zn is present in small amounts in various foods, and the waste generated from food consumption can contribute to the Zn content in municipal waste. Foods that are high in Zn include meat, seafood, dairy products, and whole grains. The waste generated from food preparation and consumption, such as food scraps and packaging, can contain small amounts of Zn.

Medical Waste: Zn also has a large use in hospitals and clinics. Zn can be used in different equipment and devices. But also more disposable materials such as surgical gloves, syringes etc but in the form of ZnO.

Batteries: Zn is a common component in various types of batteries, including alkaline, zinc-carbon, and zinc-air batteries. These batteries end up in municipal waste and can contribute to the overall Zn content. Batteries contain Zn as an anode, which corrodes over time and eventually becomes waste.

Automotive Waste: Zn is used in the production of tires, brake pads, and other automotive components. The waste generated from the maintenance and repair of automobiles can contribute to

the Zn content in municipal waste. Automotive waste also includes parts such as exhaust systems and catalytic converters, which may contain small amounts of Zn.

Some of the main results that can be drawn from this analysis are that there is no clear single source of Zn in either municipal or industrial waste, instead there are several independent sources for the Zn being present in the waste. From this some conclusions can be drawn. The usage of Zn will most likely be of huge importance for a long time forward, even if Zn stops being used in batteries for example there will still be steady demand for Zn in other products. Because of the highly varied usage of Zn there will be a steady flow of Zn in the waste. factors such as share of industrial waste to municipal waste might not have a big impact on the final amount of Zn in the product since both municipal and industrial waste has several sources of Zn.

5 Results and Discussion

To get a better understanding of what possible compounds could possibly exist in the samples were a total composition analysis performed on all samples. This analysis does not give any indication of what compounds are present in the sample, it only gives the concentration of specific elements in the sample. During the gathering process of the samples the mercury ion exchanger mentioned in section 2.2 was out of order. This led to the samples gathered having small amounts of mercury inside them that is not usually present. The amount is so small that it should not affect the other compounds in the sample in a significant way. The result of the total composition analysis can be seen in Table 2 where the average mass per kilogram over all the samples and the standard deviation are presented. The average mass per kilogram has also been converted to moles/kg to ease the understanding of the composition. The amount of water in the samples where also analyzed and it turned out that the samples contained about 71% water. The other analysis was made on the dry sample so the weight concentrations and moles concentrations are based on dry weight. A visualization of the average share of each element in weight percent can be seen in Figure 10 and mole percent can be seen in Figure 11.

Table 2: Overview of the average composition of the different Zn samples

element	mg/kg	moles/kg	standard deviation mg/kg
Cl	85100	2.4	3900
S	10100	2.4	3700
Al	11400	0.42	3400
P	542	0.017	124
Fe	4520	0.081	676
Cd	1940	0.017	350
Ca	33200	0.83	836
K	23200	0.59	6380
Si	60800	2.2	25000
Mg	45600	1.9	9550
Mn	1700	0.031	160
Na	38800	1.7	8610
Ti	642	0.013	222
As	1064	0.0012	411
Sb	788	0.0064	312
Ba	206	0.0015	82
Be	0.584	$6.5 \cdot 10^{-5}$	0.183
Pb	7120	0.034	1560
Co	56	$9.5 \cdot 10^{-4}$	4
Cu	562	0.0088	397
Cr	56.6	0.0011	34.4
Mo	15.6	$1.6 \cdot 10^{-4}$	4.18
Ni	151	0.0026	78.4
Sn	184	0.0016	112
V	15.8	$3.1 \cdot 10^{-4}$	4.32
Zn	294000	4.5	56700
B	556	0.051	77.3
Hg	0.216	$1.1 \cdot 10^{-6}$	0.0214

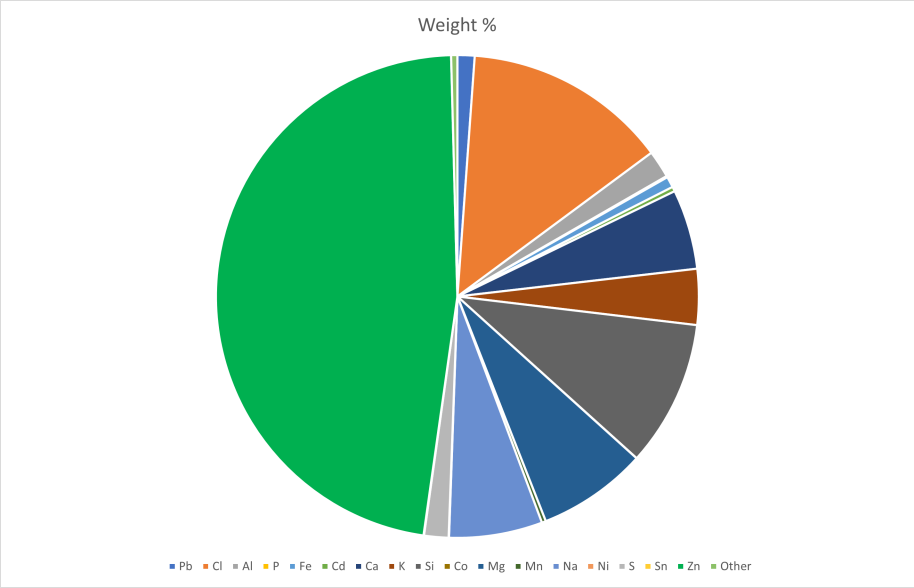

Figure 10: A overview of the average total composition of the samples in weight%

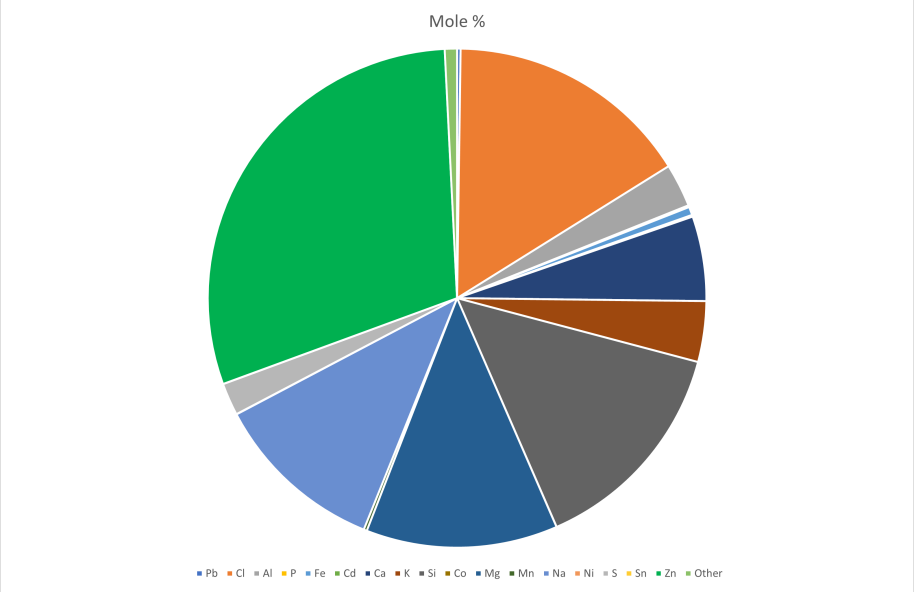

Figure 11: A overview of the average total composition of the samples in mole%

5.1 Analysis of raw samples

There were two main operating parameters that were changed in the Zn recovery plant during this thesis. The first parameter was liquid to solid ratio (L/S) which simply is the ratio between the amount of ash from the electrostatic precipitator and the amount of liquid used from the acidic scrubber, the second parameter is the pH in the reactor when the dissolving happens. Both these parameters are from the first reactor in the process numbered 3 in Figure 3. The Third parameter was the pH in the precipitation chamber where the $Zn(OH)_2$ is created which is the second reaction chamber numbered 4 in the same picture. The different samples and their corresponding parameters can be seen in Table 3 and the result of the powder X-ray diffraction can be seen in Figure 12.

Table 3: Overview of the different samples

Sample/name	date	pH dissolving	L/S	pH for precipitation
Sample 1	25/1	3.8	6	9
Sample 2	27/1	3.8	6	10
Sample 3	2/3	3.8	4.5	9
Sample 4	10/3	3.8	4	9
Sample 5	17/3	3.8	4	10

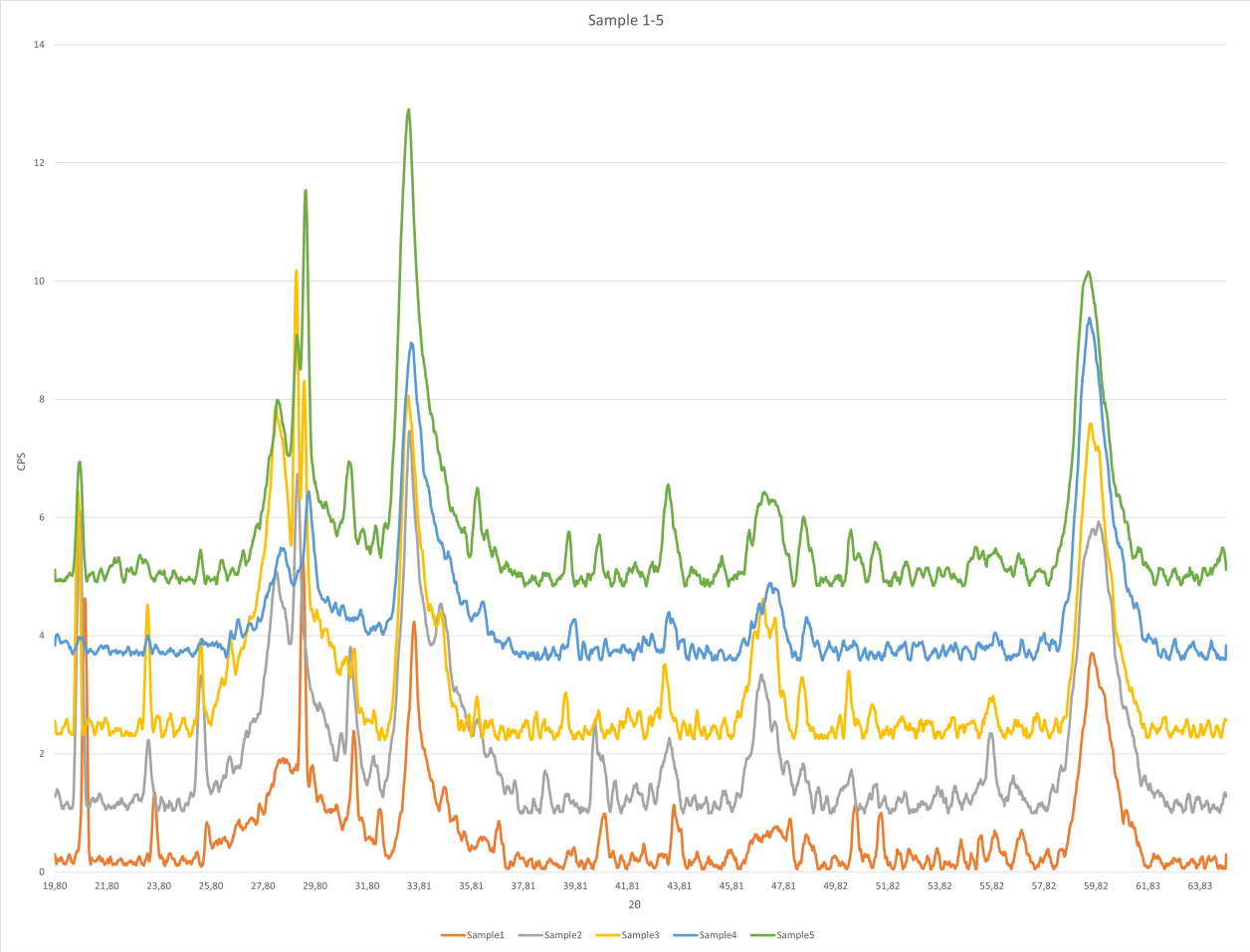

Figure 12: P-XRD on all the samples in their raw state

It is clear when looking at Figure 12 that all the samples have similar structures and that the different operating parameters have minimal impact, at least to the extent that they have been changed on these samples. Sample 4 is the one outlier since there is no big peak at about 21°. Because of that revelation it was decided to only focus the following experiment on two of the samples since they would all probably yield the same result. If the result from the two samples would differ the other samples would also be investigated. This was done to save both time and decrease the cost of the project.

In the figure a couple of "significant" peaks can be observed, the first being at about 20° then there are a couple of peaks around $28° - 33°$, and there is also a final very broad peak at the end. The

reason for it being broad can be because of the sample being partly amorphous [30]. By comparing the diffractogram in Figure 12 to the crystalline and amorphous diffractograms in Figure 4 it further strengthens the hypothesis that the sample contains both crystalline materials and amorphous materials. There are some high clear peaks at about 21° and 29.5° but there are also some characteristics of an amorphous solid.

Comparing the peaks from the diffractogram to the ones in the database became quite hard because of the amorphous structure. When also taking into account the total compound analysis of the sample in Table 2 and using the database the main candidate found was gypsum or $CaSO_4 \cdot 2H_2O$. A figure comparing the achieved diffractogram from sample 1 to gypsum can be seen in Figure 13. In the figure black graph is the achieved diffractogram from running the Bragg-Brentano analysis on sample 1 and the red lines are the peaks for gypsum from the database. Most of the major peaks from the analysis match well with gypsum. Gypsum also forms easily and is a common byproduct of many industrial processes, and all the elements for gypsum to form are present [31].

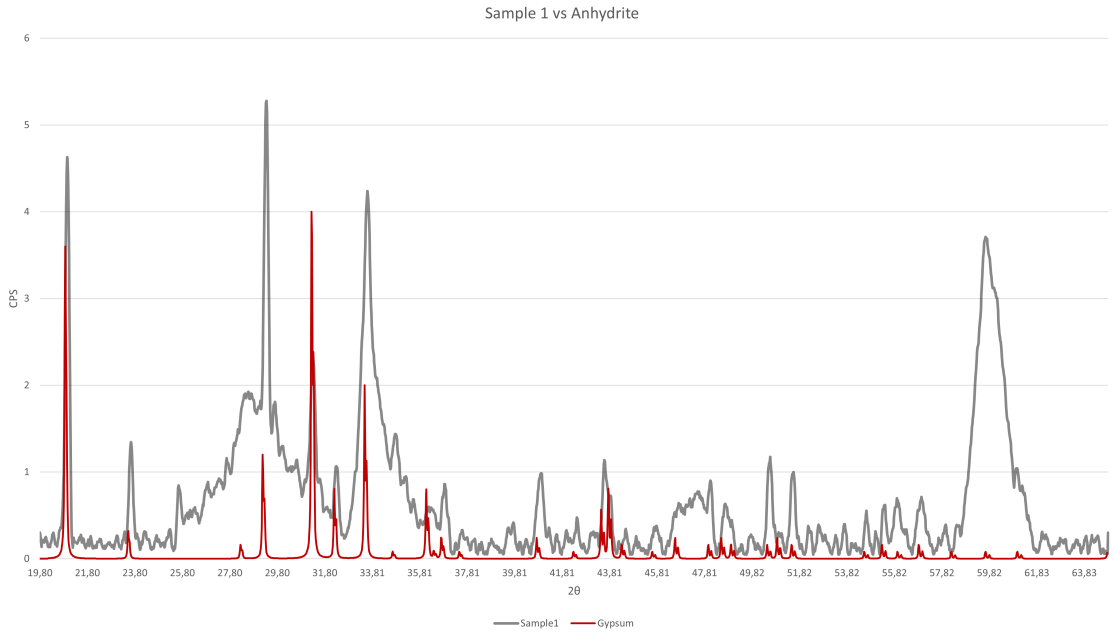

Figure 13: sample 1 matched with gypsum ($CaSO_4 \cdot 2H_2O$)

5.2 Drying of samples

There are several reasons for testing different drying temperatures. As mentioned above gypsum or dihydrite was the main candidate in the raw samples. It could be possible that there were other compounds present in the sample just that the moisture content made the structures amorphous and adding heat would both dry the sample and provide the energy needed for the atoms to move into clear crystal structures. The chemical formula for gypsum is $CaSO_4 \cdot 2H_2O$ which means it has water of crystallization present in the crystal structure. Studies show that for the dihydrite to be converted to anhydrite ($CaSO_4 \cdot 2H_2O \rightarrow CaSO_4 + 2H_2O$) a minimum temperature of about $80^{\circ}C$ was needed [31]. This meant that the hypothesis of the samples containing gypsum could be strengthened by simply drying the samples in below and above 80 degrees and noting if there is a change in how much water evaporates. The total composition analysis also shows that there is about 70% water present in the samples. From an economic standpoint it would be interesting to investigate if all water could be evaporated at room temperature since if this product were to be sold it would be unnecessary to ship the product containing 70% water and room temperature would be the most realistic temperature to dry the product in at the site.

5.2.1 Room temperature for 48 hours

The samples were left to dry at room temperature under the fume hood so there was a constant airflow. In Table 4 can the percentage of evaporated mass be seen.

Table 4: Mass evaporated for samples 1-5 after 48 hours in room temp

Sample/name	mass evaporated [%]
Sample 1	61
Sample 2	65
Sample 3	66
Sample 4	63
Sample 5	66

When comparing the amount of moisture that evaporated at room temperature compared to 105 as shown in the total composition analysis in Table 2 is about 3 to 5 percentage points lower. This is an indication that there might be gypsum which does not release its waters of crystallization in these low temperatures. The result of powder X-ray diffraction can be seen in Figure 14.

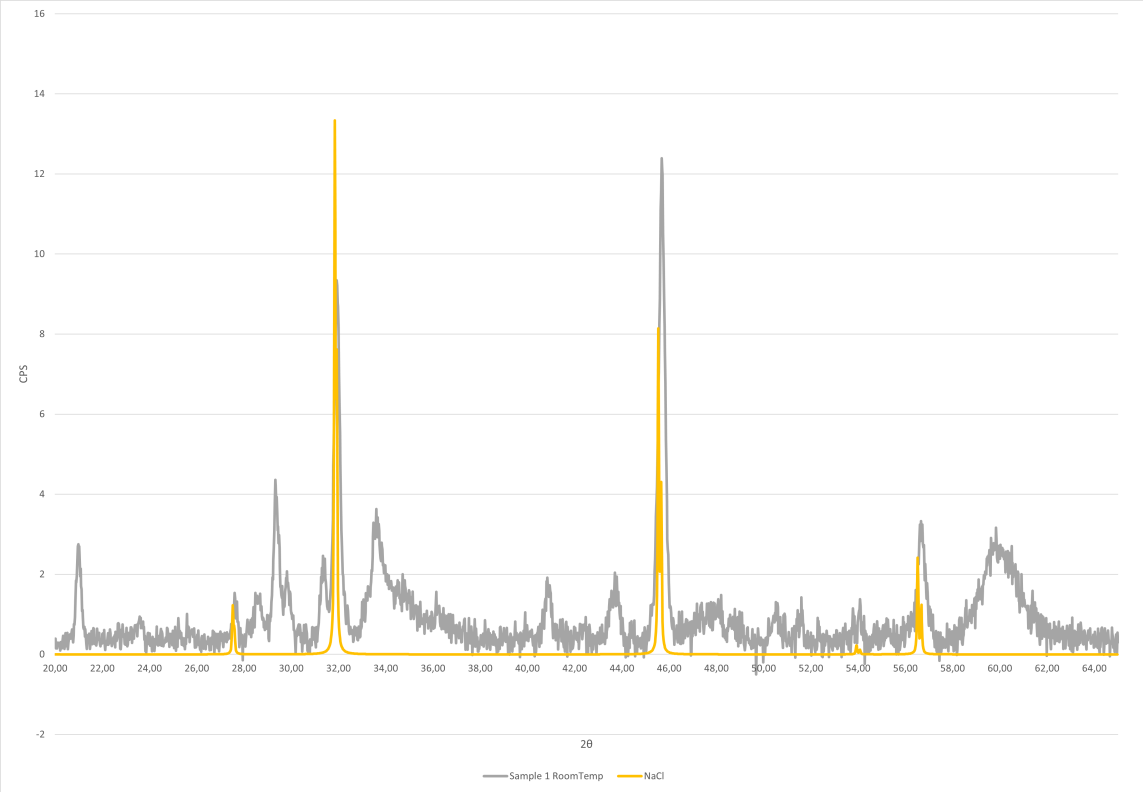

Figure 14: diffractogram over sample 1 dried in room temperature with the sodium chloride pattern

There is a clear and noticeable difference in the diffractogram from 12 to this. There are mainly three new very large clear peaks, one at 32°, one at 45.5° and one at 56.5°. The rest of the diffractogram is very similar to the raw sample. Using the databases and keeping in mind the total composition analysis these new peaks were concluded to be from halite or sodium chloride (NaCl). Sodium chloride is a very common salt and there is a lot of both chlorine and sodium in the samples. It is worth noting that there are more moles of chlorine in the samples than sodium which means that there might still be other compounds containing chlorine that has not yet been found. A possible reason for sodium chloride not being present in the earlier analysis is that there might have been enough water in the sample that the salt is in ion form. As the weight result above show the samples do contain above 60% water. The fact that so much water can evaporate at room temperature is very good since that can reduce the shipping cost significantly of the product.

5.2.2 $105°C$ for 24 hours

The sample was left to dry in an oven for 24 hours at $105°C$. $105°C$ was chosen since it should make sure that all water that is not chemically bound to a compound should evaporate. If the previous assessments regarding gypsum and the conversion temperature of gypsum to anhydrite are correct there should also be a change in the diffractogram from gypsum to anhydrite. The evaporated mass during the drying procedure can be seen in Table 5, the diffractograms from the analysis can be seen in Figure 15 and sample 1 compared to sodium chloride and anhydrite is shown in picture 16

Table 5: Mass evaporated for samples 1-5 after 24 hours $105°C$

Sample/name	mass evaporated [%]
Sample 1	71
Sample 2	74
Sample 3	72
Sample 4	74
Sample 5	73

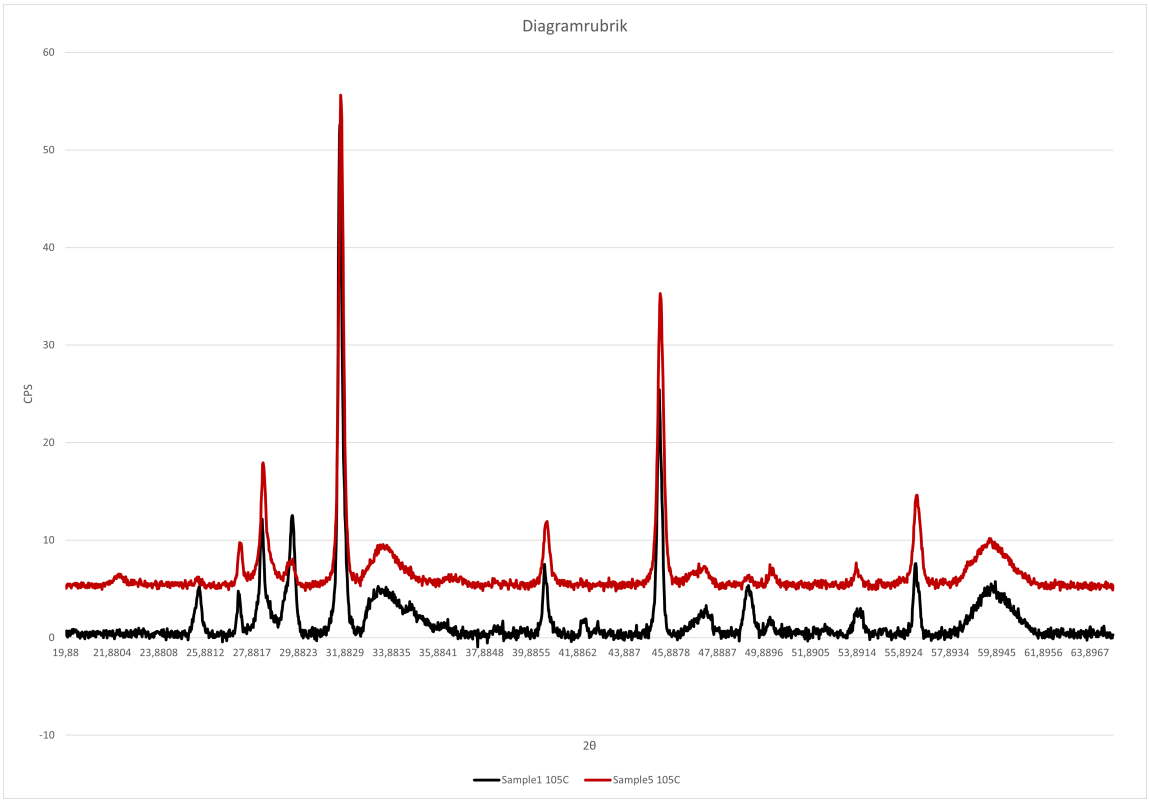

Figure 15: Sample 1 and sample 5 dried in $105°C$

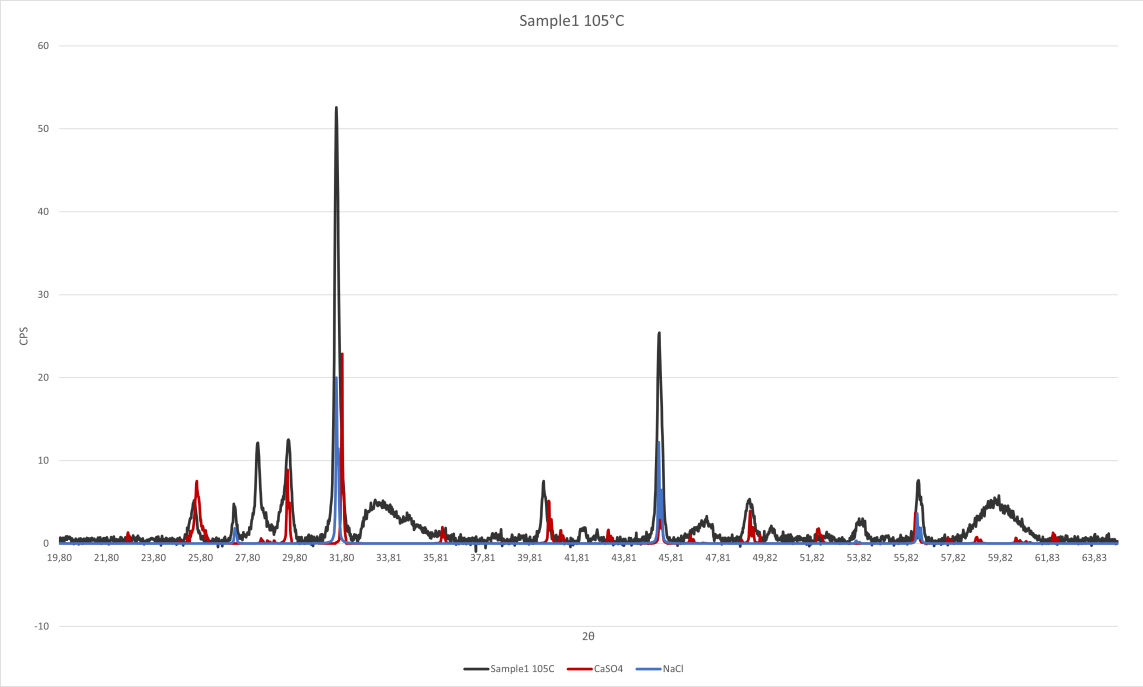

Figure 16: Patterns for Anhydrite and Sodium chloride matched with the sample dried in 105°C

A bit more water evaporated than in the experiment with the samples dried at room temperature, the newly achieved moisture levels correspond nicely to the achieved values in the total composition analysis which also was done 105°C. The two samples look very similar in shape and scale, but compared to the earlier samples these are much higher on the y-axis. This means that the detector reads more counts per second than it did in the previous tests. This can be a consequence of the sample becoming more and more crystalline as more moisture is evaporated. The pattern of sodium chloride still matches very well to the diffractogram but as hypothesized earlier the pattern of anhydrite now matches better than the gypsum one as can be seen in Figure 16. There is still no found compound that contains Zn which seems strange since a lot of Zn has been proven to be present in the samples. This might be because the databases used might be lacking data on the compound, the Zn might be present in so many different compounds that no clear compounds can be found. There might still exists amorphous compounds in the sample that do not show up on the X-ray diffraction.

5.2.3 $250°C$ for 24 hours

The fact that there still not had been any Zn compounds identified even if there such a large part of the sample consists of Zn as can be seen in Table2 heavily emphasized that the Zn compounds were amorphous. The diffractograms also had very large characteristics of amorphous material and since previous tests showed that increasing the temperature can bring forth amorphous materials the decision was made to dry the samples in $250°C$. This might not be such a valid procedure when it comes to the large scale process since this would then require a lot of heat/energy. The results of the weighing process can be seen in Table 6

Table 6: Mass evaporated for samples 1-5 after 24 hours $250°C$

Sample/name	mass evaporated [%]
Sample 1	73
Sample 2	76
Sample 3	73
Sample 4	75
Sample 5	75

The results indicate that a little bit more moisture has evaporated than when drying the samples in $105°C$. This is probably water that is somehow chemically bound to the compounds in the sample in some way such as water of crystallization. And when the temperature gets high enough there is enough energy for the bonds to break and the water to evaporate. The first thing that was noticed after the drying process was that the samples had gone through a significant color change during the drying process. The sample were much lighter brown than the previous gray/light gray color. The reason for the color change is not known but it could be because some of the new crystalline compounds that appear have that color or that there is some oxidization happening, this was not seen on the weight of the samples however. The achieved diffractogram can be seen in Figure 17

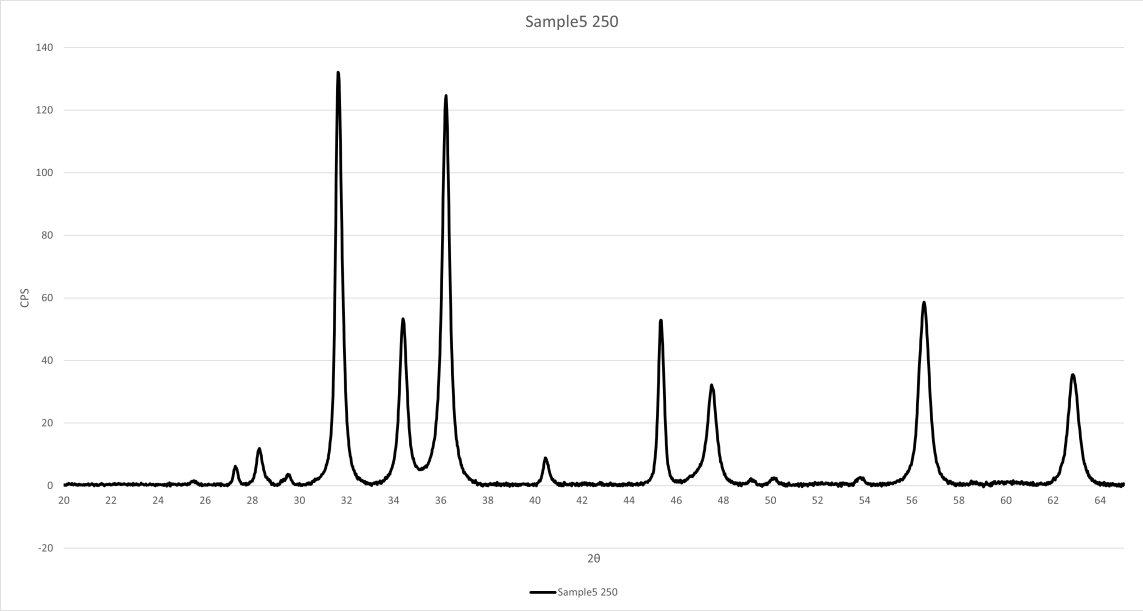

Figure 17: Sample 1 dried in 250°C

The diffractogram now looks much better with much less amorphous content and new very clear peaks. These peaks indicate on some sort of ZnO or $Zn(OH)_2$ compounds being present. Since they all have the same pattern it is almost impossible to decipher which ones are present in the sample from just the diffractograms. With the help of literature and the total composition analysis the possible compounds could be narrowed to just a few. The derived possible compounds were zinc iron oxide, zinc cobalt oxide, zinc manganese oxide, zinc nickel oxide, zinc oxide and zinc hydroxide. The different compounds can be seen in Figure 18

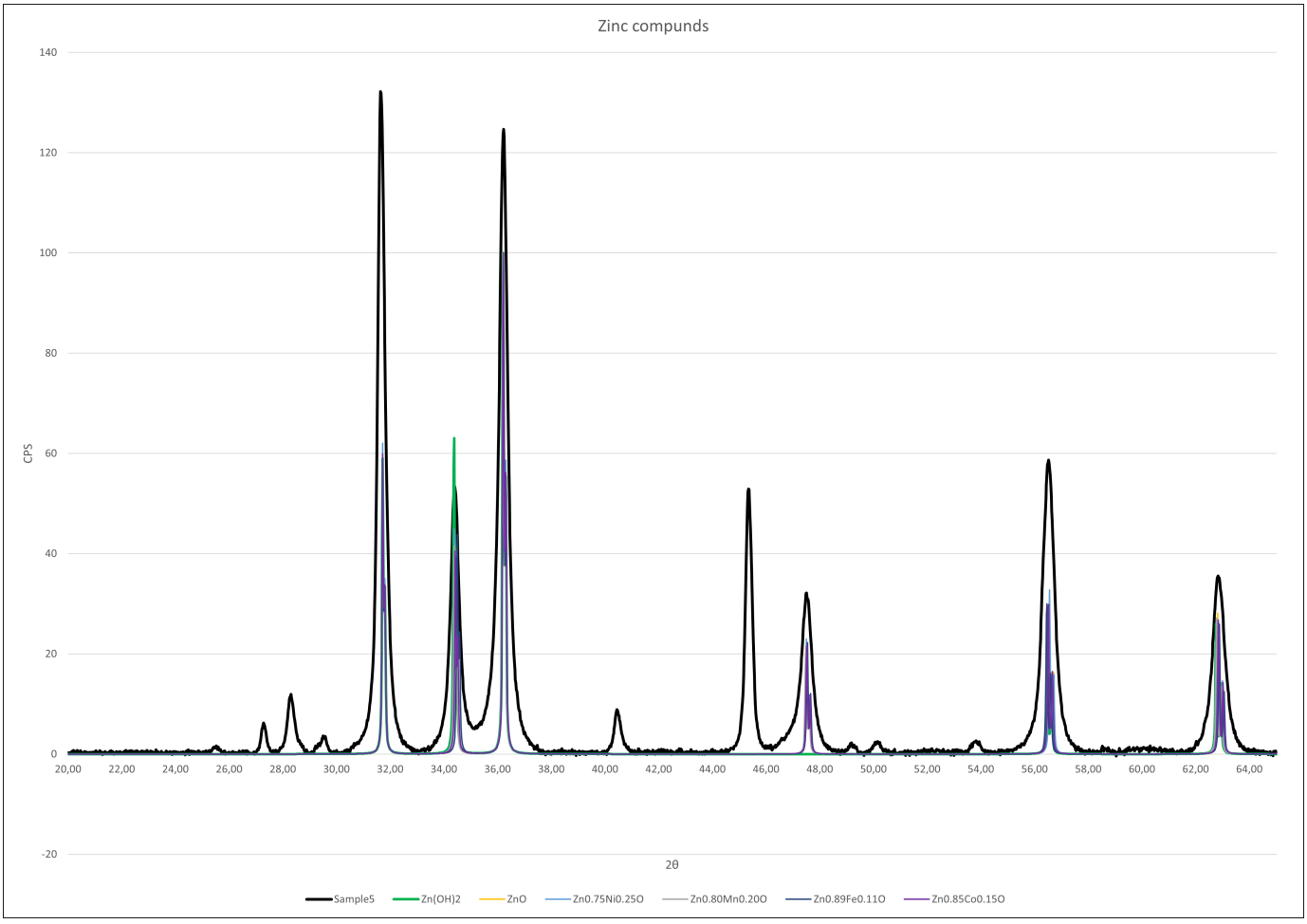

Figure 18: Possible zinc compounds in sample

All the different types of zinc oxides share the same peaks except for $Zn(OH)_2$ which does not have a peak at around 47.5°. With this we can deduct the possibility that all the zinc has formed $Zn(OH)_2$ since the peak should not be present in the diffractogram if that were the case. It is instead more likely that the Zn has formed some sort of oxide or several different types of oxides as presented in the picture. This does not rule out the possibility that some of the Zn have form zinc $Zn(OH)_2$ as initially interned. To be able to differentiate between the options other methods have to be used. Methods such as electron microscopy can possibly give a better answer. Combining the result achieved in the earlier tests it is possible to suggest a somewhat complete composition of the sample. A suggestion of the complete composition can be seen in Figure 19

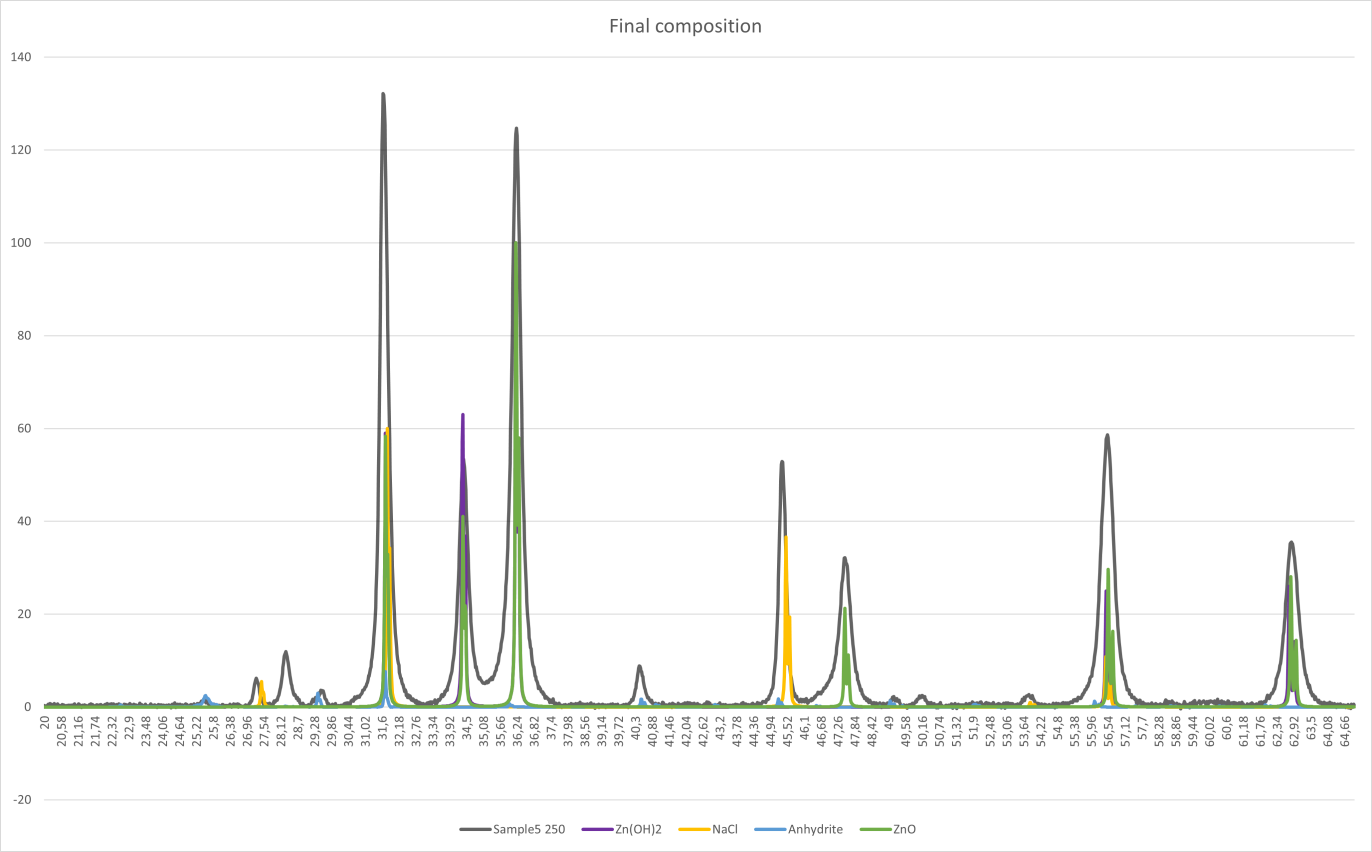

Figure 19: Suggested total composition

In the figure only ZnO is present of the earlier suggested compounds, this is only a representation to make the picture easier to read. Instead the oxide is probably a mixture of several oxides and possibly $Zn(OH)_2$. The total composition would be made of sodium chloride ($NaCl$), and anhydrite ($CaSO_4$) when dried in temperatures at around $100°C$ or above otherwise it would be gypsum ($CaSO_4 \cdot 2H_2O$). and Finlay the Zn would be in some forms of ZnO and $Zn(OH)_2$. This would explain almost every peak in the diffractogram which is the goal of the final composition. It is important to note that Zn compounds probably are present in the sample even in the lower temperatures even if they are not visible on the X-ray diffraction spectrum because they are in an amorphous state. It is not certain that the product would have to be in a crystalline form and in that case the product would not have to be dried at high temperatures. A big problem with the current product is that there is a lot chlorine in the sample which has a bad tendency to create corrosion in facilities that could retreat the Zn product to high purity. Therefore some experiments to reduce the chlorine content were performed.

5.3 Washing and filtration of the zinc product

The goal of the extra cleaning step was to investigate the possibility to remove chlorine from the sample by washing it with milli-q water or ultra pure water and then filtering it. The result from the filtration was made by first analyzing the liquid extracted from the filtration and later the samples were dried and also analyzed with X-ray diffraction. This would first show if any chlorine from the sample followed through the filtration and then it could possibly be analyzed if any amount of sodium chloride in the sample had decreased. The sample was then left to dry at room temperature because as earlier tests showed the clearest peak of NaCl could be seen in diffractograms from the samples dried at room temperature. A share of the extracted residue was then dried in 250 to also analyze the affect on higher temperatures. The resulting diffractogram for sample 1 and sample 5 can be seen in Figure 20

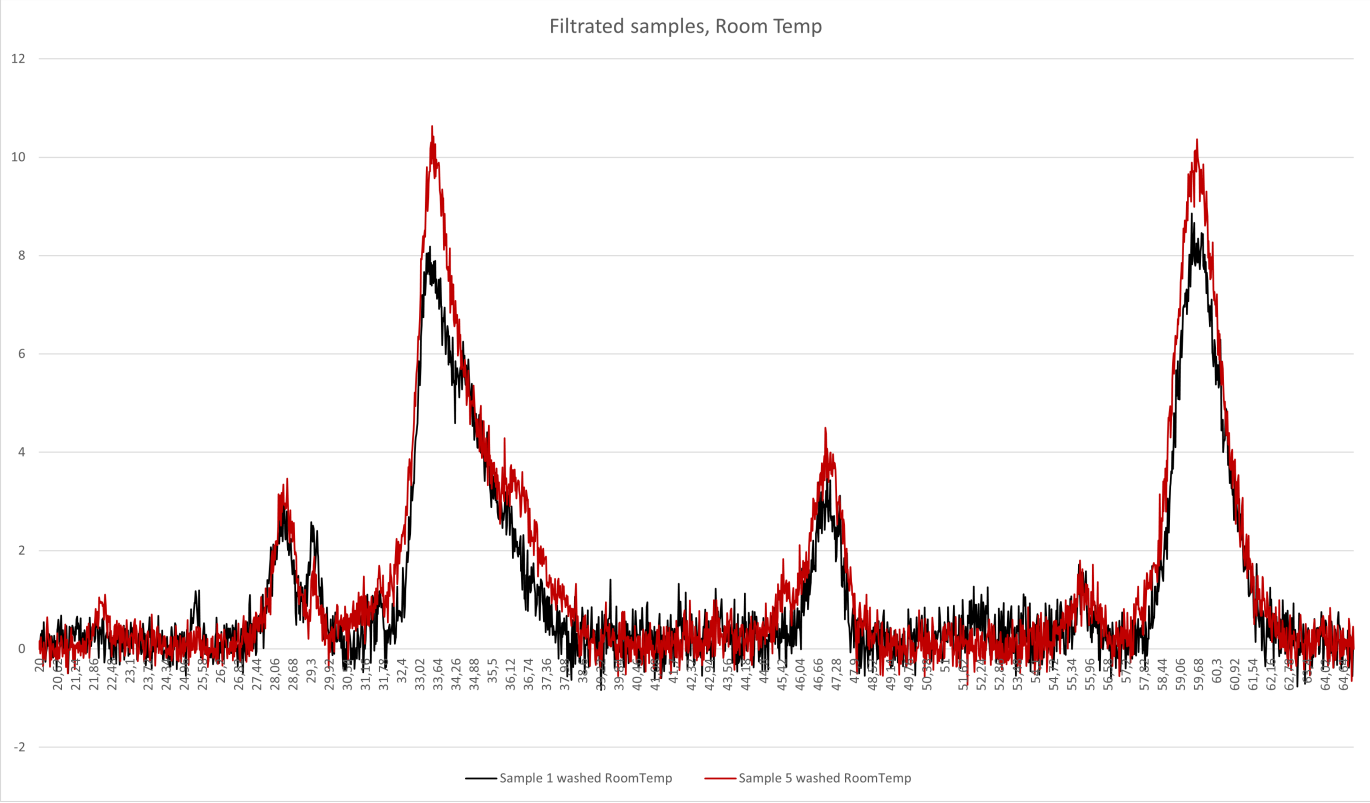

Figure 20: P-XRD diffractogram of washed samples dried in room temperature

This figure is best compared with Figure 14 from earlier since both of these experiments have

been dried at the same temperature. As predicted the peaks characteristic for sodium chloride disappeared almost completely. There might be some sodium chloride left in the sample because of the simple filtration method used. Because the sample was still wet from the filtration when moved to the drying stage there was probably some sodium chloride in the water that then ended up in the sample after it evaporated. Another thing noticed from the picture is that the peaks that are left are much larger than in picture 14. This could be a consequence of there being less sodium chloride in the sample and therefore the share of other crystalline materials in the samples increase compared to the earlier test and therefore the detector detects more photons per second in these cases. Similar patterns could be seen in the diffractograms for the washed samples dried in 250.

The results from the IC confirmed the hypothesis that a lot of the chlorine had been cleaned via washing and filtration. In Table 7 the concentration of chlorine extracted from the IC can be compared with the theoretical concentration that would be in the water. The calculations of the theoretical concentrations were based on the mass of the sample diluted, the amount of milli-Q water presented in Table 1 and also the extracted concentrations of chloride from the total composition analysis in Table 2

Table 7: The concentration extracted from the IC and theoretical concentration of chlorine in filtrate

	experimental concentration [mM]	theoretical concentration [mM]
Sample 1	162.0	203.2
Sample 5	134.3	268.3

In Table 7 the experimental values have been multiplied with their respective dilution factor to better match the theoretical concentration. The experimental value is about 80% of the theoretical value indicating that the majority of the chlorine is water soluble. There can be a number of reasons for this. One major reason could bet that all the chlorine does not form ions and there dissolve in the mixing step of the cleaning, this is supported by the fact there is more chlorine in the samples the sodium which means even if all the sodium atoms form NaCl there will still be chlorine left in the sample that can form other compounds that might not dissolve in the mixing step. The fact that the performed filtration was very simple and gravity-based meant that the residue did not completely dry during the experiment and there was still some water containing chlorine in the sample when the experiment ended. Since it is hard to measure how much water was left in the sample it is hard to draw any further conclusions regarding the difference in theoretical and experimental concentrations. It is however clear that a lot if not all of the chlorine can be removed from the sample via a simple cleaning step which is essential for the process to work.

6 Conclusion

In conclusion, there is a clear pattern that indicated that the raw samples do contain a lot of amorphous material that does become more crystalline as the samples are dried in higher temperatures. The most possible reason for this is probably that the atoms form an amorphous pattern in the precipitation tank and filter press, since the formation of the solid was so quick the ions did not have time to go from a liquid state to form a clear crystalline structure. When supplying heat the atoms gradually get enough energy to move around enough to form clear crystal structures and this is the phenomenon that can be observed in the result. There might be a correlation between water of crystallization evaporating and materials becoming more crystalline, however there was not any significant support for this theory in the literature. Instead water of crystallization should be part of the crystal structure and would still yield clear diffractograms. Other analysis methods then powder X-ray diffraction to be used to analyze what kinds of zinc compounds are present in the sample such as scanning electron microscopy. It is also not clear if the zinc compounds in the sample are the same overall temperatures, the zinc might change from one formation to another during the crystallization phase. This can also be investigated with the help of electron microscopy. There is clear evidence that a lot of the chlorine in the samples can be removed by adding an extra cleaning step to the process. The fact that the achieved product probably does not have as high concentrations of zinc hydroxide as expected earlier might not be a problem. The process of going from zinc hydroxide to zinc can also be done for zinc oxides. We can also see that the cleaning process is very robust and changing the operating parameters does not have a significant impact on the content of the sample. This is very good since a waste to energy plant has different compositions in the fuel it uses which can lead to different compositions in the fly ash as indicated by the total composition analysis.

7 Continued work

As mentioned earlier there are several ways to continue the work that has been done in this thesis, the first could be to use other analysis methods for analyzing the samples such as scanning electron microscopy. This could possibly more precisely deduct what compounds are present in the Zn product and it could also be used to investigate if the compounds are the same at all drying temperatures since it could also analyze the amorphous samples. Another way to continue is to focus removal of chlorine, since the experiments in this thesis showed that is possible to clean the sample via washing followed by simple filtration but were unclear regarding if all the chlorine was cleaned or not. A more detailed analysis regarding other possible forms of chlorine compounds than just $ZnCl_2$ and NaCl could be made. One could also investigate the existing cleaning process and implement a third step to more efficiently remove Cl or remodel the precipitation step to include the removal of chlorine.

8 Acknowledgments

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