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INVESTIGATION OF LONG TERM PILLAR STABILITY IN AN ABANDONED
GYPSUM MINE BASED ON DISSOLUTION MODELS

By

Mohammadhossein Sadeghiamirshahidi

A DISSERTATION

Submitted in partial fulfillment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

In Civil Engineering

MICHIGAN TECHNOLOGICAL UNIVERSITY

2019

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This dissertation has been approved in partial fulfillment of the requirements for the Degree of DOCTOR OF PHILOSOPHY in Civil Engineering.

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Author contribution statement

Chapter 2 of this dissertation has been published as an original article entitled “Analysis of drying and saturating natural gypsum samples for mechanical testing”, in Journal of Rock Mechanics and Geotechnical Engineering, 2018, Under a Creative Commons license (<https://creativecommons.org/licenses/by-nc-nd/4.0/>).

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Abstract

The abandoned Domtar Mine in Grand Rapids, MI underlies Interstate I-196. The mine operated for over 140 years, mining a 30-meter-deep, four-meter thick gypsum seam. In 2000, the mine was abandoned with the removal of the mine's dewatering pumps allowing groundwater to flood the mine. The initial flooding resulted in saturating the mine pillars along with some amount of pillar dissolution. Overtime, gypsum dissolution would cease as the water becomes saturated with the gypsum dissolution products. The mine, however, is located adjacent to the Grand River and has groundwater moving through the mine resulting in the potential for continuing dissolution. The stability of the mine relies on the support pillars, which are being reduced in size due to the dissolution. In this research, we analyzed the long term stability of the Mine's pillars. Samples from a quarry that mined the same gypsum seam were obtained. The moisture content of gypsum specimens was measured. The drying temperature according to ASTM standard, however, should not exceed 60 °C since heating can result in the transformation of gypsum to hemihydrate. The research investigated the temperature at which this transformation occurred using a helium pycnometer and determined that 80 °C can be used for gypsum's moisture content measurements. The saturation process of specimens for mechanical testing was also investigated, concluding a saturated gypsum-water solution is required to minimize dissolution when saturating process. The dissolution rate of gypsum in both stagnant and flowing water was experimentally investigated, confirming the dissolution of pillars is a first-order kinetics reaction. The normalized dissolution coefficient for stagnant water was measured at 1.6×10^{-3} (cm/s) following a power law for flowing water. A simple analytical model was developed to predict the change of specimen's diameter by time due to dissolution. A finite volume model was developed in FLAC3D to model the strength reduction of specimens due to both saturation and dissolution. The model was used to estimate the long-term stability of pillars in Domtar Mine. Finally, combining the analytical model and the numerical simulations, the time to failure of a pillar was estimated under different groundwater flow rates.

Chapter 1

1 Introduction

1.1 Stability of abandoned underground gypsum mine background

In many areas of the world the stability of abandoned underground mines is an important issue. Unexpected cave-ins can have significant economic impacts, and in some cases can cause fatalities. Furthermore, as the world's population grows, towns and cities have been developed over abandoned mines, increasing the concerns for the stability of these towns and cities. While in many cases, the location of the abandoned mine is known with an acceptable certainty, the mining patterns and the type and condition of support structures (pillars, wood or steel structures) used in the mine, especially in older mines, is not well known. In addition, virtually all abandoned mines have limited to no access after closure, and for mine located below the groundwater table the mines are flooded. This makes it a challenging task if not impossible to inspect the condition of the support structures after the mine is abandoned. Therefore, analyzing the long-term stability of these mines is difficult and unexpected collapses are not uncommon.

The stability of abandoned mines is further complicated in evaporite deposits such as salt and gypsum, as evaporite deposits are susceptible to dissolution (Ferris et al. (1989) and El-Shayeb et al. (2001)). When underground mines are located below the groundwater table, the water enters the mine and is pumped out to allow mining operate in dry conditions. After the mining ends, however, the dewatering is discontinued leading to the flooding of the mine workings. In abandoned anhydrite and gypsum mines, the water will cause pillar dissolution, reducing the radius (width) of the pillars. While the weight of overburden on an individual pillar is generally constant, the pillar's width is decreasing causing the stress on the pillar to increase. Gypsum dissolution will eventually ceases, though, when the water becomes saturated with respect to gypsum products created by the dissolution process. In the case of flowing water, however, dissolution will continue since the dissolution products will be carried away by water flow and advection causing the water adjacent a pillar to remain unsaturated thus allowing dissolution to continue. Consequently, dissolution continues to reduce the mine pillar's diameter until the stress on the pillar reaches the bearing capacity (strength) of the pillar (Castellanza et al. 2007). Collapses of abandoned mines operating in evaporite deposits (especially anhydrite and gypsum) are common, and have been reported by many researchers including Auvray et al. (2004), James and Lupton (1978), Barla and Jarre (1991), Auvray et al. (2008) and Johnson (2005). The time to failure in these cases depends on the stability of pillar before being degraded by gypsum dissolution, i.e., the shape and width to height ratio of pillars, depth of mine, weight of overburden, etc., the rate at which the gypsum is dissolved (weathered) and how the dissolution affects the strength of the pillar.

Gypsum dissolution is important process in rock weathering, subsidence due to gypsum karst formation, mineral durability, sediment-water interaction, soils amendment and many other cases (Colombani 2008). Researchers in different disciplines tend to use

various tests to measure the dissolution rates. In fact, many attempts have been made to obtain the dissolution rate laws for minerals especially gypsum (e.g.: Barton and Wilde (1971), Bolan et al. (1991), Dreybrodt and Gabrovsek (2000), Colombani and Bert (2007), Fan and Teng (2007) and Colombani (2008)). These studies often use powdered gypsum or artificial gypsum particles in batch or column experiments and sometimes the pinhole dissolution tests (circulation tests) are performed. Because of complicated nature of gypsum dissolution and water flow systems, it is hard (if not impossible) to use the results of these experiments for other applications such as dissolution of gypsum in mine's pillars.

Recently, however, (Castellanza et al. 2007) have investigated the effect of weathering on short term strength of mine pillars and used their modified short term strength analyses to investigate the stability of the mine pillars. They combined the weathering progression within gypsum pillars and their strength decay in order to predict the expected time to failure of pillars. In their model they introduced two constants α , the rate of progression of the weathering front, and β and rate of strength reduction, which are obtained by best fitting the experimental data to an analytical expression of the dimensionless load as a function of dimensionless time. They use the calculated α and β values to predict the expected time to failure of pillars in an abandoned gypsum mine. Obtaining these parameters is somewhat arbitrary and estimating them requires conducting many experiments, which is not time nor cost effective.

To date, the research conducted suggests that gypsum dissolution rate depends mostly on temperature and chemical composition of the flowing water, the thickness of the diffusive boundary layer around the gypsum objects (e.g. left in place gypsum pillars in abandoned mine) and the concentration gradient across the diffusive boundary layer. It has also been indicated that, the higher the velocity of steady flow and/or higher fluctuation intensity of unsteady flow, the thinner the diffusive boundary layer which leads to increased gypsum dissolution rates (James and Lupton 1978 as explained in Porter et al., 2008).

1.2 Case Study

This research investigated the long-term stability of the Domtar Mine located in Grand Rapids, MI. The Domtar Mine is an abandoned underground gypsum mine. A portion of Interstate I-196 is located over a portion of the mine, which at a depth of about 30 m (90 feet). The following section provides the history of the Domtar Mine.

Gypsum mining started in Grand Rapids, MI in the 1840's along the Grand River and continued until 2000 when the Domtar Mine ended operations due to declining gypsum prices. The Domtar Mine is located on the west side of Grand Rapids in the south half of section 27 (T7N, R12W) and the north half of Section 34 (T7N, R12W). Surface quarrying of gypsum started at this mine in 1848 and after a number of acquisitions the mine became the Grand Rapids Gypsum Company in 1860. Underground mining started in the 1860's and continued to 1975 when the mine closed due to poor economic conditions. In 1983, the mine was purchased by Domtar, Inc. and reopened with gypsum

production starting in 1984. In 1996, Georgia-Pacific purchased the Domtar Mine properties and continued to operate the mine until 2000 when the mine was once again closed. In 2000, the mine and processing facilities were demolished and the site reclaimed. Equipment in the underground mine was removed including dewatering pumps and the two remaining mine entrances backfilled with overburden materials. Observations of water in the main mine entry in 2003 indicated that the area's ground water level has been mostly reestablished and that the mine workings were completely flooded. In the early 1960's Michigan Department of Transportation (MDOT) purchased surface right-of way from the Grand Rapids Gypsum Company for the construction of Interstate 1-196 (CS 41029). 1-196, which was completed in 1963, crosses the northeast portion of the mine for approximately 2,100 feet from station +442 to +463. Existing mine maps show that the mining below the interstate occurred in the #2 gypsum seam at a depth of approximately 30 meters (90 ft) below the interstate and that no #4 or #5 gypsum seams were mined. The #2 gypsum seam thickness is approximately 4 m (12 ft). A map of the underground mine workings below 1-196 are shown in Figure 1-1.

A significant concern for Interstate 1-196 is the long-term stability from subsidence and sinkholes. In addition, the roof and floor rock of the mine contains shale, which loses strength when saturated. Extensive sinkholes as well as subsidence has occurred over the west and southern portions of the Domtar Mine (west of the interstate) due to roof and pillar collapse as well as some larger scale roof collapse that has occurred in the Georgia-Pacific Butterworth Mine adjacent and to the west of the Domtar Mine.

Figure 1-1. map of the underground mine workings below I-196 with location of sinkholes (modified from (Viton 2004))

1.3 Organization of the dissertation

This research analyzed the long-term stability of the mine Domtar Mine based on laboratory experiments and numerical modeling. Chapter 2 of this dissertation presents the research to determine the temperature at which gypsum transformation occurs under moisture content measurement conditions. An important parameter in testing the mechanical properties of gypsum is its moisture content. To measure the moisture content of the test specimens they had to be oven-dried. However, ASTM recommends that a temperature limit of 60 °C be used in the drying process. Temperatures above 60 °C causes the gypsum to transform to a hemihydrate and then to an anhydrite. Since the transformation process is accompanied by a particle density increase, a helium pycnometer was used to better understand the transition temperature to confirm that the prescribed drying temperature did not transform the gypsum used in this research. Following drying the test specimens were then saturated to replicate field conditions, i.e., in a flooded mine. Since saturation causes dissolution, a procedure had to be developed to saturate the test specimens without causing dissolution. To investigate this problem, the

following three methods were studied to saturate gypsum cores taking into account the solubility of gypsum: (1) water immersion, (2) vacuum saturation, and (3) improved vacuum saturation. The results of this investigation are also included in chapter 2.

Chapter 3 of this dissertation details the studies to determine the strength of the gypsum under dry and saturated conditions. Mechanical testing involved uniaxial compression strength (UCS), elasticity moduli and splitting tensile strength (BTS) of gypsum. New correlations were derived describing the effect of sample size on both UCS and BTS under dry and saturated conditions. Effects of blasting on these parameters were observed and the importance of choosing the proper samples discussed. Point load index tests, which are usually used as a simple substitute for indirect estimation of UCS and BTS, were conducted and correlations derived for both compression and tensile strengths under dry and saturated conditions.

Chapter 4 discusses the laboratory experiments conducted to investigate the dissolution rate of natural gypsum in both stagnant and flowing water. In this chapter, existing published dissolution rates were also reviewed and compared to our experimental results.

Chapter 5 utilizes the results of the research to investigate the rates of pillar's width and strength reduction due to gypsum dissolution. A simple analytical model was developed to predict the change of specimen's diameter by time due to dissolution. The analytical model was then evaluated using the results of the dissolution tests. A finite volume model was developed in FLAC3D to model the strength reduction of specimens due to both saturation and dissolution. The numerical model was used to estimate the long-term stability of pillars in the Domtar Mine. Finally, using the dissolution models along with the results of the numerical simulations, the time to possible failure of a single pillar in the mine was estimated under different flow rates. A detail explanation of the tests conducted in this research, the models that were developed and the results of the simulations and predictions are presented.

Finally, Chapter 6 of this dissertation summarizes the conclusions drawn from this research.

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Chapter 2

2 Analysis of drying and saturating natural gypsum samples for mechanical testing¹

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2.1 Abstract:

The stability of underground abandoned gypsum mines is dependent on the gypsum pillar's strength, and most abandoned mines are in a fully saturated condition. Moisture affects the strength of gypsum and is therefore commonly measured when testing rock strength. For most rocks, this is a simple task of weighing the rock's mass before and after oven-heating at a specified temperature and duration. For natural gypsum, however, this is not a straightforward process. Heating natural gypsum can result in dehydration and transformation of gypsum to hemihydrate and anhydrite, thus changing the physical characteristics of the gypsum such as its particle density which in turn affects the moisture content and strength measurements. To prevent transformation when determining the moisture content of gypsum, the American Society for Testing Materials (ASTM) recommends lowering the drying temperature from 110 °C to 60 °C. To investigate the temperature at which gypsum transforms to hemihydrate, we used a helium pycnometer to measure the particle densities of gypsum, hemihydrate and anhydrite. In this research, we suggest that a higher drying temperature of 80 °C can be used for drying gypsum without transforming gypsum to hemihydrate. Further, preparing saturated samples for mechanical testing, which is required in stability analyses of abandoned mines, is challenging due to the dissolution of gypsum when placed in water. To address this problem, we investigated the following methods to saturate gypsum cores taking into account the solubility of gypsum: (1) water immersion, (2) vacuum saturation, and (3) improved vacuum saturation. The research indicates that all the three methods are acceptable but they should be conducted using a saturated gypsum-water solution to minimize dissolution. Further, the research found that the improved vacuum saturation method saturated the test samples within 24 h, while duration of 30 h was required for the other two methods.

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2.2 Introduction

Gypsum mining began in Grand Rapids, Michigan in 1853. The largest underground mine in Grand Rapids was the Domtar Mine, which operated between 1857 and 2000 under the ownership of a series of companies, including the US Gypsum Company. The mine is located on the west side of Grand Rapids adjacent to the Grand River. In 1963, Interstate I-196 was constructed over the east side of the mine. The mine was abandoned and allowed to fill with water in 2000. Subsidence and sinkholes have occurred over some of the older pre-1900 mining areas, but the section of I-196 has had no observable subsidence. Groundwater, however, is moving through the mine from the mine's north side to its south side adjacent to the Grand River. While stagnant underground water can prevent long-term pillar dissolution, flowing water can result in the dissolution of the mine's support pillars compromising long-term stability. This research is part of a study to determine the long-term stability of the mine structure under I-196. A vital element of this investigation is determining the strength of the gypsum pillars as dissolution takes place and the time to failure.

For many sedimentary rocks, water can adversely affect the strength and deformability of rock as discussed by numerous researchers (Rolnick, 1954; Ballivy et al., 1976; Masuda, 2001; Auvray et al., 2008; Yilmaz, 2010; Bond et al., 2013; Mišćević and Vlastelica, 2014; Salih and Mohammed, 2017; Zhang, 2017). For moisture sensitive rocks, knowing the moisture content of the rocks is important. The most common methods for measuring rock's moisture content are convection oven drying (ASTM D2216-10, 2010) and microwave oven heating (ASTM D4643-17, 2017). For natural gypsum, however, heating the sample dehydrates gypsum and changes its chemical structure. Gypsum is a calcium sulfate dihydrate having two molecules of water in its structure ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$). Heating the gypsum causes it to lose one and half molecule of water, converting it to hemihydrate ($\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$). If heating continues at higher temperatures, the remaining half molecule of water will be removed, converting the hemihydrate to anhydrite (CaSO_4) (Rolnick, 1954).

The gypsum-anhydrite transformation in *aqueous solutions*, on the other hand, can occur through two different mechanisms depending on temperature (Azimi and Papangelakis, 2011). In aqueous solution at lower temperatures, the intermediate hemihydrate formation does not occur and gypsum transforms directly to anhydrite, while at higher temperatures, the gypsum transforms to hemihydrate first and then the hemihydrate transforms to anhydrite (Azimi and Papangelakis, 2011). These transformations not only results in a mechanical change but also causes considerable errors in the moisture content calculation. The American Society for Testing Materials (ASTM), therefore, suggests a maximum drying temperature of 60 °C instead of 110 °C commonly used for drying soil and rock. Further, ASTM does not recommend using a microwave oven for drying

gypsum. Drying gypsum samples at 60 °C or in a desiccator (as suggested by ASTM for gypsum samples) not only takes longer time, but also does not guarantee that the gypsum's moisture content will be reduced to a zero level.

The temperature at which gypsum transforms to hemihydrate and anhydrite, however, depends on a number of parameters such as the gypsum's pore water pressure, vapor pressure (for unsaturated conditions), chemicals in the pore water as well as whether the gypsum is natural or synthetic (Ramsdell and Partridge, 1929; Hudson-Lamb et al., 1996; Gysel, 2002; Mandal and Mandal, 2002). Davis (1907) tested dissolved gypsum in an *aqueous solution* to estimate the gypsum-anhydrite transition temperature. Their results indicated that this transformation occurs at 63.5 °C-66 °C for gypsum-hemihydrate and at 93 °C-107 °C for hemihydrate-anhydrite. Ramsdell and Partridge (1929) suggested a lower transition temperature at 38 °C for gypsum-hemihydrate and 98 °C for gypsum-anhydrite. However, Rolnick (1954) found that the gypsum-anhydrite transition temperature in an *aqueous solution* reported by the previous authors does not occur at a fixed temperature but is dependent on the geologic environments and can change with lithostatic and hydrostatic pressures. Gysel (2002) confirmed these findings that the transformation from anhydrite to gypsum occurs at temperatures of 43 °C, 46 °C and 53 °C at pressures of 250 atm, 500 atm and 1000 atm, respectively (1 atm=101,325 Pa). Gysel (2002) found that above 1000 atm, the transition temperatures is over 58 °C.

The research mentioned above was conducted on dissolved gypsum in an *aqueous solution* to study the environments under which gypsum is likely to precipitate such as in oceans. The research was also conducted on natural gypsum rock to determine its transition temperature. Rolnick (1954) found that heating natural gypsum to 130 °C converts gypsum to hemihydrate (Plaster of Paris) and at 370 °C to anhydrite. Mandal and Mandal (2002) reported similar results but at somewhat lower transition temperatures. They reported that heating gypsum at 90 °C for 10 h transforms it to hemihydrate while heating it at 350 °C for 10 h transform it entirely to anhydrite. Hudson-Lamb et al. (1996) tested both natural and synthetic gypsum and found that in both cases, gradually heating, at first, increases the amount of hemihydrate, and later with higher temperatures, increases the amount of anhydrite for both natural and synthetic gypsum. For example, their experiments showed small amounts of hemihydrate forming in both samples at 60 °C, while at 170 °C, the amount of hemihydrate increased with some anhydrite forming. At 450 °C, all the synthetic gypsum had transformed to anhydrite but in natural gypsum samples, hemihydrate was still present although most of the samples had been transformed to anhydrite (Hudson-Lamb et al., 1996).

A further issue in studying gypsum pillar strength is the gypsum's dissolution in freshwater when saturating the gypsum back to a fully saturated condition. Initially, gypsum mined at the Domtar Mine was below the groundwater table and therefore saturated. During mining, however, the mine was dewatered allowing the gypsum pillars to become unsaturated. At the end of mining, dewatering was stopped, allowing the groundwater table to rebound to its previous level. This is called groundwater rebound

process (Doulati Ardejani et al., 2013), which results in a gradual increase of water content and eventually saturation of the rock. Hence, while measuring the rock design parameters in a laboratory (e.g. unconfined compression strength (UCS)), different samples with different moisture contents (including dry and saturated samples) are usually prepared and tested. At least three different methods for saturating rock samples have been used by researchers, including simple saturation (water immersion), vacuum saturation, and improved vacuum saturation (ISRM, 1972; Ballivy et al., 1976; Melnyk and Skeet, 1986; Dandekar, 2013; Zhou et al., 2016). For simple saturation, rock samples are submerged in water for a specified period until they are saturated. The samples are periodically weighed until their weight gain due to saturation becomes constant. In vacuum saturation method, samples are submerged in water, and then both samples and water are degassed under vacuum for a given amount of time. In improved vacuum saturation, the sample is first degassed under vacuum and then submerged in degassed water. The samples stay under a vacuum till fully saturated (Melnyk and Skeet, 1986). In all the three methods, the time required for sample saturation is different and depends on the rock type, especially on the rock's porosity and pore size. For example, Nagaraju and Roy (2014) tested sandstone rock samples and reported that vacuum saturation of samples took less than 24 h, while Zhou et al., (2016) reported 48 h as the time required for saturation of sandstone from a different source. Therefore, the process and the time required for saturation of different rocks need to be studied for each source. Saturating gypsum, however, must also consider gypsum dissolution during the saturation process. In this research, all the three methods of preparing saturated rock core samples were investigated to minimize gypsum dissolution during saturation.

The purpose of this paper is twofold: First, to present our research using a helium pycnometer to investigate drying temperatures for gypsum without transforming it to a hemihydrate or anhydrite; second, to evaluate the three methods for saturating gypsum while minimizing the amount of gypsum dissolution.

2.3 Material and sample preparation

As mentioned above, the Domtar Mine in Grand Rapids is abandoned and flooded, thus gypsum samples were not available. There are, however, two operating surface gypsum mines located on the east side of Lower Michigan in the Michigan Basin. The gypsum deposits are part of the Michigan Formation, a Mississippian Age formation (Grimsley, 1904). Gypsum samples for this study were obtained from the Tawas Quarry near Tawas City, Iosco County, Michigan (Figure 2-1). More than 50 gypsum blocks, some of them shown in Figure 2-1c, d and e, were collected and delivered to the rock mechanics laboratory at Michigan Technological University where they were cored (using coring machine shown in Figure 2-2a), and prepared for this study. A total of 41 samples were prepared for this research, which included 39 core samples and two samples crushed into a powder. The gypsum blocks were cored at three different diameters of 28 mm, 53 mm and 76 mm. Figure 2-3 shows the prepared samples while the samples dimensions are summarized in Table 2-1. The first number in the sample ID indicates the block from

which the sample was prepared and a second number was used in the cases where more than one sample was prepared and used in this study from the same block.

The experimental program was divided into two parts. The first part was designed to investigate the transformation of gypsum to hemihydrate and anhydrite under “water content measurement conditions” using a helium pycnometer (with precision of 0.01%) to assess changes in density resulting from the gypsum transformation. The second part was designed to compare different methods of saturating the natural gypsum samples taking into account the solubility of the gypsum in water while determining the time required for saturation. These two sets of experiments are explained in more detail in the following sub-sections.

Figure 2-1 Location of the "Tawas quarry" and samples used in this research: (a) The red box shows the location of the Tawas City in Michigan (d-maps, 2018); (b) An aerial imagery of the quarry taken from Google maps; (c) Rock block #5 and (d) Rock block #8 from which samples used for density test were cored; and (e) A rock block before coring

Figure 2-2. Some of the devices used in the research: (a) Coring machine; (b) Helium pycnometer; and (c) Furnace. D and H denote the diameter and height of the sample, respectively.

2.3.1 Gypsum-hemihydrate-anhydrite transition under “water content measurement condition”

Measuring water content of rock samples usually involves drying the samples using a convection oven or microwave oven. As discussed above, heating gypsum can transform gypsum to hemihydrate and anhydrite (dehydration). ASTM D2216-10 (2010) requires the temperature of $(110 \pm 5)^\circ\text{C}$ for drying regular rock samples in the oven. The standard also addresses the possibility of gypsum dehydration and therefore states that gypsum samples should be dried at 60°C or in a desiccator at room temperature. Drying samples at 60°C or in a desiccator takes longer time, but it also does not guarantee that the gypsum's moisture content will be reduced to zero level.

To investigate drying gypsum at higher temperatures, we used a helium pycnometer to detect gypsum transformation by measuring changes in material density. According to Viana et al., (2002), *“True density is an intrinsic characteristic of a material, depending on its chemical nature and crystalline structure. It corresponds to the exact volume occupied by a material, without porosity. It is thus a fundamental parameter contributing to the characterization of a product.”* While X-ray diffraction is used to calculate the *true density* of a material, it is not routinely used in laboratory practice due to cost and equipment availability. Viana et al. (2002) added that helium pycnometry currently provides the closest approximation to true density, since helium can penetrate into the internal structure of a material allowing the closest estimate of a material's real volume. They further note that helium pycnometry is used to detect polymorphs and pseudo-polymorphs, and to follow the synthesis of a chemical substance or sequence of a chemical process. To determine the accuracy and reproducibility of helium pycnometry, they used an AccuPyc 1330 and found that its accuracy, under optimal conditions, can be as high as 0.02% for tests conducted on the same day and 0.1% for non-optimal conditions.

Figure 2-3. Samples used for moisture content and density tests (using helium pycnometer): (a) Sample #5 dry; (b) Sample #8 dry; (c) Sample #5 powdered; (d) Sample #8 powdered moist; (e) Sample #5-1 in a water bath; (f) Sample #5-1 dry; (g) Sample #5-1 saturated; (h) Sample #9-11; and (i) Sample #9-12.

In this research, a “Micromeritics AccuPyc II1340” helium pycnometer (shown in Figure 2-2b) was used to measure the particle density for (1) air-dried, (2) oven-dried and (3) microwave-dried samples. A mass balance with an accuracy of 0.01 g was used to measure the mass of the samples. The particle densities of gypsum, hemihydrate and anhydrite, given a certain level of impurities, are around 2.3 g/cm^3 , 2.6 g/cm^3 and 2.9 g/cm^3 , respectively (Robie and Bethke, 1962).

Seven samples at different sizes (i.e. samples #8 dry, #5 dry, #5-1 dry, #5-1 saturated, #5-1 in a water bath, #8 powdered moist and #5 powdered as shown in Figure 2-3) were used, which also allowed us to investigate the possible effect of sample size on the transition temperature. As discussed before, the presence of water could also affect the transition temperature and hence, we used dry and saturated/moist samples as well as samples in a water bath. Although heating the dry samples is not something that is done for moisture content test (because samples are already dry), it was tested to provide a better understanding of the gypsum-hemihydrate-anhydrite transition process. Also, heating the samples was continued even after the transition of gypsum to hemihydrate

(indicating higher temperatures cannot be used for moisture content), to obtain the full range of transition temperatures. To determine the drying time for saturated gypsum samples at room temperature, two saturated samples (#9-11 and #9-12) were air dried for 3 d to compare the results with the oven drying method. The density of samples was measured before saturating them as well as after the test (after air-drying the samples).

Figure 2-4. Samples used for saturation tests (numbers on the pictures shows the sample ID).

Finally, the two samples that were used for air drying test (#9-11 and #9-12) were employed again (#9-11 remained air-dried but #9-12 was saturated again before using for this test) for microwave test to see the effects of microwave on the transition temperature. Again, testing the dry sample was only conducted to derive a better understanding of the dehydration process. In this test, samples were heated in a 1000-W commercial microwave oven at full power for 3 min while the weight change was measured as required by ASTM D4643-17 (2017). Samples were then heated in the microwave oven for another minute and weighed again until a weight change occurred. Again, the density

of samples was measured before and after the microwave drying to examine whether the transition of gypsum to hemihydrate has occurred. The samples were microwave oven-dried for a total of 5 min.

Table 2-1. Summary of sample dimensions.

Sample ID	Diameter (mm)	Height (mm)
#8 dry	28.2	27.4
#5 dry	28.4	28.3
#5-1 dry	28.2	58.3
#5-1 saturated	28.2	58.5
#5-1 in a water bath	28.2	58.1
#8 powdered moist	Powder	Powder
#5 powdered dry		
#9-1	28.3	57.8
#9-2	28.3	32.2
#9-3	28.3	58.5
#9-4	28.3	31.4
#9-5	28.2	57.3
#9-6	28.2	26.2
#9-7	28.2	57.8
#9-8	28.2	22.3
#9-9	28.2	58.1
#9-10	28.2	57.6
#9-11	28.2	58
#9-12	28.2	58
#-16-1	28.1	59
#-16-2	28.2	59.2
#15-1	28.2	59.2
#15-2	28.2	59.2
#15-3	28.2	59.2
#17-1	28.2	59.2
#17-2	28.2	59.2
GYP-17-3	28.2	59.2
GYP-51-1	28.2	59.2
#51-2	28.2	59.2
#51-3	28.2	59.2
#1	53.34	112
#2-1	53.34	112
#2-2	53.34	112
#4	53.34	112
#3	53.34	112
#10	76.2	160
#20	76.2	160
#12-1	76.2	160
#12-2	76.2	160
#19-1	76.2	160
#19-2	76.2	160

2.3.2 Saturation methods

The standard methods for saturating rock are water immersion, vacuum saturation, and improved vacuum saturation in which the rock is put under a vacuum for a period of time and then placed in water also under a vacuum. Although these three methods can be used different rock types, the time required to saturate the samples can be different for each method. Therefore, the time efficiency of these methods needs to be tested before using them on a large number of samples. On the other hand, as gypsum dissolves in water relatively fast, saturating gypsum samples in water might cause some problems. To investigate this phenomenon, and how much it affects the saturation, all the three methods were tested using freshwater and water fully saturated using a gypsum powder (gypsum-saturated water). We also used different sample sizes to see the effects of size on time required to saturate the samples. Initially, eight gypsum core samples from the same gypsum block but with different sizes (samples #9-1 to #9-8) were used to compare the vacuum saturation, and saturation without vacuum (simple saturation) methods with both freshwater and gypsum saturated water. In the vacuum saturation method, samples were submerged in water and then samples and water were degassed under 85 kPa (25 in Hg, 1 in Hg = 25.4 mm Hg) vacuum simultaneously, for 23 d (552 h), while for saturation without vacuum, samples were only submerged in water for the same period. Samples were taken out of the water at different times, and their surface dried weights were measured to monitor the process of saturation and dissolution. To measure the mass of a surface dried sample, first the surface of sample was dried using a paper towel and then the sample was placed on a scale. At first the mass of sample was not stable and continuously decreased showing that more water was evaporating from the surface. After a short time, the weight change stopped showing that a surface dried sample was obtained. It is worth mentioning that if the sample is left on the scale for too long, the weight starts to decrease again due to the loss of moisture content internally. Therefore, the weight of sample on scale was closely monitored and as soon as the weight change became constant, the mass was used for the surface dried weight. For the improved vacuum method, two samples were saturated, one with water (sample #9-9) and the other (sample #9-10) in gypsum saturated water. For this method, samples were first degassed using 85 kPa (25 in Hg) vacuum for 24 h, and then submerged in degassed water. Vacuum (85 kPa (25 in Hg)) was then applied on submerged samples for 8 d (192 h). Samples were taken out at set times, and their surface was dried and their mass was measured. Removing samples from water at different times can also adversely affect the results, thus we carefully attempted this procedure as fast as possible to minimize the introduced errors. Lastly, 21 samples with different sizes (ten samples with $D=28$ mm and $H=56$ mm, five samples with $D=53$ mm and $H=112$ mm, and six samples with $D=76$ mm and $H=160$ mm) were saturated without vacuum using gypsum saturated water for 7 d (168 h). Samples used for this part of the test were #16-1, #16-2, #15-1, #15-2, #15-3, #17-1, #17-2, #17-3, #51-1, #51-2, #51-3, #1, #2-1, #2-2, #4, #3, #20, #12-1, #12-2, #19-

1 and #19-2. The surface dried weights were measured at different times using the same method discussed above.

2.4 Results and discussion

2.4.1 Oven drying results

The particle density of natural gypsum samples before and after drying in an oven for different periods is shown in Table 2-2. Before oven heating, samples had a particle density (ρ_s) of around 2.3 g/cm³, which is the average particle density for gypsum. The exception is rock block 8, i.e. samples #8 and #8 powdered (crushed sample), which had a particle density of 2.47 g/cm³. This higher particle density is due to the impurities which are mostly carbonates and shales with densities ranging from 2.7 g/cm³ to 2.8 g/cm³ causing the density to be higher. These impurities are shown as gray material in the samples, which can be seen in Figure 2-3a and b.

The results show that particle density of samples does not change when heating the samples up to 80 °C, indicating that no transformation (dehydration) has occurred. This is further supported by the work of Ossorio et al., (2014) who found that below 80 °C, gypsum in an aqueous solution is the primary phase. The test results also show that even prolonged heating (72 h, which is much longer than the time required for water content measurement) does not lead to the transformation of gypsum to hemihydrate. This, in fact, indicates that oven drying at 80 °C could be used for measuring the moisture content of gypsum as opposed to 60 °C suggested by ASTM D2216-10 (2010).

By raising the temperature to 105 °C, however, the particle densities start to increase (to around 2.6 g/cm³), indicating the transformation of gypsum to hemihydrate. Note that the difference between the particle density from rock block #8 and other samples has significantly reduced. This is because gypsum in sample #8 is transforming to hemihydrate with higher particle density, while the impurities with higher particle density are not changing. Thus, in this sample, there are two main ingredients with closer specific gravities compared to the starting two materials (gypsum and impurities). Therefore, the overall particle density of transformed samples with impurities (#8 dry and #8 powdered moist) are now fairly close to those of pure samples with fewer impurities.

The results further show that, raising the temperature to 204 °C slightly increases the sample particle densities but not achieve the particle density of anhydrite. Even heating the samples at 204 °C for 49 h did not increase the particle densities to that of anhydrite, suggesting that this temperature is not sufficient to transform hemihydrate to anhydrite. The slight increase does indicate that a small amount of gypsum that had been left untransformed in the sample is now transforming to hemihydrate.

Raising the temperature of heating to 320 °C in the furnace further increased the particle densities to around 2.8 g/cm³ but not as high as that of anhydrite (around 2.97 g/cm³).

Even temperature of 450 °C did not lead to the particle density of anhydrite. This density can be explained by the fact that, as described by Rolnick (1954), water in calcium sulfate minerals is not a zeolite form where the water is adsorbed by the mineral. The water in calcium sulfate minerals is indeed in the crystallization form (water is a part of the crystal), and there are only three possible forms for this mineral (gypsum-hemihydrate-anhydrite) and the only difference between these minerals are the amount of water in their crystals. In other words, unlike zeolite form, water cannot be continuously driven off by heating the samples. Based on this fact, it can be concluded that (as explained before by previous researchers like Rolnick (1954)), samples with particle densities between that of hemihydrate (2.6-2.7 g/cm³) and anhydrite (2.97 g/cm³) are not a separate mineral but are in fact, samples containing hemihydrate and anhydrite at the same time. The co-existence of hemihydrate and anhydrite in a sample after heating under high temperatures has been reported before by Hudson-Lamb et al. (1996). They used both pure gypsum and natural gypsum for their experiments and explained that while heating their samples at 450 °C, all the pure gypsum samples transformed to anhydrite, but in the case of natural gypsum samples (like our samples), hemihydrate was still present in the samples. In our tests, however, the particle density did not change from 320 °C to 450 °C, which indicates that 320 °C might be the temperature where the transformation of hemihydrate to anhydrite occurs. The result of this test also shows that the sample size and presence of water do not have a significant effect on the transition temperature under the “moisture condition test condition”.

Table 2-2. Particle densities (g/cm³) of different samples from helium pycnometer before and after drying out in oven and furnace for different periods of time.

Sample ID	Particle densities from helium pycnometer												
	Prior to heating	Dried in oven										Dried in furnace	
		24 h at 50 °C	48 h at 50 °C	72 h at 50 °C	24 h at 80 °C	48 h at 80 °C	72 h at 80 °C	48 h at 105 °C	144 h at 105 °C	24 h at 204 °C	49 h at 204 °C	45 h at 320 °C	48 h at 450 °C
#8 dry	2.47	2.47			2.47			2.67	2.67	2.71	2.71	2.81	2.81
#5 dry	2.3	2.3			2.3			2.59	2.58	2.62	2.63	2.79	2.8
#5-1 dry	2.31	2.31	2.3	2.31	2.31	2.31	2.31	2.6					
#5-1 saturated	2.31	2.31	2.31	2.31	2.31	2.32	2.31	2.59					
#5-1 in a water bath	2.3*	2.3	2.3	2.3	2.3	2.3	2.3	2.6					
#8 powdered moist	2.47*	2.47		2.47				2.67	2.67	2.71		2.8	2.8
#5 powdered dry	2.3							2.59	2.59	2.62		2.8	2.8

*The particle densities of moist samples were measured by helium pycnometer before adding water to them.

2.4.2 Air drying results

As noted above, we found that it took 16-18 h to oven-dry gypsum samples at 80 °C, while not transforming the gypsum to a hemihydrate by removing water molecules from the gypsum's internal structure but instead removing pore and adsorbed water. To

investigate the time it takes for air-drying, two saturated samples (#9-11 and #9-12) were air-dried in a laboratory at about 24 °C and average room humidity. The change in the sample weight compared to the initial saturated weight with time is shown in Figure 2-5. As it can be seen, all the weight change occurs in the first 30 h, after which the weight is constant, indicating that the sample is air-dried. The dry density of samples (before saturation) and also the density after saturation and air drying are presented in Table 2-3.

It is worth mentioning that, while the sample size was small compared to a mine pillar, it can be assumed that saturated mine pillars over the mine's life can lose their moisture at least on the pillar's surface and to some distance within the pillar. Groundwater can, however, still move vertically through the pillar, keeping the pillar's center region in a saturated condition. However, the pillar surface, which will be subjected to dissolution when the mine becomes flooded, will most likely be unsaturated, thus affecting the pillars dissolution rate at the beginning of flooding.

Figure 2-5. Results of air-drying the samples.

2.4.3 Microwave drying results

Microwave drying was used on two gypsum core samples, one air-dried and one saturated. The particle densities, before and after the test, are presented in Table 2-3. The particle density of the dry sample before and after the microwave drying was the same. For the saturated sample, on the other hand, the density increased after the microwave drying, showing that some gypsum to hemihydrate transformation has occurred. The increase in particle density is likely due to the transformation of gypsum to hemihydrate as a result of the presence of pore water that in turn generates higher temperatures from the microwave radiation. This confirms the ASTM suggestion that the microwave drying cannot be used for measuring the moisture content of gypsum rock samples. It also shows that while using the microwave drying, the presence of water has a significant effect on the transition process.

Table 2-3. Densities (g/cm^3) of samples from helium pycnometer before and after microwave drying.

Sample ID	Density from helium pycnometer	
	Before microwave drying	After microwave drying for 5 min
#9-11	2.3	2.3
#9-12 saturated	2.31*	2.41

*The dry density of saturated samples was measured by helium pycnometer before saturation.

2.4.4 Gypsum saturation results

The results of vacuum saturation and saturation without the vacuum (simple water immersion) for freshwater and gypsum-saturated water are shown in Figure 2-6. This figure shows the change in weight of the sample at different times compared to initial dry sample. In Figure 2-6, the solid lines show the vacuum saturated test results, while dashed lines show saturation without the vacuum. Comparing the two methods indicates that the use of a vacuum does not have a significant effect on the time required for saturation for freshwater or for gypsum-saturated water. The results also indicate that when the saturation is done with water only (blue and black lines), the gypsum dissolution is faster than the sample saturation and therefore the sample weight is decreasing (negative numbers in the chart). After a period of time, however, as the gypsum dissolution products concentration increases in water, dissolution slows down and eventually stops. The sample is already saturated at this point, but since the gypsum can form supersaturated solutions (as explained by Lebedev and Kosorukov, 2017), additional gypsum will dissolve, thus sample losing additional weight as the solution becomes oversaturated. After sufficient gypsum is dissolved and the solution becomes oversaturated, gypsum starts to precipitate, increasing the weight. This cycle of oversaturation and precipitation continues for a while until the equilibrium is achieved after about 240 h. Also, comparing the amount of weight change in samples #9-5 and #9-6 shows the effect of sample size on the process when only water is used. The larger sample experiences a larger weight loss due to the dissolution. That is due to the larger surface area in contact with water, which increases the dissolution.

In those tests started with gypsum-saturated water on the other hand, the sample gains weight as it begins to be saturated at first, but soon some of the gypsum starts to be dissolved again due to the ability of gypsum to form supersaturated solutions. The same cycle as explained before continues until equilibrium is reached again after about 240 h. In this case, however, size of the sample does not have a significant effect on the results. Inspecting the results shows that the major part of the weight change (which is due to saturation or dissolution in case of using unsaturated water) occurs in the first 30 h. The weight change after that is minimal, and is mostly due to the cycle of forming oversaturated solution and precipitation of gypsum from the oversaturated solution. Therefore, it appears that duration of 30 h is sufficient time for saturating the samples using both methods.

Figure 2-6. Saturation results of vacuum saturation method and saturation without vacuum method both with freshwater and gypsum-saturated water.

The improved vacuum saturation was also conducted on two samples (#9-9 and #9-10) using both freshwater and gypsum-saturated water, with the results shown in Figure 2-7. It can be seen from the results that the main weight change occurs within 24 h and after that, the weight change was minimal. As explained before, when the main weight change is reached, the samples can be considered saturated. The small changes after that are most likely due to the cycle of forming oversaturated solution and precipitation of gypsum as explained above. Therefore, the test was not continued beyond 192 h to show when the cycle reaches equilibrium. It can be concluded that improved vacuum saturation method did decrease the time required to saturate the samples from 30 h to 24 h. However, additional 24 h preparation time (i.e. 24 h for degassing the sample) is required, which makes the total time of 48 h to saturate the samples.

Comparing the results of three methods for samples with the same sizes, when gypsum-saturated water was used, indicates that the weight gain due to the saturation is about the same for all the three methods. This indicates that the samples could be saturated using any of these methods with the only difference being the time required to reach the saturation.

It should be noted, however, that the degree of saturation was not measured during this research. Saturation was assumed to occur when the weight change during saturation became constant. This was an attempt to obtain the samples as saturated as possible by using the methods commonly adopted in practice. As for the three methods investigated, only time of saturation varied.

To further investigate saturation time for different sample sizes, 21 more samples with different sizes were tested (using the simple immersion method) and the results are shown in Figure 2-8. The sample sizes are provided in Table 2-2. It can be seen that for all sample sizes, saturation is essentially complete after 30 h.

Figure 2-7. Results of improved vacuum saturation method with both freshwater and gypsum-saturated water.

Figure 2-8. Results of saturation without vacuum for samples with different sizes.

2.5 Conclusions

In this research, the drying and saturating of gypsum samples for mechanical testing were investigated. The main findings of the research are summarized below:

The gypsum-hemihydrate transition temperature under moisture content measurement condition is well above 80 °C.

Heating the gypsum sample at 80 °C even for 72 h does not appear to transform gypsum to hemihydrate, indicating that water content of gypsum samples can be measured using the oven drying method at temperatures up to 80 °C.

Microwave drying cannot be used to measure the water content of gypsum rock, although the transition from gypsum to hemihydrate does not appear to be significant.

The hemihydrate-anhydrite transition temperature under moisture content measurement condition is about 320 °C.

Even at 450 °C, all the hemihydrate in a natural sample will not totally transform to anhydrite and the sample will still contain both hemihydrate and anhydrite.

All three rock sample saturation methods can be used for saturation gypsum rock cores. Improved vacuum saturation was able to saturate gypsum within 24 h but required additional sample preparation time to place gypsum under vacuum before saturation (leading to a total time of 48 h for saturation). On the other hand, 30 h is required to

saturate the gypsum rock samples using vacuum saturation or saturation without vacuum methods.

The water should first become saturated with gypsum before it can be used to saturate the gypsum core samples to minimize the dissolution of the samples during the saturation process.

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Chapter 3

3 Mechanical properties of Michigan Basin's natural gypsum rock before and after saturation²

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3.1 Abstract

A stability analysis of an abandoned underground gypsum mine requires the determination of the strength of the mine pillars. This is especially important for flooded abandoned mines where the gypsum pillars become saturated after mining operations and are subjected to dissolution. Further, mine pillars are also subjected to blast vibrations that generate some level of macro and micro-fracturing in the pillar. Testing specimens of natural gypsum must, therefore, simulate these conditions as close as possible. In this research, the scale effect, as well as effect of blasting and saturation on uniaxial compression strength (UCS), elasticity moduli and splitting tensile strength (BTS), are investigated for natural gypsum rock in an investigation of the stability of an abandoned gypsum mine. New correlations were derived describing the effect of sample size on both UCS and BTS under dry and saturated conditions. Effects of blasting on these parameters were observed and the importance of choosing the proper samples is discussed. Point load index tests, which are usually used as a simple substitute for indirect estimation of UCS and BTS, were also conducted and correlations were derived for both compression and tensile strengths under dry and saturated conditions.

Keywords: *Uniaxial Compression Strength, Brazilian Splitting Tensile Strength, Point-Load Index, Scale effect, Michigan Basin gypsum*

3.2 Introduction

Determining the mechanical properties of gypsum rock is important in many engineering projects including the stability analyses of abandoned underground gypsum mines, especially for mines that become flooded after mining operations have ended. Uniaxial compressive strength (UCS) is the most widely used rock parameter used in design and stability analyses (Marolt Čebašek and Frühwirth 2018; Munoz and Taheri 2017; Salehin 2017; Salih and Mohammed 2017; Xie et al. 2011). This key rock strength parameter is

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usually measured in the laboratory using specific sized cylindrical samples selected from intact cores. Despite the importance of samples sizes and its effects on the rock's UCS, it is not always possible to prepare samples that meet the required standards, mainly due to the time-consuming and expensive process of sample preparation. The American Society for Testing and Materials (ASTM) recognizes this problem and allows samples with sizes outside the proposed range provided that "suitable notation" is made reporting the actual size of samples (ASTM D7012-14). In these cases, however, the correct UCS still needs to be determined (using the scale effect analyses) and the corrected UCS used in the stability analyses. Although scale effects have been studied for different rocks (e.g. (Hoek and Brown 1980; Yoshinaka et al. 2008)), it is important to investigate these effects for gypsum rocks, to be able to use the results of different sized samples where standard samples are not available. Correlations derived from scale effect analyses can also be used to investigate the existence of micro-flaws in samples (Yoshinaka et al. 2008). Micro-flaws in the rocks can be from natural geologic nature or caused by mining activities such as blasting used in underground mines or surface quarries.

An alternative approach to avoid time-consuming and expensive sample preparation for UCS, is to use indirect methods such as point load Index (PLI) tests to estimate the UCS (ASTM D5731-16). Although PLI tests can be conducted axially and diametrically on cylindrical samples as well as on samples with irregular shapes, the diametrical tests on cylindrical samples are considered the most reliable and are commonly used in design (Bieniawski 1975; Chau 1998; Heidari et al. 2012). PLI tests will not, however, eliminate the need for UCS tests as, despite extensive research on the correlations between the Point Load Index (PLI) and UCS, the empirical correlations are specific to each rock type and needs to be experimentally established for different rock type.

Another important parameter in the stability of underground mines is the roof rock's tensile strength. It is difficult to measure tensile strength of rock directly so an indirect method, the splitting tensile strength test (commonly known as Brazilian tensile strength (BTS) test), is used (ASTM D3967-16). The accuracy of the Brazilian test, however, is somewhat compromised due to excessive stress concentrations at load contact points (Wong and Jong 2014). Modifications are offered to reduce these stress concentrations such as using curved platens or curved spacers (Yu et al. 2009). According to ASTM, both modifications are acceptable, but they recommend using bearing cardboard strips with a thickness of 0.01 times the sample diameter between the sample and loading platen (ASTM D3967-16). BTS of rocks can also be estimated from PLI if the specific correlation between BTS and PLI are known.

Finally, in flooded abandoned mines, the left in place pillars that were unsaturated during the mining operations, start to become saturated. Although the effects of saturation on gypsum strength has been studied (e.g., (Ali 1979; Castellanza et al. 2007; Doktan 1983; Heidari et al. 2012; Hoxha et al. 2006; Lisk 1975; Yilmaz 2010)), there is a wide range of results. For example, a 22% reduction in UCS of gypsum reported by Lisk (1975), while Ali (1979) reported a 47-49% reduction. Further, Doktan 1983, tested gypsum samples from two areas and reported a 25% reduction in UCS of samples from one location and a

42% reduction in the second area (Doktan 1983). Dortan qualitatively attributed the differences between the two locations to different fabric, composition, grain size and weathering state of the rocks. Samples tested by Preston (1980) were obtained from different sources as well and showed 25.5, 25.9, 35.0 and 42.2 percent reductions in UCS after saturation. Doktan and Preston both reported that the highest reductions of strength were observed in the samples with coarsest grains (Doktan 1983; Preston 1980). In 2012, Heidari et al tested air-dried, and saturated samples from Gachsaran Formation, Iran, and their results show an average of 31.5% reduction in strength due to saturation (Heidari et al. 2012). While all the results show a strength reduction due to saturation, the quantitative value of reduction varies largely from case to case suggesting that the strength reduction should be determined experimentally for each project location.

In this study, mechanical properties of Michigan Basin's gypsum and the effects of sample size, blasting, and saturation on these properties were studied. This was necessary for the stability of an abandoned gypsum mine in Michigan, which is located under a busy highway. The main focus of this study was to (1), measure the mechanical properties of Michigan Basin's gypsum and understand the scale effect on its compressive and tensile strength by developing equations correlating sample size with UCS and sample size with BTS, (2) to determine the effect of blasting on UCS of gypsum rock and (3) to understand the effect of saturation on mechanical properties (UCS, Elasticity Modulus, BTS and PLI) of gypsum determined by testing dry and saturated samples and finally, (4) to establish correlations between PLI, UCS and BTS.

3.3 Site Location, Materials and Methods

This study is part of a project concerning the stability of the Domtar Mine, an abandoned underground gypsum mine near Grand Rapids, Michigan, USA (Figure 3-1). About 640 meters of a busy highway (interstate I-196) pass directly over a northeast portion of the Mine (Also known as Domtar Mine) located at a depth of about 30 meters below I-196. In some areas of the mine a lower series of gypsum seams were also mined. Over the mine's life subsidence and sinkholes have formed, raising concerns about the long-term stability of the mine underneath I-196. The 3.5 meters thick gypsum seam that had been mined on and off for over 150 years ending in 2000. While all of the underground gypsum mines in the Michigan Basin are now closed, there are still two operating quarries near Tawas City, on the east side of the state, adjacent Lake Huron. The Domtar mine's gypsum seam is part of Michigan Formation (Mississippian age) in the Michigan Basin. After mining ended in 2000, the mine was reported flooded by 2003 (Vitton 2004). Since the mine is flooded, our access to collect samples for testing was limited and therefore samples from Tawas quarry (shown in Figure 3-1), which belongs to the same formation (Grimsley 1904) was used for this research.

Gypsum blocks were obtained from the National City Quarry near Tawas, MI and transported to the Rock Mechanics Laboratory at Michigan Technological University. Samples were cored at the following three diameters: (1) 2.8 cm (1.1 in), (2) 5.4 cm (2.1 in) and (3) 7.6 cm (3 in)). Following coring the samples were cut on a diamond saw to a

Height (H) to Diameter (D) of equal or greater than 2.1. An attempt was made to surface grind the cores but was unsuccessful due to gypsum dissolution and breakage of the samples during grinding. The parallelism of the samples, however, was measured using the device shown in Figure 3-2D, to make sure they are in an acceptable range. Half of the prepared samples were air dried for eight weeks before testing, while the other half was saturated using the methods explained in (Sadeghiamirshahidi and Vitton Submitted March 2018). The samples that broke during surface grinding or the ones that did not meet the parallelism requirements were then cut to smaller samples for point load tests or to disks for Brazilian test. Half of these samples were also air dried while the other half was saturated before testing started.

Figure 3-1. Study area (Map was developed using ArcMap version 10.5.1, and the Data for developing the map was obtained from USGS (USGS))

Gypsum samples with different diameters were prepared to study the effect of sample size on strength parameters. Hoek And Brown (Hoek and Brown 1980) addressed the

importance of sample size and provided a correlation between sample size and a dimensionless form of UCS (Eq. 1), as follows:

$$UCS/UCS_{(50)} = (50/d)^{0.18} \quad (1)$$

Where $UCS_{(50)}$ is the uniaxial compressive strength of samples with a diameter (D) of 50 mm and (d) is the diameter of samples in millimeters (Hoek and Brown 1980). The correlation is based on published experimental results for different rock types. Because the relationship is based on various rock types, there can be a difference between the actual strength and the strength predicted by the correlation for some rock types. Other researchers have also developed similar but improved correlations, among which equation (2) proposed by (Yoshinaka et al. 2008) is one of the more common correlations.

$$UCS/UCS_{(50)} = (d_e/d_{e50})^{-k} \quad (2)$$

In Eq. 2, d_e is equivalent length (which is defined as the cube root of the sample volume), d_{e50} is the equivalent length of the standard sample ($D=50\text{mm}$), and K is a material constant that depends on rock type (hard or soft) and the presence or absence of rock micro-flaws. They categorized rocks into two groups, soft rocks that they defined as rocks with UCSs less than 25MPa, and hard rocks with UCSs greater than 25 MPa. Based on their laboratory and in-situ tests results they concluded that K can vary from 0.1 to 0.3 for the hard rock without micro-flaws and from 0.3 to 0.9 for highly micro-flawed hard rock while it is always less than 0.5 for soft rocks (Yoshinaka et al. 2008). These micro-flaws in the rocks can be from natural geologic processes or possibly from blasting operations used in underground mines or surface quarries. In the early stages of mine design, the UCS is usually measured from cores from a drilling program not subjected to blasting. This can result in overestimating the strength of pillars when blasting is used as part of the mining operations.

Figure 3-2. Apparatuses used in the Lab for A) UCS; B) BTS; C) PLI; and D) Parallelism measurement

After the preparations, UCS of dry and saturated samples were measured using a MTS rigid frame servo-hydraulic compression machine with a top bearing platens as shown in Figure 3-2A. The UCS of samples, as well as all three types of elasticity modulus (Secant, Tangent and the Average slope of the straight-line portion of the stress-strain curve) were calculated for both dry and saturated sample. An MTS Static-Hydraulic compression machine (Figure 3-2B) was used for BTS testing, and a portable point load testing machine (Figure 3-2C) was used to conduct the PLI tests. Both BTS and PLI tests were conducted on dry and saturated samples. For PLI tests, however, only samples with diameters of 2.8 cm were tested. In addition, a portable X-ray fluorescence analyzers (PXRF) was used to study the composition of the gypsum. Each sample was scanned three times (each time for 30 seconds) using the soil mode which is the optimized option for detecting lighter elements (Ca, K, S, P, Cl and I). Finally, carbonate content (calcite equivalent) of the samples were measured according to ASTM D4373-14 using hydrochloric acid (HCl) in an enclosed reaction cylinder.

3.4 Results and discussion

3.4.1 Gypsum Composition Results

Gypsum samples from Tawas quarry had two distinctive white and pink colors along with secondary gray impurities. To determine the composition of samples, carbonate content tests were conducted on the samples. The results of this testing are summarized in Table 3-1. As it can be seen the samples with the higher gray impurities had the highest carbonate content (about 8-10%), while the white and pink samples with little to no gray impurities had the lowest carbonate contents at about 1%.

Table 3-1. Results of carbonate content tests

Sample ID	Comment	CaCO ₃ (wt%)
CC1	Pink- with some gray impurities	2.14
CC2	Significant gray impurities	8.16
CC3	Pink-with some gray impurities	2.73
CC4	Significant gray impurities	5.49
CC5	Significant gray impurities	9.52
CC6	White	1.30
CC7	White	1.30

Table 3-2. Summary of PXRF results

Sample ID	Comment	S (wt %)	Ca (wt %)	Fe (wt %)	Sr (wt %)
PXRF1	White	52.27	32.73	0.01	0.09
PXRF2	Gray	23.68	35.57	0.74	0.23
PXRF3	White	54.78	34.19	0.01	0.09

PXRF4	Pink	50.96	31.81	0.01	0.03
PXRF5	Gray	47.88	34.90	0.06	0.03
PXRF6	Pink	53.16	33.07	0.01	0.09
PXRF7	Pinkish white	48.84	32.23	0.02	0.12
PXRF8	Dissolved in flowing water for 24 h and then oven dried at 105 C for 48 h	61.75	36.94	0.02	0.08

The PXRF results of eight samples, which determine the percentage by weight of main components, are presented in Table 3-2. The samples were scanned to determine the composition of the three colors. While calcium (Ca) can be from either gypsum or calcium carbonates, sulfur (s) would represent gypsum. As it can be seen, the percentage by weight of sulfur (s) is lower for gray areas showing a higher percentage of carbonate which is in agreement with the carbonate content test results. The PXRF results also indicate that the composition of all samples are relatively consistent.

3.4.2 Uniaxial Compression Test Results

Uniaxial compression strengths of air-dried samples with different sizes are summarized in Table 3-3. To better indicate the relationship between sample size and the UCS, uniaxial compression strengths of samples are plotted against sample diameters and presented in Figure 3-3. It can be seen from this plot that, as expected, larger samples have lower strengths. This is generally believed to be due to a larger amount of flaws existing in larger samples. The plot also shows, however, a larger scatter in the data for smaller samples compared to larger samples. One possibility for this larger scatter might be due to production blasting causing a higher percent of micro-fracture in the gypsum. While there is a chance of obtaining a small sample with minimum to no cracks (flaws) from blasted rocks, there still might be some small samples with some cracks (see Figure 3-4). For larger samples, on the other hand, there always most likely always be cracks in the sample (Figure 3-4). Therefore, since the number of flaws controls the strength of rock, small samples show more substantial variance from the average. To further investigate this matter, the average UCS for each size was calculated and plotted on the chart developed by Hoek-Brown (Hoek and Brown 1980) as shown in Figure 3-5. To develop this chart, the data presented by Hoek-Brown was approximated using PlotDigitizer (version 2.6.8) and plotted along with our results. Hoek and Brown used specimens with $D = 50$ mm as their reference (standard) and developed their equation (Eq. 1) which is also included in the chart (Figure 3-5). As the closest diameter we had in our tests to their standard diameter was 54 mm, we also plotted the Hoek-Brown's equation using $D = 54$ mm as the reference which is shown with the dashed line in Figure 3-5. As it can be seen two curves are very close to each other, meaning the change of reference diameter from 50 to 54 mm does not hugely affect the results. This allows us to develop a similar equation for Michigan Basin's Gypsum using the UCS of samples with $D=54$ mm ($UCS_{(54)}$) as the reference as shown in Figure 3-6. As it can be seen, a similar equation to that of the Hoek-Brown, only by changing the exponent in the equation to 0.81, describes the effects of sample size on the strength of Michigan Basin's Gypsum.

Table 3-3. Summary of UCS tests results on dry samples

Sample ID	Sample Diameter (mm)	Dry UCS (MPa)	Tangent Modulus (GPa)	Secant Modulus (GPa)	Average Modulus of Linear Portion of Axial Stress-Strain Curve (GPa)	Failure Mode	Dry UCS ₍₅₄₎ (Mpa)
DRY-1	28.24	29.14	2.75	1.51	3.99	Axial Splitting	17.60
DRY-2	28.27	30.64	3.42	1.62	3.75	Axial Splitting	18.53
DRY-3	28.26	26.21	3.67	1.67	3.65	Axial Splitting	15.84
DRY-4	28.23	25.10	2.79	1.57	4.35	Axial Splitting	15.16
DRY-5	28.22	48.31	6.31	2.76	7.00	Shearing along single Plane	29.17
DRY-6	28.25	43.97	5.86	2.45	6.77	Shearing along single Plane	26.58
DRY-7	28.21	11.97	1.10	0.57	0.99	Axial Splitting	7.22
DRY-8	28.27	15.93	1.35	0.81	1.15	Axial Splitting	9.63
DRY-9	28.31	34.68	4.08	1.91	4.53	Axial Splitting	20.99
DRY-10	28.33	37.54	4.00	1.65	4.07	Multiple Fracturing	22.74
DRY-11	28.37	38.49	5.24	2.76	7.65	Multiple Fracturing	23.35
DRY-12	53.03	23.95	4.30	2.55	4.28	Multiple Fracturing	24.15
DRY-13	53.95	11.35	2.15	1.31	2.27	Axial Splitting	11.61
DRY-14	54.07	14.76	2.76	1.36	4.51	Shearing along single Plane	15.12
DRY-15	54.04	16.28	3.81	1.74	3.67	Shearing along single Plane	16.67
DRY-16	54.1	26.94	4.96	2.49	5.46	Multiple Fracturing	27.62
DRY-17	75.71	20.85	6.61	2.53	7.00	Y-Shape	28.08
DRY-18	75.78	11.84	2.73	1.63	2.77	Shearing along single Plane	15.96
DRY-19	75.77	14.17	4.05	2.03	4.48	Shearing along single Plane	19.09
DRY-20	75.82	14.00	4.13	2.00	4.47	Shearing along single Plane	18.88
DRY-21	75.82	14.72	1.99	1.04	3.43	Multiple Fracturing	19.86
DRY-22	75.65	8.78	2.22	0.93	2.06	Shearing along single Plane	11.82
DRY-23	75.7	12.81	4.39	1.79	5.13	Shearing along single Plane	17.26

Figure 3-3. UCS of air-dried samples with different sizes

Figure 3-4. Schematic demonstration of flaws in a rock block before and after blasting and its effect on core samples

Equation 2 developed by (Yoshinaka et al. 2008) was also used where $k=0.81$ fitted with our results as shown in Figure 3-6. According to the criteria provided by (Yoshinaka et al. 2008), having, k , in the range of 0.3 to 0.9 represents highly micro-flawed hard rock which further suggests the possible effects of blasting on our samples.

UCS of saturated samples with different sizes were also measured, and results are summarized in Table 3-4, and the relation between sample sizes and the UCS are shown in Figure 3-7. The same charts explaining the size effect for saturated samples were also plotted in Figure 3-8. For saturated gypsum, the exponent in the Hoek-Brown equation was calculated to be 0.72 (Figure 3-8) and the, k , in the (Yoshinaka et al. 2008) equation was 0.72 which again suggests the micro-flawed condition of samples due to blasting. The comparison between saturated and air-dried samples shown in Figure 3-9 indicate a 41% reduction in UCS after saturation. It is worth mentioning that for both saturated and

dry samples, larger samples, ($D > 2.8\text{cm}$) retained some residual strength after failure meaning the failure was not as brittle as shown in Figure 3-10a, but almost all of the small samples ($D=2.8\text{cm}$) had abrupt brittle failures (Figure 3-10b). Three types of elasticity modulus (shown in Figure 3-10) were calculated for all samples (dry and saturated), and the averages are tabulated in Table 3-5. The results show that all the moduli were reduced by between 30 to 50% after saturation. Four types of failure mode, axial splitting (Figure 3-11a); single shear (Figure 3-11b); multiple fracturing (Figure 3-11c) and Y-shape failure (Figure 3-11d), were seen in dry samples but in saturated samples, almost all of them failed along a single shear plane. Despite some hypotheses in the literature trying to explain these failure modes for example by using damage evolution of the rocks and correlating the failure mode to strength or sample size as explained by (Basu et al. 2013), no such correlation could be established for gypsum from the results of our tests. This could be because of blasting effects on our samples.

Figure 3-5. Average of Gypsum UCSs for different sizes Plotted on Hoek-Brown's Chart (After Hoek-Brown 1980 (Hoek and Brown 1980))

3.4.3 Point Load Index Tests

The results of PLI tests of dry and saturated samples are summarized in Table 3-6. To derive the correlation between UCS and PLI, the results of PLI tests (I_{s50}) for both dry and saturated samples have been plotted versus UCS tests results as shown in Figure 3-12 and Figure 3-13, respectively. It should be noted that all the UCS results were converted to $UCS_{(54)}$ using the equation 3 for dry samples and equation 4 for saturated samples that were previously derived for Michigan Basin gypsum (Figure 3-6 and Figure 3-8, respectively) before plotted against I_{s50} .

$$UCS/UCS_{(54)} = (54/d)^{0.81} \quad (3)$$

$$UCS/UCS_{(54)} = (54/d)^{0.72} \quad (4)$$

It should also be noted that the trend lines were forced to pass through zero as the authors believe that having an arbitrary constant (y-intercept) without any physical explanation in the equation is unwarranted. Furthermore, forcing the trend line through the origin did not significantly reduce the coefficient of determination, R^2 . Also, the definition used by ASTM-D5731 (general form of $UCS = K I_{s50}$) (ASTM D5731-16) for “index to strength conversion factor, (k)” does not include any arbitrary y-intercept. It is also important to mention that, to develop these charts, all the UCS results and PLI results were sorted from lowest to highest and plotted against each other. The index to strength conversion factor, k, was 6.6 for dry samples and 7.7 for saturated samples.

Figure 3-6. Different criteria developed to describe the influence of sample size on UCS of air-dried natural gypsum from Michigan Basin

Table 3-4. Summary of UCS tests results on saturated samples

Sample ID	Sample Diameter (mm)	Saturated UCS (MPa)	Tangent Modulus (GPa)	Secant Modulus (GPa)	Average Modulus of Linear Portion of Axial Stress-Strain Curve (GPa)	Saturated UCS ₍₅₄₎ (MPa)
SAT-1	28.24	14.26	1.23	0.91	2.50	8.56
SAT-2	28.27	15.34	2.25	1.51	3.00	9.21
SAT-3	28.26	15.78	1.99	1.61	2.02	9.47
SAT-4	28.23	13.34	1.83	1.23	2.02	8.00
SAT-5	28.22	23.29	3.12	1.92	3.34	13.97
SAT-6	28.25	20.00	3.69	2.33	4.18	12.01
SAT-7	28.21	8.84	0.72	0.88	0.98	5.30
SAT-8	28.27	11.56	0.61	0.47	1.12	6.94
SAT-9	28.31	14.90	1.10	1.01	2.21	8.96
SAT-10	28.33	27.09	3.20	2.03	3.97	16.29
SAT-11	28.37	30.25	3.15	2.01	3.76	18.21
SAT-12	53.03	9.94	1.36	0.80	1.96	9.39
SAT-13	53.95	9.90	1.00	0.88	2.62	9.47
SAT-14	54.07	10.06	1.37	0.90	2.45	9.63
SAT-15	54.04	9.34	0.18	0.36	0.43	8.94
SAT-16	54.1	12.96	0.74	0.49	1.02	12.42
SAT-17	75.71	10.87	1.45	1.64	2.76	13.27
SAT-18	75.78	11.13	2.33	1.23	4.41	13.60
SAT-19	75.77	6.07	2.23	1.35	2.21	7.41
SAT-20	75.82	7.73	2.76	1.78	3.34	9.44
SAT-21	75.65	7.11	2.24	1.54	2.82	8.67
SAT-22	75.7	9.97	2.28	1.46	2.50	12.17

Figure 3-7. UCS of saturated samples with different sizes

Figure 3-8. Different criteria developed to describe the influence of sample size on UCS of saturated natural gypsum from Michigan Basin

3.4.4 Brazilian Tensile Strength Results

Indirect tensile strength of air-dried samples with three diameters was measured using Brazilian Test (BTS). The results are summarized in Table 3-7, and the relation between

sample sizes and the BTS are provided in Figure 3-14. As it can be seen from Figure 3-14, the BTS results also decreases with increasing sample size, but in this case, the scattering of results for all sizes are similar. This is because samples in BTS, compared to UCS samples, are smaller and the chance of having samples intact or with some micro-flaws is relatively similar for all three sizes. An attempt was made to develop a criterion explaining the relationship between sample size and BTS and the results are shown in Figure 3-15. The coefficient of determination (R^2) is about 0.86, which is lower than coefficients for UCS, which could be due to a wider range of thickness to diameter ratios used in these tests (between 0.3 to 0.7). The range of sample height to diameter for UCS was relatively constant around 2.1 (2.04 to 2.2). Unfortunately, a larger number of samples with different diameters were not available to determine such a relationship for saturated BTS.

From the correlation between sample size and BTS (equation 5), $BTS_{(54)}$ was calculated for all samples and plotted against I_{s50} as shown in Figure 3-16.

$$BTS/BTS_{(54)} = 0.81 (54/d)^{0.92} \quad (5)$$

Figure 3-9. Comparison between air-dried and Saturated UCS

Figure 3-10. Two typical types of stress-strain curves obtain for gypsum samples tested in this study

Table 3-5. Three type of Elasticity Moduli calculated for Dry and Saturates samples

Modulus (GPa)	Air-Dried Samples	Saturated Samples
Secant	1.77	1.2
Tangent	3.68	1.86
Linear Portion	4.11	2.53

As it can be seen in Figure 3-16, the coefficient of determination (R^2) between BTS and PLI is very low (0.35) meaning the correlation is not very reliable for use in practice. For this case again the trend line was forced through the origin. It is worth mentioning that, not forcing the line through zero increases the coefficient of determination to 0.82 changing the correlation to $BTS(54) = 0.86I_{s50} + 4.3$ which is not preferred by the authors due to lack of any physical explanations for the random y-intercept in the equation.

Heidari et al, 2012 have conducted both axial and diametrical PLI tests on gypsum samples and reported that although diametrical PLI tests show a good correlation with UCS, there is a significant reduction in correlation between PLI and BTS. They attributed this reduced correlation to inhomogeneities in the gypsum samples, e.g., the presence of micrite veins or microflaws in the samples, as they act as a weakness plane (Heidari et al. 2012). Failure planes of samples for BTS tests are shown in Figure 3-17, which shows that they all split in half despite having discontinuities (gray veins of different material mostly carbonates and shale) with different a direction in respect to applied load. This indicates that heterogeneity of samples did not play an important role in failure. We suggest instead, however, that blast induced microflaws might be the key factor controlling the failure in the samples. Failure planes in PLI tests (as shown in Figure 3-18) were also independent of the direction of discontinuities. It is worth mentioning that, independency of failure mode and the direction of discontinuities for sedimentary rocks have also been reported by (Li and Wong 2012). They speculated that the low-grade metamorphism fuse the beddings of sedimentary rocks and prevent them from acting as a weakness plane.

Figure 3-11. Failure modes observed in UCS testing of Michigan Basin Gypsum

Table 3-6. Summary of PLI tests results on dry and saturated samples

Sample ID	Dry Is(50) (Mpa)	Sample ID	Saturated Is(50) (Mpa)
IsDRY-1	1.14	IsSAT-1	0.70
IsDRY-2	1.69	IsSAT-2	0.80
IsDRY-3	1.69	IsSAT-3	0.84
IsDRY-4	1.73	IsSAT-4	0.88
IsDRY-5	1.78	IsSAT-5	1.14
IsDRY-6	1.79	IsSAT-6	1.17
IsDRY-7	1.84	IsSAT-7	1.17
IsDRY-8	2.49	IsSAT-8	1.17
IsDRY-9	2.73	IsSAT-9	1.24
IsDRY-10	2.78	IsSAT-10	1.28
IsDRY-11	2.99	IsSAT-11	1.40
IsDRY-12	3.00	IsSAT-12	1.59
IsDRY-13	3.03		
IsDRY-14	3.14		
IsDRY-15	4.19

Figure 3-12. UCS₍₅₄₎ VS. IS(50) for Air-Dried samples

Figure 3-13. UCS₍₅₄₎ VS. IS(50) for Saturated samples

Table 3-7. Summary of the Brazilian Tensile strength tests results

Sample ID	Diameter (mm)	Splitting tensile strength (MPa)	BTS(54)	Sample ID	Diameter (mm)	Splitting tensile strength (MPa)
BTSDry-1	28.19	8.18	5.54	BTSSat-1	75.69	1.95
BTSDry-2	28.19	9.06	6.15	BTSSat-2	75.47	1.99
BTSDry-3	28.30	8.86	6.03	BTSSat-3	75.51	1.42
BTSDry-4	28.15	8.87	6.01	BTSSat-4	75.59	2.22
BTSDry-5	28.30	8.06	5.49	BTSSat-5	75.91	2.85
BTSDry-6	28.29	8.07	5.49	BTSSat-6	75.92	2.53
BTSDry-7	28.26	8.13	5.52	BTSSat-7	75.76	2.82
BTSDry-8	28.20	7.84	5.32	BTSSat-8	54.33	5.05
BTSDry-9	28.33	12.32	8.39	BTSSat-9	53.41	2.95
BTSDry-10	28.49	11.76	8.05			
BTSDry-11	28.30	10.28	7.00			
BTSDry-12	54.20	5.71	7.06			
BTSDry-13	54.20	4.85	6.01			
BTSDry-14	53.98	7.93	9.78			
BTSDry-15	53.90	8.47	10.42			
BTSDry-16	54.04	6.95	8.57			
BTSDry-17	75.80	2.48	4.18			
BTSDry-18	75.90	2.85	4.81			
BTSDry-19	75.78	3.66	6.16			
BTSDry-20	75.74	3.65	6.15			
BTSDry-21	75.75	4.53	7.63			
BTSDry-22	75.81	3.45	5.81			
BTSDry-23	75.68	3.81	6.40

Figure 3-14. Splitting Tensile Strength (BTS) of samples with different diameters

Figure 3-15. Sample size effect on BTS of gypsum

Figure 3-16. Correlation between I_{s50} and BTS(54) for dry samples

Figure 3-17. Failure modes in BTS tests: the red dashed lines are drawn just next to the failure planes. a) sample with $D=7.6$ cm, b) Two separated sides of the sample “a” with the location of discontinuity just to the right of the failure plane highlighted with dashed yellow line, c) sample with $D=5.4$ cm and d) sample with $D=2.8$ cm, the black veins (discontinuities) in samples “c” is almost perpendicular to the failure plane while in sample “d” they are almost parallel to the failure plane

Figure 3-18. Failure modes in point load tests in samples with discontinuities situated at different directions, in respect to the applied point load (sample b with no apparent discontinuity)

Figure 3-19. Correlation between BTS of saturated samples with $D=7.6$ cm and Saturated I_{s50}

BTS of nine saturated samples were also tested (Table 3-7). The results were plotted against saturated I_{s50} and shown in Figure 3-19. As mentioned previously, there were not enough samples with different diameters to determine the size effect on the BTS of saturated gypsum, so the correlation only represents the relationship between BTS of the samples with 7.6 cm diameter and I_{s50} . Although the determination coefficient of 0.93 shows a high correlation between BTS and I_{s50} , it can be used only if the BTS is measured using samples with a 7.6 cm diameter.

3.5 Conclusions

Mechanical properties of Michigan Basin's gypsum in dry and saturated conditions were investigated in this study. Based on the results of this study the following conclusions were provided:

- For the gypsum samples from the Michigan Basin, the average standard uniaxial compression strength, $UCS_{(54)}$, of dry gypsum was around 19 MPa and 11 MPa for saturated gypsum indicating a 41% reduction due to the saturation. While these results are for blasted rock, the intact dry rock with minimum flaws $UCS_{(54)}$ can be as high as 29 MPa and 18 MPa for intact saturated rock. These intact rock strengths were estimated by calculating the highest strength from smallest samples ($D=2.8$ cm) and converting it to $UCS_{(54)}$ using the equations derived in this paper.
- The elastic moduli of samples decreased by 30 to 50 percent due to saturation.
- The point load index to uniaxial compressive strength conversion factor of 6.6 for dry samples and 7.7 for saturated samples were measured for blasted gypsum from Michigan Basin.
- Testing samples with a wider range of sizes, the scatter in the test results are more noticeable in smaller samples than in larger samples. This is because when working with small samples, one sample could be prepared with minimum flaws while the concentration of flaws in another sample is high. In contrary, almost all of the large samples have a high amount of micro-flaws. On the other hand, the Brazilian test results tend to have less scatter since the range of sample sizes is less than with the UCS size samples.
- Scale effect on uniaxial compression strength of dry and saturated gypsum is different and can be presented using equations 3 and 4. The size of the samples has a similar effect on splitting tensile strength, which can be estimated using equation 5.
- While the point load index and splitting tensile strength of dry samples did not show a high correlation, a point load index to splitting tensile strength conversion factor of 2.4 was found for saturated samples with 7.6 cm diameter.

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Chapter 4

4 Laboratory study of gypsum dissolution rates for an abandoned underground mine³

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4.1 Abstract

Groundwater reestablishment in abandoned gypsum mines causes pillar dissolution until the groundwater reaches the groundwater's gypsum saturation potential. In some cases, however, especially in shallow mines, groundwater can continue to flow through the mine resulting in the additional dissolution of the mine's support pillars leading to possible pillar collapse. Pillar dissolution will depend on the amount and quality of the groundwater flowing through the mine and the dissolution rate of the gypsum. The Domtar Mine in Grand Rapids Michigan operated for over 140 years mining a 30-meter-deep high-quality gypsum deposit. In 2000, the mine was abandoned with the removal of the mine's dewatering pumps that allowed the groundwater to flood the mine. The mine is located along the north side of the Grand River and has groundwater flowing through the mine to the Grand River. Interstate I-196 is located over the east side of the mine. To analyze the stability of the mine's pillars, the gypsum dissolution rate was investigated. In this research, laboratory experiments were conducted to investigate the dissolution rate of natural gypsum in both stagnant and flowing water. Existing published dissolution rates were reviewed and compared to the investigation's results. The tests confirmed that the dissolution can be represented by a first-order kinetic equation. The normalized dissolution coefficient was measured for stagnant water at 1.6×10^{-3} (cm/s) following the power law of $k = 0.0021F^{0.1854}$ for flowing water, where F is the water's flow rate and k is dissolution coefficient.

Keywords: *Abandoned underground mine, long-term Stability, Gypsum dissolution, first-order kinetics, Domtar Gypsum Mine, Michigan basin gypsum*

4.2 Introduction

Underground mines located below the groundwater table require dewatering operations. Once mining is completed, however, pumping is discontinued allowing the mine to fill

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with groundwater (Doulati Ardejani et al. 2013). This process, referred to as groundwater rebound, results in reduced pillar strength of moisture sensitive rock due to an increase of moisture as the pillars become saturated. This phenomenon has been studied and discussed by numerous researchers (e.g., (Auvray et al. 2008; Ballivy et al. 1976; Guha Roy et al. 2017; Karakul and Ulusay 2013; Masuda 2001; Preston 1980; Shukla et al. 2013; Vergara and Triantafyllidis 2016; Yilmaz 2010; Zhou et al. 2016a; Zhou et al. 2016b)). In addition to saturation, groundwater rebound in gypsum mines also results in dissolution of the mine's support pillars. Dissolution will continue until the groundwater becomes saturated with respect to gypsum dissolution products. While the groundwater's geochemistry can be complex and dependent on many variables, in general, once the groundwater is at saturation, gypsum dissolution will cease, and in some cases, precipitation can occur. In open systems, however, when groundwater continues to flow through the mine, such as in the case of shallow mines in areas of elevated topography, dissolution can continue. In this situation, pillar strength is reduced resulting in mine instability and possible failure (collapse). The time to failure (if failure does occur) depends mainly on the gypsum's dissolution rate (Castellanza et al. 2007).

Knowledge of gypsum dissolution rates is important in many areas including (but not limited to) gypsum karst formation, deformation of natural gypsum rocks, amending acidity of soil for agricultural purposes, drinking water quality, and water motion measurements (Colombani 2008; Gorban and Miyamoto 1985; Hoxha et al. 2006; Jeschke et al. 2001; Pachon-Rodriguez and Colombani 2013; Raines and Dewers 1997). In these cases, an understanding of the process and gypsum dissolution rate was obtained through experiments using different specimen preparation procedures and specimen sizes. In a study on karst formation, for example, standard specimens sizes (also called tablets) of 40–45 mm in diameter and 7–8 mm in thickness were used (Klimchouk and Aksem 2005). The following two methods are commonly used to calculate the gypsum dissolution rate in gypsum karst formation studies: a) measuring gypsum tablet weight loss in the field or in simulated conditions in the lab, e.g. Klimchouk and Aksem (2005); and b) micro-erosion measurements of surface thicknesses erosion, e.g. Ford and Williams (2007). In agricultural and soil sciences, on the other hand, two gypsum specimen sizes are generally used. The first method uses gypsum fragments (particles of powdered gypsum) with sizes from less than 0.5 mm to 40 mm (Bolan et al. 1991; Kemper et al. 1975). The second method uses gypsum discs of about 15 mm in diameter and 2 mm thick. The gypsum discs are prepared by either cutting natural rocks or pressing gypsum powder into a die under maximum pressure of about 1.5 MPa (Bolan et al. 1991). In these studies, batch, centrifuge, and column tests are conducted to determine the gypsum dissolution rates. To investigate natural gypsum rock deformation under dam foundations, cubic specimens sizes of about 100 mm cubed or larger are used (Aljubouri and Al-Kawaz 2007).

Not only are different specimen sizes and tests used in gypsum dissolution studies, but also different kinetic laws and equations are used to model the gypsum dissolution behavior. For example, Kemper et al. (1975) used a first-order reaction model of gypsum dissolution rate in flowing water assuming that the reaction was proportional to the

difference between the solution concentration at saturation (C_s) and the solution concentration at the time the solution comes into contact with gypsum (C). This form of first-order reaction is represented by equation 1.

$$dm/dt = K(C_s - C) \quad (1)$$

where (m) is the gypsum mass, (t) is time and (K) is the dissolution coefficient, which is a function of water flow velocity, temperature, and chemistry of flowing water. In addition to these inherent dependencies, this definition makes the dissolution coefficient (K) dependent on the surface area of gypsum in contact with water. A similar kinetic equation was developed later by James and Lupton (1978) to eliminate the dependency on the surface area as shown in equation 2.

$$dm/dt = kA(C_s - C) \quad (2)$$

where A is the surface area of gypsum in contact with water. It is worth mentioning that although the two definitions are similar, the units of the coefficients are different. In equation 1, mass (m) is expressed in grams, time (t) in seconds (s) and concentrations (C_s and C) are in grams per centimeter cubed (g/cm^3) resulting in dissolution coefficient (K) having units of centimeter cubed per seconds (cm^3/s). Using the same units and centimeter squared (cm^2) for surface area (A) leads to the units of centimeters per seconds (cm/s) for the normalized dissolution coefficient (k) in equation 2. A similar equation (Eq.3) has also been used by researchers such as (Bolan et al. 1991) in which the gypsum concentration is used as the dependent variable (dC) instead of gypsum mass (dm).

$$dC/dt = K'(C_s - C) \quad (3)$$

Again, the definition used in equation 3 leads to a different unit for the concentration based coefficient (K') which is one over seconds (s^{-1}). Despite differences in these expressions, they are based on the same premise that gypsum dissolution is kinetically a first-order reaction, which has been used by many researchers (e.g. (Keisling et al. 1978; Kemper et al. 1975; Keren and Shainberg 1981)).

Some researchers, however, have reported that a second order reaction better represents the dissolution reaction. Gorban and Miyamoto (1985) measured the dissolution rate of gypsum particles in both distilled water and salt solutions and found that first-order kinetics did not accurately predict the dissolution rates when the concentration of calcium ions in solution was in excess of 50% of the gypsum's solubility. Their research data suggest that a second-order kinetic reaction (Eq.4) better models the dissolution process.

$$dC/dt = K''(C_s - C)^2 \quad (4)$$

Equation 4 leads to yet another definition for the second order dissolution coefficient (K'') with units of ($\text{mol}^{-1}\text{cm}^{-3}\text{s}^{-1}$). Following this study, Frenkel et al. (1989) confirmed that Ca^{2+} and SO_4^{2-} concentrations control the order of reaction. When Ca^{2+} and SO_4^{2-} ions are removed from the solution, for example by resins that they used in their experiments, gypsum dissolution becomes a first-order reaction; otherwise, it follows second order kinetics. This is in agreement with research conducted by Berner (1981), who explained that Ca^{2+} and SO_4^{2-} come into solution from the gypsum surface at higher rates than are transported away from the surface. Since the reaction rate is controlled by the slowest process, gypsum dissolution is a transport-controlled process. Building on these findings, Bolan et al. (1991) later measured gypsum dissolution rates in soils and water and concluded that in soil, gypsum dissolution is a first order process since the soil acts as a sink for Ca^{2+} and SO_4^{2-} ions, while in water it is a second-order process.

Clearly, the gypsum dissolution rate depends on the environment where the dissolution is occurring. Furthermore, even with investigations using the same kinetics equations, the range of dissolution rates derived from experiments varies dramatically. For example, although studies concerning karst formation usually use first order kinetics, the range of gypsum dissolution coefficients reported (e.g. (Ford and Williams 2007; Klimchouk and Aksem 2005)) has a wide range from 1.83×10^{-7} to 2.46×10^{-4} (cm^3/s). Differences in dissolution coefficients are partially explained by the fact that they use the dissolution coefficients that are dependent on surface area, as explained above, when using different specimen sizes. The discrepancy increases even more when including data reported using the same first-order kinetics but from other disciplines using different experimental methods or different specimen types. Even in individual studies, measuring the dissolution coefficient for different conditions, significant variations are common. For example, Aljubouri and Al-Kawaz (2007) used the same size specimens and test method to calculate the dissolution rate of gypsum under both still and flowing water with the results varying from 8.1×10^{-6} (cm/s) to 5×10^{-4} (cm/s). They also compared their results from tests conducted in still water (closed system) to those of Langmuir (1997), who reported a value of 2×10^{-4} (cm/s), and James and Lupton (1978), who reported a value of 1.4×10^{-4} (cm/s). Aljubouri and Al-Kawaz (2007) used the first order kinetics equation with normalized dissolution coefficient that are independent of the specimen's surface area (Eq. 2). Since the surface area of the specimens is known, normalized coefficients can then be recalculated so that they can be compared to dissolution coefficients derived from other first-order kinetics (Eq.1) where the dissolution coefficient is dependent on surface area. This recalculation results in coefficients ranging from 4.86×10^{-3} (cm^3/s) to 3.3×10^{-1} (cm^3/s), which compared to those reported by Klimchouk and Aksem (2005) and Ford and Williams (2007) illustrates how the results can vary for different conditions.

Testing gypsum from different sources (even different seams within a single deposit) can also produce very different dissolution coefficients. For example, Bolan et al. (1991) examined the dissolution rate of gypsum for the following four types of gypsum: (1) analytical grade gypsum, (2) flue-gas gypsum, (3) phosphor-gypsum (produced from phosphate ore), and (4) mined natural gypsum. Disk and powder specimens were prepared from each gypsum source. The dissolution tests for both disk and powder

specimens were conducted in the presence and absence of soil. They reported that the gypsum dissolution in the absence of soil (dissolution in “just water”) is a second order reaction, while in the presence of soil it is a first order reaction. For dissolution in the absence of soil, the second order coefficients for powdered specimens from different sources ranged from 120 to 700 ($\text{mmol}^{-1} \text{ liter}^{-1} \text{ min}^{-1}$), while for disk specimens the range was from 90 to 390 ($\text{mmol}^{-1} \text{ liter}^{-1} \text{ min}^{-1}$). For dissolution in the presence of soil, the concentration based dissolution coefficients for the powder specimens ranged from 900 to 2500 min^{-1} , while the range for the disk specimens was from 320 to 820 min^{-1} .

Recently, Castellanza et al. (2007), investigated the effect of flooding in abandoned underground gypsum mines on pillar stability. They initially developed a predictive model to estimate the time to pillar failure following mine flooding. While their initial model did not replicate the experimental data, they modified their approach to model the reduction in compressive strength of gypsum and anhydrite due to exposure to a wetting front. The modified model utilizes two constants α and β , where the constant, α , represents the rate of pillar weathering, which is due to gypsum dissolution, and the constant, β , represents the rate of pillar strength reduction due to pillar weathering. These constants were obtained through a trial and error process of fitting their modified model to the experimental data. Obtaining these two parameters (i.e. α and β) is somewhat arbitrary and estimating them requires conducting numerous experiments, especially as the value of the parameters depend on the flow rate, among other factors, and need to be calculated for flow rate.

Modelling gypsum dissolution rates, especially for mine pillar dissolution, is complex as indicated by the literature cited above. Further, James and Lupton (1978), in their research on foundation distress caused by gypsum and anhydrite dissolution, summarized the following four parameters that affect gypsum dissolution in underground settings: (1) temperature, (2) chemical composition of the flowing water, (3) the thickness of the diffusive boundary layer around the gypsum, and (4) the concentration gradient across the diffusive boundary layer. The diffusive boundary layer in turn depends on flow regime in that the higher velocity of steady state flow or a higher fluctuation intensity of unsteady flow, the thinner the diffusive boundary layer that leads to increased gypsum dissolution rates.

Since it is difficult to utilize the dissolution coefficients from the literature for specific gypsum deposits, it becomes necessary to determine the dissolution rates of gypsum when investigating the long-term stability of an abandoned gypsum mine, especially with groundwater moving through the mine. The purpose of this study is to present the findings of a simple experimental program with limited information (representing pillar dissolution in abandoned underground mines) to determine the dissolution coefficients of gypsum in an abandoned underground gypsum in the Michigan Basin.

4.3 Case study, materials and specimens

The research presented in this paper is part of an investigation into the long-term stability of the Domtar Mine (Figure 4-1), an abandoned underground gypsum mine located in Grand Rapids, MI. The main concern is that Interstate I-196 crosses over the northeast portion of the Domtar Mine for a length of approximately 640 meters (2100 feet). Existing mine maps show that the mining below the interstate occurred at a depth of approximately 27 to 30 meters (90 to 100 feet) where the gypsum seam thickness is approximately 3.5 meters (12 feet). Extensive subsidence and sinkholes have been occurring over the west and southern portions of the Domtar Mine (west of the interstate) due to roof and pillar collapse. The location of the mine underground working areas below I-196, as well as the location of the sinkholes, are shown in Figure 4-2.

Gypsum mining started in Grand Rapids, MI in the 1840's along the Grand River and continued until 2000 when the Domtar Mine was closed. Since 2000, the mine has been abandoned, equipment in the underground mine including dewatering pumps were removed and the portal decline backfilled with overburden materials. Observations of water in the main mine entry in 2003 indicated that the groundwater level has been mostly reestablished and that the mine workings completely flooded as can be seen in Figure 4-3 (Vitton 2004). The water is believed to be dissolving the gypsum pillars, reducing the radius (width) of the pillars while the weight of overburden on the pillars is constant. This means that the stress on the pillars is increasing. The dissolution of gypsum in water, however, will eventually stop if the water becomes saturated with respect to gypsum dissolution products, which is the case when the groundwater is stagnant. Groundwater at Domtar Mine, however, is flowing from northwest to southeast to the Grand River (see Figure 4-3) allowing dissolution products to be carried away by advection (with the water flow) preventing the groundwater from becoming saturated. This suggests that the rate at which the stress is increasing on the pillar (due to decreasing pillar's width) and eventually the time-to-failure (collapse) depends on the rate at which the gypsum is dissolving (weathered) in flowing water. To investigate the dissolution rate of gypsum under these conditions, dissolution tests were conducted on natural gypsum specimens. Since the Domtar mine is flooded, it was not possible to obtain test specimens from the mine, and therefore, test specimens were obtained from the National City Quarry located near Tawas City (also known as Tawas Quarry) on the east side of the state near Lake Huron as shown in Figure 4-1. According to Grimsley (1904), gypsum deposits in Grand Rapids area and Tawas City area are Mississippian age and part of the Michigan Formation (lower Grand Rapids series). Gypsum blocks (Figure 4-4a) were collected and transported to Michigan Technological University where they were cored (Figure 4-4b), cut and tested under stagnant and variable flow rates. Specimens were prepared in three different sizes (Figure 4-4c) which allowed us to study the effect of specimen size on the dissolution rate as well.

4.4 Experimental procedures

The gypsum blocks obtained from the National City quarry were cored to produce test specimens at the following three diameters (D): 2.8 cm (1.1 in), 5.1 cm (2 in) and 7.6 cm (3 in) and cut into lengths (H) of 5.8 cm (2.3 in), 10.7 cm (4.2 in) and 16 cm (6.3 in), respectively. To study the gypsum's dissolution rate, the cores had to be fully saturated. To prevent dissolution during the saturation process, the specimens were saturated using water previously saturated with gypsum as explained by Sadeghiamirshahidi and Vitton (2018).

To study the dissolution of gypsum under no flow conditions, four specimens (D=2.8 and H=5.8 cm) were submerged in stagnant water in four separate containers. The amount of dissolution and the dissolution rate depends on the volume of water in which gypsum core specimens are immersed. It was necessary, therefore, to maintain the ratio of water volume to core volume roughly proportion to the ratio of mine's water volume to pillars volume in the Domtar mine. While the older sections of the Domtar Mine have highly irregular pillars, especially those that were mined at the start of mining, the newer mine sections have a more standard square room and pillars. The rooms in the newer section are about 3m (10ft) high and about 9m (30ft) wide while the width of left in place pillars are about 6.1m (20ft), as shown schematically in Figure 4-5. When the mine is flooded, pillar dissolution becomes a function of the volume of water surrounding each pillar. In other words, the amount of dissolution and dissolution rate depends on the ratio of water volume (water volume in the tributary area around a pillar) to pillar volume in a mine as shown in Figure 4-5. The width of the tributary area in Domtar Mine is 15.1 m (Figure 4-5) and the room's height is 3 m, and therefore, the volume of water around each pillar is around 654 m^3 ($15.1 \text{ m} \times 15.1 \text{ m} \times 3 \text{ m} = 654 \text{ m}^3$). The approximate volume of each pillar is 111.6 m^3 ($6.1 \text{ m} \times 6.1 \text{ m} \times 3 \text{ m} = 111.6 \text{ m}^3$). Therefore, the ratio of the water volume to the pillar volume in Domtar Mine is 6.1 ($654 \text{ m}^3 / 111.6 \text{ m}^3 = 6.1$). Hence, in the “no flow” experiments, containers with inside diameters of 6.9 cm (Figure 4-6) were filled with water to the height of specimens (level of water in containers was the same as the height of the specimens which was 5.8 cm). Therefore, the volume of water in our “no flow” experiments was approximately 216.77 cm^3 ($\pi \times 6.9^2_{\text{cm}} / 4 \times 5.8 \text{ cm} = 216.77 \text{ cm}^3$) and volume of the specimens was 35.69 cm^3 ($\pi \times 2.8^2_{\text{cm}} / 4 \times 5.8 \text{ cm} = 35.69 \text{ cm}^3$), which provide approximately the same volume of water to gypsum ratio ($216.77 \text{ cm}^3 / 35.68 \text{ cm}^3 = 6.1$) as in the Domtar Mine. The specimens were submerged in stagnant water for 51 hours, and the *surface dried mass* of specimens were measured after 2.5 hours, 4.5 hours, 24 hours and at the end of the experiment (51 hours). The mass loss between consecutive surface dried mass measurements was divided by the time lapse between the two measurements to calculate the dissolution rate for that particular interval (stage). Throughout the experiments, the water temperature was also measured at different stages of each test to make sure that it was in the range of expected temperatures in the mine ($14 \pm 2 \text{ }^\circ\text{C}$). Surface dried masses were measured by removing the specimens from their containers, drying the specimen's surface using a paper towel and then placing them on a scale and monitoring the mass as it changed for a short time due to more water

being evaporated from the specimen's surface. When the specimen mass stopped changing, the surface dried mass was recorded. It should be noted, however, that after this measurement is recorded the specimen mass starts to decrease again, if left on the scale, as the specimen starts to lose internal water and care must be taken to record the surface dried mass of specimen the first time the change in the mass stops. After the surface dried masses were recorded, the specimens were placed back in their container, and the dissolution tests continued until the next measurement.

The next phase of the research was measuring the dissolution rate of gypsum in flowing water at different flow rates. For this phase, four specimens with diameter's of 2.8 cm (volume of each specimen was approximately 35 cm³ and the total volume of the four specimens was 140 cm³) were placed in a single permeater cell with an inside diameter of 11.5 cm (inside surface area of 104 cm²). To obtain the correct ratio of water to gypsum in the cell (which is roughly 6.1); multiple porous stones were placed under the specimens so that the height of water inside the cell is 8.2 cm (see Figure 4-7a and b). A better set up would have been to place the porous stones on both the top and bottom of the specimens. The height of each set of four specimens, however, were not equal and although the difference in heights was very small (less than 0.01 cm), it was enough to prevent the porous stones from lying flat on the specimens. Therefore, porous stones were only placed on the bottom of the specimens as shown in Figure 7. This allowed the water entering the cell to directly contact the gypsum and not have a gap between a porous stone and the gypsum specimen. The flow rate through the cell was controlled by opening and closing a valve at the bottom of the cell. Five different flow rates (0.4 (ml/s), 0.77 (ml/s), 2 (ml/s), 5.7 (ml/s) and 10.4 (ml/s)) were used and for each flow rate the dissolution continued for 48 hours. Similar to the stagnant water, surface dried mass of specimens was recorded multiple times during the tests (i.e., after 3 hours, 24 hours, and 48 hours of dissolution).

In the final experiment, which was to investigate the affects of specimen size on dissolution rate, four specimens were tested at a flow rate of 6 ml/s, which was close to the 5.7 ml/s used for smaller specimens. In these tests, two specimens with D = 5.1 cm and H = 10.7 cm (two cells shown on the right in Figure 4-7c) and two other specimens with D = 7.6 cm and H = 16 cm (two cells on the left in Figure 4-7c) were used. The desired water to gypsum ratio (equal to that of Domtar mine) was achieved by using separate cells for each specimen, and adjusting the number of porous stones placed under the specimens (see Figure 4-7c). Finally, the last set up (two specimens with D = 5.1 cm and H = 10.7 cm and two specimens with D= 7.6 cm and H= 16 cm) was used with a lower flow rate of 2 ml/s.

4.5 Results and discussions

Four specimens (D=2.8; H=5.8 cm) were used to measure the dissolution rate of gypsum in still water. Surface dried masses of specimens at different time intervals are summarized in Table 4-1. The tests were stopped after 51 hours, as the changes in the masses over the last 27 hours (from 24 to 51 hours of dissolution) was insignificant (less

than 0.046 percent), indicating that dissolution was complete. This can be seen in Figure 4-8 where the mass of specimens at different times during the dissolution test in stagnant water is presented. The figure indicates that the mass of specimens decreases relatively fast at the beginning of the test due to dissolution but slows down, and eventually the mass becomes essentially constant after 24 hours.

The dissolution rates for each stage (between the two consecutive surface dried mass measurements) were calculated using equation 5.

$$dm/dt = (SDM_{es} - SDM_{bs}) / t \quad (5)$$

where, SDM_{es} is surface dried mass of the specimen at the end of the stage, SDM_{bs} is surface dried mass of the specimen at the beginning of the stage, and t is the elapsed time between the two measurements (beginning and end of the stage). Four dissolution rates (one for each stage) were calculated for each specimen and results are presented in Table 4-2. The results indicate that the dissolution rates calculated for each stage are relatively consistent for all four specimens. In the first stage the dissolution rate is 0.046 (g/h) at the beginning of the test, but with time as the amount of dissolution product increases in the water, the dissolution rate gradually decreases and eventually dissolution stops. This change in dissolution rate by time can be seen in Figure 4-9 where the average dissolution rate of four specimens in each stage is plotted against time.

The concentration of gypsum dissolution products in water (C) at the end of each stage was also indirectly calculated by subtracting the specimen's mass at the end of that stage from the initial mass and dividing it by the volume of water in which the specimen was being dissolved. These results are tabulated in Table 4-3. As previously explained, we assumed that the dissolution had been completed after 24 hours (as the change in mass from 24 to 51 hours was insignificant), which means the concentrations at 51 hours, represent the concentration at saturation (C_s). Comparing our saturation concentrations with reported gypsum saturated water concentrations of around two grams per liter or 0.002 g/cm^3 (Bock 1961) indicates that the dissolution is also completed at about this time, i.e., 24 hours.

To investigate the kinetics governing the gypsum dissolution in stagnant water under these conditions, the $C_s - C$ for each stage (average of four specimens) was plotted against the dissolution rate calculated for that stage (Figure 4-10). The best fit to the data as shown in Figure 4-10, was found to be a linear trend line with coefficient of determination = 0.9895 indicating that the dissolution rate in stagnant water follows first-order kinetics (Eq. 1) with a dissolution coefficient (K) of $37.13 \text{ (cm}^3/\text{h)}$, which is equal to $1.0 \times 10^{-2} \text{ (cm}^3/\text{s)}$. As discussed, we could also use the other first-order kinetic reaction (Eq.2) to provide a normalized dissolution coefficient (k), which is not dependent on specimen size (surface area). Since the average surface area of our specimens is 6.38 cm^2 , the normalized dissolution coefficient (k) is $1.6 \times 10^{-3} \text{ cm/s}$. It is worth mentioning that this value is higher than the normalized coefficients reported by previous researchers, for stagnant water conditions, which are in the range of 1.4×10^{-4} to $5 \times 10^{-4} \text{ cm/s}$ (Aljubouri

and Al-Kawaz 2007; James and Kirkpatrick 1980; James and Lupton 1978; Langmuir 1997).

The dissolution tests for four specimens (D=2.8; H=5.8 cm) was also conducted under flowing water condition using five different flow rates. For each flow rate, the surface dried mass of specimens was measured three times during the test (after 3 hours, 24 hours, and 48 hours of dissolution) and each time the dissolution rate was calculated using equation 6 (total of three dissolution rates for each specimen).

$$dm/dt = (SDM_{cr} - SDM_{int}) / t \quad (6)$$

where, SDM_{cr} is surface dried mass of the specimen at time t , SDM_{int} is initial surface dried mass of the specimen at the beginning of the test, and t is the time lapse from the start of the test. Unlike the dissolution in stagnant water where the dissolution rates were different for different stages, the three dissolution rates calculated for each specimen in flowing water were close showing a steady dissolution condition. The dissolution rates measured for all four specimens were also relatively close. Therefore, the average dissolution rates of all four specimens were calculated for each flow rate (F) and plotted against the respective flow rate (Figure 4-11). As can be seen from the plot, the effect of flow rate on the gypsum dissolution rate can be modeled using a power law. It is worth mentioning that in this plot, the dissolution rate of gypsum in stagnant water ($F=0$) is also included. To include this point, we made two adjustments to the data. First, a very small flow rate ($F=0.01$ ml/s) was used instead of zero. This is because the best fit to the data (with highest R^2), is a power law as explained above, but it is not possible to have a flow rate of zero ($F=0$) when using the power law. The second problem was that unlike other dissolution rates, the dissolution of gypsum in stagnant water is not constant. As explained previously, it starts at higher rates and then gradually slows down until it eventually stops. Our experiments show, however, that the majority of dissolution (mass loss) happens in the first five hours, so the average dissolution rate for this period was used in the plot.

Instead of dissolution rates (dm/dt) used in Figure 4-11, the normalized dissolution coefficient (k) can also be plotted against flow rate (F) which would provide a similar power law but with a different constant as shown in Eq 7:

$$k = 0.0021 F^{0.1854} \quad (7)$$

where k is normalized dissolution coefficient (cm/s) and F is flow rate (ml/s).

Photos of the specimens at the end of dissolution at different flow rates are shown in Figure 4-12. As can be seen from this figure, the diameter of specimens is reduced from an initial diameter of around 28 mm. This reduction, however, is not uniform and most of the dissolution occurs at the two ends of the specimens. This differential dissolution can be explained by increased turbulence at both the top and bottom of the specimens which has also been discussed by James and Lupton (1978). At the top of specimens, the flow

directly encountering the top of the specimens causes the turbulence and increases the dissolution. Despite the laminar flow at the specimen's bottom (where the porous stones were placed), the flow on encountering the porous stones possibly generated some turbulence causing additional dissolution at the base of the specimens.

It is also worth noting that the use of porous stones in this testing does not necessarily represent the mine flow condition. In an underground mine, the pillars are connected to the mine's roof and floor and have no direct contact with the water (except for the pore water). In our laboratory set up, however, porous stones allow water to access the gypsum, which changes the flow condition at the ends of the specimens.

To investigate the effect of specimen size on dissolution rate, four larger specimens (two with $D = 5.1$ cm and two with $D = 7.6$ cm as shown in Figure 4-7) were also tested under 6 (ml/s) flow rate for 48 hours. As with the smaller specimens, surface dried mass of each specimen was measured three times during the test, and three dissolution rates calculated for each specimen. These three dissolution rates calculated for individual specimens were almost the same showing a steady dissolution reaction rate. Similarly, the dissolution rates calculated for the same sized specimens were also close. This experiment was repeated for three hours under a flow rate of 2 ml/s. The dissolution rates calculated for the same sized specimens under a flow rate of 2 ml/s were also close.

For both flow rates (2 and 6 ml/s), the average of dissolution rates (dm/dt) measured for specimens with the same size was calculated and plotted against respective specimen's surface area (Figure 4-13). In this plot, we also used the results of smaller specimens with flow rates close to those used on larger specimens (2 and 5.7 ml/s). As expected, increasing the surface area increases the dissolution rate because more gypsum is exposed to water at any given time. The normalized dissolution coefficients calculated for these specimens with different surface areas are shown in Figure 4-14. According to first order kinetics used in this research (Eq.2), k should be independent of the surface area of the specimens, while according to equation 7, k does change by changing the flow rate. Figure 4-14 does show the dependency of k to flow rate, and it shows that for each flow rate, k is relatively constant although with small differences for different specimen sizes. A possible reason that k is not more consistent for different sizes, is most likely the small variations in the flow rates in tests due to the manual controls used.

The values of k measured in our study, however, are higher compared to normalized coefficients reported by other researchers. A possible reason for these higher values is that natural gypsum specimens used in this study have some level of impurities. While calculating the concentrations and dissolution rates, the specimen's "total mass" loss of was used without an attempt to separate the mass loss due to gypsum dissolution itself and the mass loss due to detachment of impurities caused by gypsum dissolution. In other words, although the impurities in the specimens are not as soluble in water (at least not as soluble as gypsum) and would remain attached to the specimen to some degree (see Figure 4-15a and 15b), after enough gypsum is dissolved around them, they would suddenly become detached from the specimen in chunks (see Figure 4-15c and 15d),

increasing the mass loss. For example, Figure 4-15a and 15b show a gypsum specimen after 144 hours of dissolution. It can be seen in the photo how the gypsum has been dissolved around the insoluble impurities (gray material in the picture), but some of the impurities are still attached to the specimen. Figure 4-15c and 15d show some of the detached impurities on the porous stones at the end of the tests. The impurities losses, however, should not be excluded from dissolution rate calculations since impurity loss also contributes to decreasing pillar's radius by detachment from the pillars.

4.6 Conclusions

The dissolution of gypsum cores (cylindrical specimens) representing the pillars in an abandoned underground mine was experimentally investigated in this study. The results show that the dissolution of gypsum in the cylindrical specimens can be represented by the first order kinetic reaction shown in Eq. 2 by substituting the variable A with the surface area of the specimens. This kinetic equation renders the normalized coefficient independent of the surface area. Thus, dissolution rates derived from small specimens can provide a rough estimate for the dissolution rates for pillars. The normalized dissolution coefficient, however, does depend on the mine water's flow rate following the power law presented in Eq. 7. The normalized coefficient was also experimentally derived for stagnant water ($F=0$) with a result of 1.6×10^{-3} (cm/s) which is somewhat higher than the published data.

Large differences in reported gypsum dissolution coefficients are common in the literature, which is due to the complexity of the dissolution process. In other words, the dissolution of gypsum depends on many factors, hence the dissolution coefficients change by changing the test conditions and/or specimen sources. The tests in this study were designed to represent, as much as possible, the conditions encountered in a gypsum mine. A potential explanation for higher values of normalized dissolution coefficients obtained in this study might be that the dissolution rates were calculated from the total mass loss of specimens. This total mass loss includes the detachment of impurities in natural gypsum specimens caused by dissolution of gypsum around them. It can be argued that this should not be excluded from dissolution rates when studying long-term stability of underground mines since the detachment still contributes to the reduction of pillars' width and ultimately strength. Finally, the results showed that the reduction of the specimen's diameter is not uniformly distributed along the specimen's height.

Figure 4-1. Location of the abandoned Domtar Mine and the quarry from which the specimens were obtained. The picture on the top right is Google's aerial imagery of the quarry, and the picture on the bottom right shows the a working area (room and pillar) in Domtar Mine

Figure 4-2. Domtar Mine underground working areas in respect to interstate I-196 and some other streets in the area as well as the location of sinkholes that have occurred

Figure 4-3. A cross section of the study area showing the gypsum seams and the groundwater table

Figure 4-4. Natural Gypsum Specimens used in this study; a) Some of the gypsum blocks from Tawas Quarry, b) Coring machine used to core the specimens, and c) Three specimens with three different sizes

Figure 4-5. Room and pillar method used in Domtar Mine

Figure 4-6. Dissolution test in stagnant water

Figure 4-7. Dissolution test set up for flowing water condition

Table 4-1. Mass of specimens at different stages of dissolution test in stagnant water

Specimen ID	Surface Dried mass of Specimens (g)				
	Before Dissolution test started	After 2.5 Hours of Dissolution (Stage 1)	After 4.5 Hours of Dissolution (Stage 2)	After 24 Hours of Dissolution (Stage 3)	After 51 Hours of Dissolution (Stage 4)
# 9-1	64.85	64.74	64.69	64.56	64.55
# 9-3	67.32	67.20	67.14	67.02	66.99
# 9-5	65.16	65.05	64.98	64.83	64.81
# 9-7	66.08	65.96	65.89	65.72	65.70

Table 4-2. Gypsum Dissolution rate at different stages of tests in stagnant water

Specimen ID	Dissolution Rate (g/h)			
	<i>From start to 2.5 hours (Stage 1)</i>	<i>From 2.5 hours to 4.5 hours (Stage 2)</i>	<i>From 4.5 hours to 24 hours (Stage 3)</i>	<i>From 24 hours to 51 hours (Stage 4)</i>
# 9-1	0.044	0.025	0.007	0.0004
# 9-3	0.048	0.030	0.006	0.0011
# 9-5	0.044	0.035	0.008	0.0007
# 9-7	0.048	0.035	0.009	0.0007
Average	0.046	0.031	0.007	0.0007

Figure 4-8. Mass loss of specimens during dissolution test in stagnant water

Figure 4-9. Change in Dissolution rate of gypsum with time in stagnant water

Table 4-3. The concentration of gypsum dissolution products in water at different stages of the tests

Specimen ID	Concentration of gypsum dissolution products in water (g/cm ³)			
	After 2.5 hours	After 4.5 hours	After 24 hours	After 51 hours
# 9-1	0.0006	0.0009	0.0016	0.0016
# 9-3	0.0007	0.0010	0.0016	0.0018
# 9-5	0.0006	0.0010	0.0018	0.0019
# 9-7	0.0007	0.0010	0.0020	0.0021

Figure 4-10. Gypsum dissolution rate kinetic law derivation

Figure 4-11. Effect of flow Rate on gypsum dissolution rate (average of four specimens with $D=2.8$; $H=5.8$ cm)

Figure 4-12. Four specimens with initial diameters of about 28 mm after 195 hours of dissolution tests under different flow rates

Figure 4-13. Relation between dissolution rate and surface area of specimens

Figure 4-14. Normalized dissolution coefficients of specimens with different surface areas

Figure 4-15. a) A gypsum core after being dissolved in flowing water (at different flow rates) for 144 hours; b) a close up of the top of the specimen shown in (a); c) some of the impurities detached from a 7.6 cm diameter specimen at the end of dissolution test; d) some of the impurities detached from a 5.4 cm diameter specimen at the end of dissolution test

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Chapter 5

5 Long-Term stability and time to failure of a single pillar in a flooded abandoned gypsum mine⁴

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5.1 Abstract

Assessment of the long-term stability of abandoned underground mines is a difficult task especially when working areas are located under groundwater table where they will be flooded upon abandonment of the mine leading to the saturation of pillars. Saturation decreases the strength of pillars to some extent causing some stability problems. In evaporate deposits such as gypsum, further decrease in the strength of pillars occurs due to dissolution and reduced pillar width. The rate of strength reduction and the time to possible failure in these mines depends on the dissolution rate which is strongly affected by the rate of groundwater movement through the mine. In areas where groundwater is stagnant, the dissolution stops as the groundwater reaches its saturation potential with respect to dissolution products. In areas where groundwater continues to flow through the abandoned mine, however, dissolution will continue and can affect the long-term strength of the mine's support structures. In this paper, the rates of pillar's width and strength reduction due to gypsum dissolution were investigated experimentally, using core samples in the lab. A simple analytical model was developed to predict the change of specimen's radius by time due to the dissolution. A finite volume model was also developed in FLAC3D to model the strength reduction of specimens due to both saturation and dissolution. The model was then used to estimate the long-term stability of pillars in an abandoned gypsum mine located in Grand Rapids, Michigan that is located under a busy interstate highway and other transportation structures. Finally, using the dissolution models along with the results of the numerical simulations, the time to possible failure of a single pillar in the flooded abandoned mine was estimated under different flow rates.

Key words: *Abandoned gypsum mine; Groundwater flow rate; Dissolution rate; Log term stability, finite volume modelling, FLAC3D*

⁴ This chapter is a reprint of the paper "Long-Term stability and time to failure of a single pillar in a flooded abandoned gypsum mine" that will be submitted for publication as an original article in Rock Mechanics and Rock Engineering, 2019

5.2 Introduction

In room and pillar mining method, mine chambers are sometimes left behind at the end of mine's lifetime leaving the overburden resting only on pillars of untouched materials, without any protection against possible progressive weathering of pillars. The dewatering process, which is necessary during the mine's lifetime when mining below the groundwater table, would stop upon abandonment of mine allowing the groundwater to rise to its original level (Doulati Ardejani et al. 2013). This causes extensive weathering which decreases the strength of pillars (Auvray et al. 2008; Guha Roy et al. 2017; Kim and Changani 2016; Kim et al. 2017; Maheshwari et al. 2009; Poulsen et al. 2014; Vergara and Triantafyllidis 2016; Zhou et al. 2016b) and in some cases leads to pillar's collapse as reported by many researchers (El-Shayeb et al. 2001; Ferris et al. 1989). In the case of evaporitic rocks, particularly in abandoned anhydrite and gypsum mines, even more collapses have been reported (Auvray et al. 2004; Barla and Jarre 1991; James and Lupton 1978; Johnson 2005). This is because in abandoned anhydrite and gypsum mines, the water dissolves the gypsum and anhydrite pillars reducing their width (radius). The weight of overburden on that pillar is constant while the width is decreasing which means the stress on the pillar is increasing. It should be noted that, dissolution of gypsum in water eventually ceases when the water is stationary due to the fact that the water becomes saturated with respect to gypsum dissolution products. In case of the flowing water, however, the dissolution will continue until the gypsum is dissolved completely. This is because the dissolution products will be advected away by the water flow and the water in contact with the gypsum remains unsaturated. In abandoned flooded mines with flowing groundwater, the dissolution continues till the stress on the pillar, which is increasing as the pillar's width is decreasing, is close or equal to the bearing capacity (strength) of the pillar at which time the pillar will collapse (Castellanza et al. 2007). The time required for pillar width to reduce enough to cause failure is called time to failure which depends on the rate at which the gypsum is dissolved in water or the weathering of the gypsum pillars. The gypsum dissolution rate itself depends on different parameters among which the flow rate, temperature and chemistry of the flowing water are the most important ones. Many attempts have been made to understand and formulate the kinetics governing the dissolution rate of minerals especially gypsum (Barton and Wilde 1971; Bolan et al. 1991; Colombani 2008; Colombani and Bert 2007; Dreybrodt and Gabrovsek 2000; Fan and Teng 2007). To summarize the findings of these researches, the gypsum dissolution rate mostly depends on the thickness of the diffusive boundary layer around the gypsum objects (e.g. left in place gypsum pillars in abandoned mine) and the concentration gradient across the diffusive boundary layer. According to James and Lupton, 1978 (James and Lupton 1978) higher rates of steady flows and/or higher fluctuation intensities of unsteady flows lead to thinner diffusive boundary layer which in turn increases the gypsum's dissolution rate (Porter et al. 2000). In other words, the dissolution rate increases as the flow rate increases. As explained before, change in gypsum dissolution rate can change the time to failure of left in place pillars in abandoned mines. Despite the extensive research on dissolution rate and mechanical properties of gypsum, there is limited researches on the stability of abandoned underground gypsum mines (Castellanza et al. 2007; Castellanza et al. 2010; Doktan

1983; Hoxha et al. 2006; Wang et al. 2008). One of the most significant studies has been done recently by Castellanza et al. 2007 and 2010 (Castellanza et al. 2007; Castellanza et al. 2010) where they combined the weathering progression within gypsum pillars and strength decay of the pillars in order to predict the expected time to failure of those pillars. In their model, they introduced two constants: α ; rate of progression of the weathering front in the pillar and, β ; rate of strength reduction. The value of these two constants are obtained by fitting an analytical expression of a dimensionless load as a function of the dimensionless time to the experimental data. They then used their calculated α , and β values to predict the expected time to failure of pillars in an abandoned gypsum mine. Obtaining these parameters either requires conducting many experiments, which is neither a time nor a cost efficient method, or otherwise is very arbitrary. In the present work, an alternative simple analytical model (based on experimental work and dissolution kinetics) is presented to predict the change of pillar's radius by time due to the dissolution. The model only uses the gypsum dissolution rate in flowing water and geometry of the pillar. Further, the reduction of specimen's strength due to saturation and dissolution has been numerically modelled. The numerical model was then used to investigate the long-term stability of pillars in an abandoned underground gypsum mine in the state of Michigan, USA. Finally, time to failure of pillars under different possible flow rates was estimated using the developed analytical model and the results of the numerical models.

5.3 Site Description:

The Domtar Mine (previously known as Grand Rapids Gypsum Mine) is located at the west side of the Grand Rapids, MI, USA. The mine was initiated as a surface quarry in 1848, then in 1860's the underground mining started and continued (with couple of shut downs and reopenings) until 2000. Gypsum was mined in several sub-horizontal seams mostly in seam No. 2 (shown with an asterisk in Figure 5-1a) that is located approximately 30 m (100 ft.) beneath the Interstate I-196 (see Figure 5-1b). The mine is located below the ground water table and the water had to be pumped out of the working areas during the mine's lifetime. After closing the mine in 2000, all the dewatering processes were ceased and the mine workings were reported flooded in 2003 (Vitton 2004). Seam No. 2 and the bedrock beneath it are not flat and both dip towards the Grand River which causes the ground waters to flow along the seam (Vitton 2004). The flow of water prevents the water from saturation (with respect to gypsum dissolution products) which leads to continuous decreasing of pillars width (radius) due to gypsum dissolution. The average thickness of the seam No. 2 is 3.5 m (12 ft.), but during the mining process, about 0.3 m (1 ft.) was not mined in both floor and roof to minimize the contamination of gypsum product and prevent the weathering of top and bottom shale layers, so the mining height is around 3 m (10 ft.). The gypsum was mined with room and pillar method in which blocks of gypsum (pillars) are left in place to support the weight of overburden (all the rock and dirt above the mine workings). According to Vitton. 2004 (Vitton 2004), size and shape of the pillars vary a lot but the minimum diameter of pillars is 6 m (20 ft.). The size and shape of rooms also vary a little, but they are mostly rectangular tunnels

with the height of about 3 m (10 ft.) and width of around 6 – 9 m (20 -30 ft.). The thickness of bedrock (shale with thin layers of gypsum) above the seam No. 2 varies from zero (no roof rock) to 9 m (30 ft.) with average of 6 m (20 ft.) as shown in Figure 5-1a.

Figure 5-1b shows the location of the interstate with respect to the mining plan. Several subsidence and sinkholes have occurred over the years in the vicinity of the highway, which has raised some concerns about the stability of the mine, and the risks that it poses to the interstate. The locations of the sinkholes are also shown in Figure 5-1b.

Figure 5-1. a) Formations and soil/rock layers in the study area (after Vitton, 2004 (Vitton 2004)), b) location of underground mine in respect to interstate I-196 and recently developed sinkholes, c) Location of Domtar mine, Tawas Quarry, Michigan Formation and Interstate I-196 (Map was developed using ArcMap version 10.5.1 and the Data for developing the map was obtained from USGS (USGS))

5.4 Materials and Methods

5.4.1 Gypsum Rock samples

As explained before Domtar mine is currently flooded, limiting access to the mine for collecting samples for experimental studies. However, couple of active gypsum quarries across the state are mining the gypsum from the same formation (Michigan Formation) as Domtar mine (Grimsley 1904). One of these quarries is Tawas Quarry located between Tawas city and National City on the east side of the state as shown in Figure 5-1c. For our study, gypsum blocks were collected from Tawas quarry and transported to the Rock Mechanics Lab in Michigan Technological University where cores with three different sizes (i.e. diameters of approximately 28 mm, 54 mm and 75 mm with height to diameter ratios of roughly 2.1) were prepared and tested.

5.4.2 Experimental procedures

As explained by Vitton (2004) [28], it is certain that the groundwater in the abandoned Domtar mine flows from recharge areas in the highlands (north west side of the mine) towards the Grand River (east and south east side of the mine). The flow rate, however, changes at different times and from place to place across the mine and depends on many factors including drought or wet years, amount of precipitation, existence and size of solution channels (cavities in the bedrock caused by dissolution of soluble material like gypsum and limestone), frequency and distribution fractures and joints. For this reason, the dissolution rates under different flow rates need to be known. The dissolution rate of specimens with three different sizes under six different flow rates had already been studied by the authors and a correlation between flow rate and dissolution rate is reported (Sadeghiamirshahidi and Vitton Submitted 2018a). In the present study, more dissolution tests were conducted on 16 specimens with 28 mm diameter under three flow rates (four samples under 1.5 ml/s, four samples under 5 ml/s and four samples under 10 ml/s flow rate) and the change in the diameter of specimens (due to the dissolution) was monitored. While each specimen was exposed to the flowing water for total of 166.5 hours, the diameter of specimens were measured four times throughout the test (at the beginning of the test, after 47.5 hours, after 101 hours and at the end of the test). The amount of reduction in the diameter of a specimen is not the same all across the length (height) of the sample, so each time the diameter was measured at the top, middle and bottom of the specimen and the average was calculated. Additionally, four larger samples (two at 54 mm and two at 75 mm diameters) were tested under a flow rate of approximately 5.5 ml/s to study the sample size effect on the reduction rate in the specimen's radius. The dissolution test set up used in this study is shown Figure 5-2.

Figure 5-2. Dissolution test setup: a) four specimens with diameters of 28 mm under dissolution b) one specimen with diameter of 54 mm under dissolution c) one specimen with diameter of 75 mm under dissolution

Uniaxial Compression Strength (UCS) of dry and saturated specimens along with the effect of sample size and blasting on the UCS had also been studied by the authors and the results have been published (Sadeghiamirshahidi and Vitton submitted 2018b). In this study, the UCS of samples at the end of the dissolution test were also measured using a MTS rigid frame servo-hydraulic compression machine with a top bearing platens (it is the same machine that was used to conduct the UCS tests on dry and saturated samples).

5.4.3 Theoretical formulation and modelling of the rate of pillar's radius reduction due to gypsum dissolution

Kemper et al., 1975 (Kemper et al. 1975) showed that the gypsum dissolution rate in flowing water is proportional to the difference between the solution concentration at saturation (C_s) and the solution concentration (C) at the time it comes into contact with Gypsum (Eq. 1).

$$\frac{dm}{dt} = KA(C_s - C) \quad (1)$$

In Equation 1, (m) is the gypsum mass and (K) is the dissolution coefficient, which is a function of water flow rate, temperature and chemistry of flowing water (Kemper et al. 1975).

Experimental study of Sadeghiamirshahidi and Vitton, 2018 (Sadeghiamirshahidi and Vitton Submitted 2018a) confirmed that dissolution of gypsum pillars follows the kinetics represented by equation 1. Using the definition of density (Eq. 2) this dissolution rate can be re-written in terms of volume change instead of mass change (Eq. 3):

$$m = \rho \cdot V \quad (2)$$

$$\frac{dm}{dt} = \rho \frac{dV}{dt} \quad (3)$$

where ρ is the gypsum density and V is the volume of the gypsum exposed to water (pillar volume in case of abandoned mines).

Figure 5-3a, shows a cylindrical sample used in our dissolution tests after 195 hours of dissolution ((Sadeghiamirshahidi and Vitton Submitted 2018a)). As explained before and can be seen in Figure 5-3a, the dissolution reduces the diameter of sample but the amount of reduction is different along the length (height) of the sample. The differences, however, are not significant and assuming the same simple cylindrical geometry for the pillars (Figure 5-3b) the rate at which the volume of pillar changes can be calculated by the change of the radius (R) of the pillar due to the dissolution (the height of the pillar (h) remains constant in the mine) (Eq. 4 and 5):

Figure 5-3. Changing diameter of samples due to dissolution: a) Experimental results of a sample with initial diameters of about 28 mm after 195 hours of dissolution tests under different flow rates; b) Schematic geometry of the pillars used for the model

$$V = \pi R^2 h \quad (4)$$

$$\frac{dV}{dt} = \pi h \frac{d(R^2)}{dt} \quad (5)$$

$$A = 2\pi R h \quad (6)$$

Substituting Eq. 5 into Eq. 3, and then substituting Eq.3 and 6 in Eq. 1 yields:

$$\pi h \rho \frac{d(R^2)}{dt} = 2K\pi R h (C_s - C) \quad (7)$$

Using $d(R^2) = 2RdR$ in Eq. 7, rearranging and integrating of Equation 8 and solving for R yields:

$$R = \frac{K}{\rho} (C_s - C) t \quad (8)$$

In Equation 8, R is actually the amount of decrease in the radius of pillar due to dissolution of gypsum (see Figure 5-3b) which should be subtracted from the initial radius of the pillar (R_0) to get the remained radius (r) of the pillar at any time (t):

$$r = R_0 - R \quad (9)$$

We used equations 8 and 9 to predict the change in diameter of gypsum cores used in our dissolution tests.

5.4.4 Numerical modeling (Finite Element Model)

The explicit finite volume formulation of Modified Hoek-Brown (MHB) failure criterion (equation 1) in FLAC3D (Fast Lagrangian Analysis of Continua in 3 Dimensions) was used to numerically model the UCS tests conducted on dry, saturated and dissolved samples.

$$\sigma_1 = \sigma_3 + UCS \left[m_b \frac{\sigma_3}{UCS} + s \right]^a \quad (10)$$

Where σ_1 is the major effective principal stress at failure, σ_3 is the minor effective principal stress at failure, UCS is the uniaxial compressive strength of intact rock, m_b is a reduced value of Hoek's material constant (m_i), s is rock mass constant ($s = 1$ for intact rock) that depends on Geological Strength Index (GSI) and the degree of disturbance (D) caused by blast damage and stress relaxation, and a is another rock mass constant that depends on GSI (Hoek et al. 2002).

In each step of this MHB numerical model, the final set of principal stresses ($\sigma_{f1}, \sigma_{f2}, \sigma_{f3}$) are calculated using the following equations based on the set of principal stresses at the beginning of the step ($\sigma_{i1}, \sigma_{i2}, \sigma_{i3}$) and the set of principal strain increments ($\varepsilon_1, \varepsilon_2, \varepsilon_3$):

$$\sigma_{f1} = \sigma_{i1} - E_1 \varepsilon_1 - E_2 \varepsilon_3 \quad (11)$$

$$\sigma_{f2} = \sigma_{i2} - E_2 \varepsilon_1 - E_2 \varepsilon_3 \quad (12)$$

$$\sigma_{f3} = \sigma_{i3} - E_1 \varepsilon_3 - E_2 \varepsilon_1 \quad (13)$$

$$E_1 = K + 4G/3 \quad (14)$$

$$E_2 = K - 2G/3 \quad (15)$$

where K is the bulk modulus and G is the shear modulus. In FLAC3D, K and G can be directly used as the input to the model or instead, Young's modulus, E , and Poisson's ratio, ν , can be inserted and the software converts them to bulk and shear moduli using equations 16 and 17 (FLAC3D-Fast Lagrangian Analysis of Continua in Three-Dimensions user's guide-Fifth Edition. 2012):

$$K = \frac{E}{3(1 - 2\nu)} \quad (16)$$

$$G = \frac{E}{2(1 + \nu)} \quad (17)$$

In this study, the MHB model was used to first numerically model the UCS tests conducted on dry, saturated and dissolved gypsum cores in the lab. Uniaxial compressive strengths obtained from the experiments (in some cases with small changes) along with Hoek-Brown parameters for intact rock was used as input parameters of the model and the numerically predicted stress-strain curves were compared to the experimental curves. As a rock is gradually loaded in a UCS test, it goes through three stages (see Figure 5-4): 1) existing cracks preferentially aligned to the applied stress close, 2) starts with near linear elastic stress-strain behavior and as the stress increases initiating cracks start to propagate in a stable fashion, and 3) cracks begin to coalesce and propagate in an unstable fashion (Jaeger et al. 2007). Each of these stages have different moduli but, the MHB model, as can be seen from equations 11 to 17, assumes a constant elastic modulus for the rock. To address this problem, a lower elastic modulus was used in the first 900 steps in UCS of dry samples (300 steps for saturated samples and samples after dissolution) and 60 to 80 percent of secant modulus measured from experiments was used after 900 steps (300 steps for saturated samples and samples after dissolution). As the mechanical properties of saturated samples are usually used in design and long-term stability analyses of abandoned mine pillars, stress-strain curves of samples after dissolution was also modeled using the UCS and elastic modulus of saturated samples but with reduced GSI and Hoek-Brown's s-parameter to understand the effect of dissolution on the strength of pillars. Parameters used in these models are summarized in Table 1.

The developed model was then used to first study the stability of a single pillar in the Domtar mine under dry (representing before abandonment) and saturated (representing short term stability after abandonment) conditions. In these models, a simplified version of the cross section presented in Figure 5-1 was used to develop the pillar and its tributary area in FLAC3D as shown in Figure 5-5. Finally, appropriate input parameters, obtained from modeling the dry and saturated pillar along with data from modeling the UCS of dissolved specimens using the saturated mechanical properties, was used to study the effect of dissolution on the long term stability of pillars in Domtar mine (effect of dissolution on the pillar stability). This was achieved by gradually reducing the diameter of the pillar in the model while keeping the height of the pillar constant. In modelling the pillar, modified Hoek-Brown criteria was used for Gypsum and Shale layers but Mohr-Coulomb failure criteria was used for the overburden. The average dry and saturated UCS and young moduli obtained from our previous experimental study (Sadeghiamirshahidi and Vitton submitted 2018b) were used in these models. The Hoek-Brown parameters used in the models are summarized in Table 5-2.

Figure 5-4. Three stages a rock goes through before yield (failure)

Table 5-1. Data used in the Modified Hoek-Brown model in FLAC3D for modeling the UCS tests

Sample		Experimental Data				Data used in Modified Hoek-Brown model in FLAC3D						
		Initial Diameter (mm)	Diameter After Dissolution (mm)	UCS (MPa)	Secant Modulus (GPa)	GSI	s	a	mb	Young Modulus (bar)		UCS (bar)
										First 900 Steps	After 900 Steps	
Dry Sample	DRY-28	28	-	25	1.57	100	1	0.5	8	4000	15000	240
	DRY-54	54	-	16	1.74	100	1	0.5	8	4000	16000	160
	DRY-75	75	-	14	2	100	1	0.5	8	4000	17000	139
Saturated samples	SAT-28	28	-	16	1.61	100	1	0.5	8	4000	10000	157
	SAT-54	54	-	10	0.9	100	1	0.5	8	2000	8000	100
	SAT-75	75	-	8	1.78	100	1	0.5	8	2000	12000	73
Samples after 166.5 hours of dissolution	DISS-28	28	24.84	7	0.9	100	1	0.5	8	4000	5000	70
	DISS-54	54	51.84	5	1	100	1	0.5	8	2000	6000	46
	DISS-75	75	73.48	3	0.6	100	1	0.5	8	1000	3700	33
						Using data from saturated samples to predict the UCS of samples after dissolution:						
	DISS-28	28	24.84	16	1.61	85	0.2	0.5	5	4000	10000	157
	DISS-54	54	51.84	10	0.9	85	0.2	0.5	5	2000	8000	100
	DISS-75	75	73.48	8	1.78	85	0.2	0.5	5	2000	12000	73

Figure 5-5. Geometry of the pillar and the tributary area used in FLAC3D modeling

Table 5-2. Modified Hoek-Brown parameters used in FLAC3D modeling of pillar under different conditions

Condition of Pillar	Layer	Modified Hoek-Brown Model Parameters					
		UCS (MPa)	Young Modulus (GPa)	GSI	s	a	mb
Dry	Gypsum Layers	19	1.4	80	0.11	0.5	3.91
	Shale Layers	30	14.0	80	0.11	0.5	2.93
Saturated	Gypsum Layers	11	1.4	80	0.11	0.5	3.91
	Shale Layers	23	12.0	80	0.11	0.5	2.93
Dissolving Pillar	Gypsum Layers	11	1.4	80	0.11	0.5	3.91
	Gypsum Pillar	11	1.4	65	0.02	0.5	2.2
	Shale Layers	23	12.0	80	0.11	0.5	2.93

5.5 Results

5.5.1 Results of dissolution tests and change in the diameter

The diameter of specimens measured at different times during the dissolution tests are summarized in Table 5-3. Total of 16 samples were tested, 12 with initial diameter of around 2.8 cm, two with initial diameter of around 5.4 cm and two with initial diameter of around 7.5 cm. Four of samples with diameters around 2.8 cm were tested under water flow rate of 1.66 ml/s, four under 9.94 ml/s and the last four under 10.3 ml/s. The average diameter of each set of four samples tested under the same flow rate is also calculated each time and presented in the table (Bold numbers). As an example, samples #4 to #8 at different stages of dissolution tests under flow rate of 4.94 ml/s are shown in Figure 5-6. The average diameter of two samples with 5.4 cm diameter as well as average diameter of two samples with 7.5 cm diameter at each stage of the tests is also presented in the table. The analytical model suggested by equations 8 and 9 were used to predict these average diameters (bold numbers in Table 5-3) at different times during the dissolution tests and the results are shown in Figure 5-7. As it can be seen from the figure, the model is able to predict the average diameter of samples at different times while they are being dissolved in water.

Table 5-3. Average of three diameter readings (from the top, middle and bottom of each specimen) at different times during the dissolution tests

Sample	Flow Rate (ml/s)	Average of 3 Diameter readings (top, middle and bottom of specimens) (mm)			
		Initial	After 47.5 hrs. Dissolution	After 101 hrs. Dissolution	After 166.5 hrs. Dissolution
Specimen #1	1.66	28.26	27.48	26.37	24.18
Specimen #2		28.30	27.56	26.60	25.05
Specimen #3		28.29	27.39	25.96	24.94
Specimen #4		28.30	27.61	26.04	24.84
Average		28.29	27.51	26.24	24.75
Specimen #5	4.94	28.25	26.52	24.70	21.82
Specimen #6		28.20	26.50	24.29	22.40
Specimen #7		28.29	27.12	25.09	23.43
Specimen #8		28.27	26.74	25.24	23.36
Average		28.25	26.72	24.83	22.75
Specimen #9	10.3	28.25	26.76	24.72	21.52
Specimen #10		28.23	26.52	24.45	21.48
Specimen #11		28.44	25.67	23.17	21.10
Specimen #12		28.27	26.87	23.91	20.63
Average		28.30	26.46	24.06	21.18
Specimen #13	5.7	54.08	53.19	52.10	50.87
Specimen #14		54.00	52.88	52.15	51.82
Average		54.04	53.04	52.12	51.34
Specimen #15	5.6	75.81	75.16	74.32	73.67
Specimen #16		75.65	75.11	74.50	73.48
Average		75.73	75.14	74.41	73.57

Figure 5-6. Samples #4 to #8 (initial diameters of approximately 28 mm) at different stages of dissolution tests

Figure 5-7. Average diameter of samples at different times during the dissolution tests along with the predicted diameters using equations 8 and 9 (assuming $C=0$ for all samples)

5.5.2 Results of UCS tests and the numerical modeling of stress-strain curves

Results of the UCS tests on dry, saturated (from our previous study (Sadeghiamirshahidi and Vitton submitted 2018b)) and dissolved samples (from the current study) are presented in Figure 5-8. As it can be seen different sample sizes were used in UCS tests. It is worth mentioning that a correlation between sample size and UCS of dry and saturated samples has been developed (Sadeghiamirshahidi and Vitton submitted 2018b) using UCS of samples with diameter of 54 mm [$UCS_{(54)}$] as the reference (standard). The correlation was then used to convert all the UCS values of different sized samples to standard values [$UCS_{(54)}$]. The average $UCS_{(54)}$ of dry samples was found to be 19 MPa while for saturated samples it was 11 MPa (see (Sadeghiamirshahidi and Vitton submitted 2018b) for more details).

The UCS tests of dry and saturated samples with different sizes was modeled in this study using FLAC3D. As an example, the results of a UCS test on a dry sample and the numerical modeling of stress-strain curve of the same test is presented in Figure 5-9. The elastic-plastic state of the model along a vertical cross section through the center of the sample is also shown. As can be seen in the figure, before yielding (the peak in stress-strain curve), the entire sample in the model is in elastic state (state: "None" means no plastic strain has occurred, Figure 5-9a), but after the peak stress (as shown in Figure

5-9b) the sample is under shear strain (yield). These results shows that not only the model is capable of modelling the stress-strain curve of the UCS tests but also the elastic-plastic state of the sample can be correctly predicted and used to determine whether the yield (failure) has happened in a material. The experimental and numerically predicted stress-strain curves of dry, saturated and dissolved samples with different sizes are demonstrated in figures 10 to 12, respectively. As explained before, UCS of dissolved samples were modeled in FLAC3D using two methods. In the first method, the data (ultimate strength and young modulus) from the actual tests on the dissolved samples were used as the input to the FLAC3D model. In the second method, the strength parameters of saturated samples were used in the FLAC3D model but the GSI was reduced by 15 points. The results of both methods are shown in Figure 5-12.

Figure 5-8. Results of UCS tests on dry, saturated and dissolved samples

Figure 5-9. The results of UCS test on a dry sample with diameter of 75 mm and the numerical modelling of the same test

Figure 5-10. Experimental and modeled stress-strain curves of dry samples with different sizes

Figure 5-11. Experimental and modeled stress-strain curves of saturated samples with different sizes

Figure 5-12. Experimental and modeled stress-strain curves of dissolved samples with different sizes. Two methods were used to model the dissolved samples, first the data

from the actual tests on the dissolved samples used as the input to the FLAC3D model (FLAC3D using Experimental UCS) and second the strength parameters of saturated samples were used in the FLAC3D model but the GSI was reduced by 15 points (FLAC3D using Saturated UCS)

5.5.3 Results numerical modeling of a single pillar stability

Stability of a single pillar in Domtar mine under dry and saturated conditions as well as the stability after the dissolution has been numerically modelled and the results are summarized here. The distribution of vertical stresses, vertical displacements and the elastic-plastic state (as an indicator of stable or unstable) of the dry pillar and its tributary area along a vertical cross section through the center of the pillar are shown in Figure 5-13. As it can be seen from Figure 5-13a, after excavation most of the stress is transferred to the pillar but majority of the displacements, although not very large (mostly 1 to 3 cm with the maximum of 6 cm), occur in the roof (Figure 5-13b). Figure 5-13c shows that some areas including the pillar and the roof, go through shear and/or tensile yielding but the stresses would redistribute after the yielding occurs and those areas become stable again after redistribution of stresses. These areas are shown with shear-p (indicating shear yielding in the past and stable after the redistribution of stresses) or tension-p (indicating tensile yielding in the past and stable after the redistribution of stresses) in the figure.

Figure 5-13. Results of numerical modeling of a single dry pillar in Domtar Mine: a) Distribution of vertical stresses (in Pascal) after excavation, b) Vertical displacement (in meters) after excavation and C) Final elastic-plastic state of the pillar

The elastic-plastic state of the saturated pillar was the same as that of the dry pillar, but the roof experienced larger vertical displacements (mostly 3 to 10 cm with a maximum of 15 cm) as shown in Figure 5-14.

Effect of dissolution on the stability was investigated by changing the GSI of the gypsum in the pillar from 80 to 65 and reducing the diameter of pillar. The results showed that the

effect of dissolution (and the consequent reduction of pillar diameter) starts to appear when the diameter of pillar reaches 4.5 m (from initial 6 m). At this diameter, the roof starts to fail under tension (Figure 5-15a), and if the dissolution continues after this point, shear failure starts to develop in the pillar at diameter of 4 m (Figure 5-15b) and the pillar would completely fail under shear when the diameter reaches 3 m (Figure 5-15c).

Figure 5-14. Vertical displacement in the saturated pillar

5.5.4 Results of time to failure of individual pillars

Using 3 meters as the diameter at which the pillars would fail, as suggested by the results of numerical modeling of a pillar in previous section, along with equations 8 and 9, the time to failure of a single pillar subjected to different flow rates (and subsequently different dissolution rates) was calculated and the results are shown in Figure 5-16. As it can be seen from the figure, depending on the flow rate, the time to failure of a single

pillar could change from 60 years for small flow rates to about 20 years for larger flow rates.

Figure 5-15. Elastic-plastic state of the model at different stages: a) when the diameter of the pillar is 4.5 meters, b) the diameter of the pillar is 4 meters, c) the diameter of the pillar is 3 meters

Figure 5-16. Time to failure of a single pillar under different flow rates (Asterisks show the failure time calculated using equations 8 and 9 assuming $C = 0$ and the failure diameter = 3 m)

5.6 Discussion

The ultimate purpose of the present work is to predict the time to collapse of left in place gypsum pillars in an abandoned mine at the state of Michigan in United States. Usually, during the lifetime of a mine, the groundwater is constantly pumped out of the working areas. After the mine lifetime, however, the groundwater usually is allowed to recover to its previous level (flooding the mine workings). Despite the reduction in the strength of pillars after saturation, this flooding is not usually considered a huge risk especially when proper designs and larger safety factors have been used for the mine. Even in evaporitic mines such as gypsum mines, with the possibility of dissolution of the supporting left in place pillars, it is not considered very concerning based on the assumption that the dissolution of pillars (and resulting strength lost) will cease after the water around the pillars get saturated with respect to dissolution products. It is not a very bad assumption in the case of stagnant water. In case of flowing groundwater, however, the dissolution will continue till the radius (width) of the supporting pillars becomes so small that they cannot bear the weight of overburdens anymore and eventually collapse. In the case of gypsum pillars for example, the rate at which the radius of the pillar decreases depends on the rate at which the gypsum dissolves in flowing water. The dissolution rate of gypsum itself depends on the water flow rate, temperature, chemistry of water passing around the pillars. In this study, we developed a simple analytical model based on the first order dissolution kinetics to predict the change in the radius of gypsum core samples due to dissolution in the lab. The model successfully predicted the radius of small specimens at different times during the dissolution tests. For larger specimens, however, the model slightly overestimated the reduction in the specimens' radii. This is possibly due to the assumption that the solution concentration (C) is zero in flowing water. While this assumption works for smaller samples, the dissolution products from large samples are much higher and the water cannot completely carry them away. This increases the C in the water around the samples and reduces the dissolution rate. Gypsum product saturation concentration (C_s) is 2.5 g/cm^3 . Assuming $C = 0.4 \text{ (g/cm}^3\text{)}$ for samples with 54 mm diameter and $C = 0.7 \text{ (g/cm}^3\text{)}$ for samples with 75 mm diameter, the predicted diameters during dissolution tests improves significantly as can be seen in Figure 5-17.

We also conducted UCS tests on gypsum cores after being subjected to dissolution tests under different flow rates. A numerical model based on the modified Hoek-Brown failure criterion was then developed using FLAC3D to simulate the experimental stress-strain curves as well as elastic-plastic state of the samples. In order to develop this model, the results of UCS on dry and saturated specimens from a previous study conducted by the authors (Sadeghiamirshahidi and Vitton submitted 2018b) were used to calibrate the model. Both dry and saturated samples were assumed to be intact rock samples ($\text{GSI} = 100$) and the ultimate strengths and secant moduli measured from the experiments were used as the main input parameters of the model. The model was then used to simulate the stress-strain curves and elastic-plastic state of the dissolved samples. The results showed that dissolution affects the ultimate strength of samples by two means: first by reducing the diameter of samples, and second, by reducing the geological strength index of the samples (from 100 to 85). In other words, in addition to reduced diameter, dissolution

also changes the mechanical properties of samples, probably by altering the structure of the rock and/or condition of discontinuity surfaces. The results also showed that, the behavior of rock samples in the laboratory cannot be simulated by a single elasticity modulus due to large displacements at the beginning of the tests which is caused by closure of existing cracks preferentially aligned to the applied stresses. Therefore, in FLAC3D model, a very low elasticity modulus (approximately 10 to 20 % of secant modulus) should be used at first and after a series of steps the elasticity modulus should be increased to 60 to 80 percent of secant modulus. It is worth mentioning that, failing to use the low elasticity modulus at the beginning of the simulation does not affect the ultimate strength predicted by the model. It only causes the model to underestimate the strain and the displacement at failure of the samples. In other words, the model changes from elastic to plastic state under the same stress whether or not the lower elasticity modulus is used at the beginning of the model. After the model successfully simulated the lab tests, the model was used to model the long-term stability of the pillars in the Domtar Mine. The results shows that the roof in the abandoned mine starts to fail under tension when the diameter of pillar reaches 4.5 m due to dissolution, and at diameter of 4 m shear failure starts to develop in the pillar itself and the pillar will completely fail under shear at the diameter of 3 m. Using 3 meters as the failure diameter in combination with equations 8 and 9, the time to failure for a single pillar was estimated for different flow rates. According to these calculations a single pillar would fail between 20 to 60 years depending on the flow rate. The ground water flow in abandoned mines are usually very slow so the failure time of a single pillar would be closer to 40 to 50 years. This is again based on the assumption that solution concentration (C) is zero around the pillars. As discussed for the results of experiments for larger samples, this assumption for pillars would probably lead to overestimating the reduction of pillar's radius due to dissolution. In other words, it takes longer for pillars to reach the critical diameter (3 m) than what is predicted by our model. For example, the prediction failure time for a single pillar assuming $C = 1.5 \text{ g/cm}^3$ was also conducted and the results are shown in Figure 5-18. It shows that in this case, time of a single pillar failure increases to around 110 to 120 years. This model, however could be used as a guideline when preparing for the worst-case scenario. The model could also be improved with more experimental and field tests. It is also worth noting that, the models provided in this paper are based on the minimum diameter of pillars in the mine (6 meter). As explained before, size and shape of the rooms and pillars vary a lot in the mine meaning a lot of pillars have larger diameters in the mine that would take much longer to fail. Also, a single pillar failure does not lead to the collapse of the whole mine. In other words, it does not mean that the mine necessarily collapse after 40 to 50 years (or 110 to 120 years assuming $C = 1.5 \text{ g/cm}^3$), but stability problems could increase significantly at that time.

5.7 Conclusion

Flooding the mine working areas with groundwater after the mine life in evaporite mines like gypsum was thought to be nonhazardous due to the assumption that the dissolution of gypsum pillars will cease after the saturation of surrounding water. This is a reasonable

assumption in the case of stagnant water but for those cases with flowing water, the dissolution will continue until radius of the pillars becomes small enough to cause the pillars to collapse. The rate of decrease in radius of the pillars depends on gypsum dissolution rate which itself depends on the water flow rate, temperature, chemistry of water passing around the pillars. A very simple model was presented in this paper to predict the change in the radius of left in place gypsum pillars in abandoned mines. A finite volume model was also developed to predict the long-term stability of the pillars subjected to dissolution in flooded abandoned mines. The model was then used to analyze the long-term stability of pillars in Domtar gypsum mine, MI, USA. The results showed that dissolution of gypsum not only reduces the diameter of the pillar, but also reduces the ultimate strength of gypsum rock. The results also showed that when gypsum dissolution reduces the diameter of pillar to about 4.5 m, the roof in the abandoned mine starts to experience tensile yielding. If the dissolution continues and the diameter is reduced to 4 m, shear failure starts to occur in the pillar itself and eventually at the diameter of 3 m the pillar will completely fail. The results further showed that, assuming zero concentration of gypsum dissolution products in groundwater at all times (this assumption tends to overestimate the rate at which the width of mine pillars are reduced), pillars with minimum width in the mine (6 m) start to fail approximately after 40 to 50 years of being exposed to flowing water. This time increases to 110 to 120 years assuming 1.5 g/cm^3 as concentration of dissolution products (C) in groundwater. Although, this does not mean that the mine would collapse about 50 (or 120 years assuming $C = 1.5 \text{ g/cm}^3$) years after abandonment, the stability problems could increase around that time.

Figure 5-17. Average diameter of samples at different times during the dissolution tests along with the predicted diameters using equations 8 and 9 (assuming $C=0$ Assuming for samples with 28 mm diameter, $C = 0.4 \text{ (g/cm}^3\text{)}$ for samples with 54 mm diameter and $C = 0.7 \text{ (g/cm}^3\text{)}$ for samples with 75 mm diameter)

Figure 5-18. Time to failure of a single pillar under different flow rates assuming $C = 0$ and $C = 1.5 \text{ g/cm}^3$ and the failure diameter = 3 m)

5.8 References

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Chapter 6

6 Conclusions

In this research, long term stability of pillars in Domtar Mine, an abandoned underground gypsum mine in Grand Rapids, MI was investigated. The main findings of the research are summarized below:

1. The research required the drying and saturating of the gypsum test samples for mechanical testing. Heating gypsum to prepare dried samples can dehydrate the gypsum and change its chemical structure. Gypsum is a calcium sulfate dihydrate with two molecules of water in its structure ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$). Heating the gypsum causes it to lose one and half molecule of water, converting it to hemihydrate ($\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$). If heating continues at higher temperatures, the remaining half molecule of water will be removed, converting the hemihydrate to anhydrite (CaSO_4). The temperature at which gypsum transforms to hemihydrate and anhydrite, however, depends on a number of parameters such as the gypsum's pore water pressure, vapor pressure (for unsaturated conditions), chemicals in the pore water as well as whether the gypsum is natural or synthetic. Further as the transformation process continues with higher temperatures the particle density increases. A helium pycnometer was used to determine the temperature at which the chemical transformation occurs using density as a proxy. The research showed that the gypsum-hemihydrate transition temperature under a moisture content measurement condition is above 80 °C. ASTM Standard D2216, however, limits the drying temperature to 60 °C. Heating the gypsum sample at 80 °C even for 72 hours did not transform gypsum to hemihydrate, indicating that water content of gypsum samples can be measured using the oven drying method at temperatures up to 80 °C. The results also showed that microwave drying cannot be used to measure the water content of gypsum, although the transition from gypsum to hemihydrate does not appear to be significant with microwave heating.
2. Prior research shows that a fully saturated gypsum can lose upwards to 40% of its strength compared to a dry gypsum. Therefore, mechanical testing was conducted on both dry and saturated gypsum specimens. Saturating gypsum in freshwater, however, results in gypsum dissolution. At least three methods have been used by researchers for saturating rock samples. The three methods are (1) simple saturation (water immersion), (2) vacuum saturation, and (3) improved vacuum saturation. Results of the research confirmed that the three saturation methods can be used for saturation gypsum. However, the water must first be saturated with gypsum before it can be used to saturate the gypsum core samples, thus minimizing gypsum dissolution. Improved vacuum saturation was found to saturate gypsum within 24 hours but required additional sample preparation time to place gypsum under vacuum before saturation (leading to a total time of 48 h for saturation). On the other hand, 30 hours is required to saturate the gypsum specimens using vacuum saturation or saturation without vacuum methods.

3. Gypsum specimens were tested in both dry and saturated conditions. For the gypsum samples from the Michigan Basin, the average uniaxial compression strength, $UCS_{(54)}$, of gypsum was 19 MPa for dry samples and 11 MPa for saturated gypsum indicating a 41% reduction due to the saturation. While these results are for blasted rock, the $UCS_{(54)}$ of intact rock with minimum flaws can be as high as 29 MPa for intact dry rock and 18 MPa for intact saturated rock. The results also showed that elastic moduli of samples decreased from 30 to 50 percent due to saturation.
4. The point load index of gypsum was also measured in this study and compared to the uniaxial compressive strength. The point load index to uniaxial compressive strength conversion factor, K , for dry samples was 6.6 and 7.7 for saturated samples were measured for gypsum from Michigan Basin.
5. The gypsum dissolution of cylindrical specimens, representing the pillars in an abandoned underground mine, was experimentally investigated. The results show that the dissolution of gypsum in the cylindrical specimens can be represented with the first order kinetic reaction ($dm/dt = kA(C_s - C)$) by substituting the variable A with the surface area of the specimens. This kinetic equation renders the normalized coefficient (k) independent of the surface area. Thus, dissolution rates derived from small specimens can provide a rough estimate for the dissolution rates for pillars. The normalized dissolution coefficient, however, does depend on the mine water's flow rate following the power law ($k = 0.0021 F^{0.1854}$). The normalized coefficient was experimentally measured for stagnant water ($F=0$) to be 1.6×10^{-3} (cm/s), which is somewhat higher than the published data.
6. Review of the dissolution rates in the literature indicate that there is a large difference between studies. This suggests that the dissolution of gypsum depends on many factors, e.g., changing the test conditions and/or specimen sources. The tests in this study were designed to represent, as much as possible, the conditions encountered in a flooded underground gypsum mine. The dissolution rates determined in this research, however, were generally higher than those reported in the literature. An explanation for the higher values of normalized dissolution coefficients obtained in this study might be due to the dissolution rates being calculated from the total mass loss of the specimens. This total mass loss includes non-gypsum particle (impurities) detached during dissolution of the gypsum. It can be argued that the loss of impurities should be included in dissolution rates when studying long-term stability of underground mines since the detachment still contributes to the reduction of pillar's diameter and ultimately strength. Finally, the results showed that the reduction of the specimen's diameter is not uniformly distributed along the specimen's height.

7. The following model was developed in this research to predict the change in the radius of the gypsum pillars in abandoned mines [$R = \frac{k}{\rho} (C_s - C) t$], where, R is the decrease in the radius of pillar due to dissolution of gypsum, k is normalized dissolution coefficient, ρ is the gypsum density, t is time, C_s is the solution concentration at saturation and C , is the solution concentration at the time it comes into contact with gypsum. Using this equation, the pillar radius (r) of the pillar at any time (t) can be calculated ($r = R_0 - R$), where R_0 is initial radius of the pillar before dissolution.

8. A finite volume model was also developed to predict the long-term stability of the pillars subjected to dissolution in flooded abandoned mines. The model was used to analyze the long-term stability of pillars in Domtar Gypsum Mine. The results showed that when gypsum dissolution reduces the diameter of pillar to about 4.5 m, the roof in the abandoned mine starts to experience tensile yielding. If the dissolution continues and the diameter is reduced to 4 m, shear failure starts to occur in the pillar itself and eventually at a diameter of 3 m the pillar will completely fail. The results also showed that, assuming zero concentration of gypsum dissolution products in groundwater at all times (this assumption tends to overestimate the rate at which the width of mine pillars are reduced), pillars with minimum width in the mine (6 m) start to fail approximately after 40 to 50 years of being exposed to flowing water. This time increases to 110 to 120 years assuming 1 g/cm³ as concentration of dissolution products (C) in groundwater. Although, this does predict that the mine would collapse in about 50 (or 120 years assuming $C = 1$ g/cm³) years after abandonment, the stability problems could increase around that time.

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