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Abstract

Experiments were performed with Ibeco backfill (Milos, Greece) and MX-80 (Wyoming, USA) bentonite and in selected cases sulphide solutions. The main target was to determine the solubility of the sulphides in the bentonites and their equilibrium concentrations. The concentration was determined to be lower than the limit of quantification of the methylene blue method, however, oxidation and/or H_2S loss, may have caused a lowering of the sulphide concentration in the samples, thus introducing an uncertainty. Sulphide was also added in some samples, and when sulphide was added as Na_2S solution, the results indicated that the bentonite reduced the amount of sulphide in the solution. The mechanism of this can be absorption or some kind of transformation or reaction with the sulphide, but these details are currently unknown. The observation that bentonite reduces the amount of sulphide in solution when Na_2S is added supports that no sulphide could be detected when Na_2S was not added. Two different methods were used for sulphide detection, the methylene blue method and the copper sulphate method (precipitating sulphide as CuS). The final experiments were done in a glove box with < 1 ppm O_2. It was found that it was critical that all bentonite (montmorillonite) was removed from the solution before the reagents were added for the sulphide determination due to interactions between the reagents and the bentonite.

Based on the data, a very rough estimation was done indicating that 1–10 kg of sulphide seemed to be removed per 1 000 kg of bentonite. This can be compared with that 0.5 wt% or 50 kg of sulphide in minerals is currently allowed per 1 000 kg of bentonite buffer.

Sammanfattning

Experiment utfördes med Ibeco BF (Milos, Grekland) och MX-80 (Wyoming, USA) bentonit samt i vissa fall sulfidlösningar. Huvudsyftet med studien var att bestämma jämviktskoncentrationen av sulfid i vatten-bentonit blandningar. Vid spektrofotometrisk bestämning av löst sulfid i vattenlösningarna erhölls koncentrationer som var lägre än metodens rapporteringsgräns (limit of quantification), men det fanns problem med oxidation eller annan förlust av sulfid i experimentet som delvis kan ha påverkat resultatet. När natriumsulfidlösning tillsattes, upptäcktes det att bentoniten minskade mängden sulfid i lösning till en koncentration som var lägre än den förväntade. Mekanismen för detta kan vara absorption, omvandling eller någon typ av reaktion, men är utifrån dessa data inte möjlig att vidare utreda. Observationen att bentonit minskar mängden sulfid i lösning då natriumsulfid tillsattes stärkte observationen att ingen sulfid kunde detekteras i lösning jämviktad enbart mot bentonit. Två olika metoder användes för att detektera sulfid: metylenblåttmetoden och kopparsulfatmetoden (fäller ut CuS). De sista försöken gjordes i en handskbox med mindre än 1 ppm O_2. Det observerades att det var kritiskt att all bentonit (montmorillonit) togs bort från vattenlösningen innan reagensen tillsattes, då dessa interagerade.

Grova approximationer indikerar att cirka 1–10 kg sulfid verkar tas bort från lösningen per 1 000 kg bentonit. Detta kan jämföras med att kravet på bentoniten när det gäller sulfider är 0,5 viktprocent, eller 50 kg sulfid i mineral per 1 000 kg lera.

Contents

1 Introduction

Bentonite clay is planned to be used as a buffer material protecting the copper canisters in the KBS-3 method (SKB 2006, 2011) developed by the Swedish Nuclear Fuel and Waste Management Company (SKB). It is crucial that the bentonite not only protects the canister, but it should also not be harmful to the canister in any way. Potentially the bentonite may contain minerals, or other phases, that may have an impact on the canister corrosion (e.g. sulphide minerals such as pyrite). Pyrite is known to frequently occur in bentonites and the maximum allowed sulphide content of the buffer (the bentonite close to the copper canister) is 0.5 wt% (Posiva SKB 2017). Most bentonites investigated by SKB are below or well below this limit, however there are bentonites such as the Milos bentonite which can be fairly close to this limit (Karnland et al. 2006). The solubility of pyrite (FeS_2) is very low under repository conditions but it is uncertain if it will set the equilibrium concentration of sulphide. There are studies indicating rapid oxidation of pyrite with the oxygen initially present in the bentonite blocks (Svensson and Hansen 2013, Gaucher et al. 2009). The amount of pyrite in a deposition hole is relatively small however, the total amount of sulphide minerals in the bentonite backfilling the deposition tunnels is much larger. This sulphide is however expected to be too far away from the copper canister to be a problem, based on the assumption that the sulphide pore water concentration is very low and transport kinetics are very slow.

In this project, the target is to measure the sulphide concentration in water after dispersion of bentonite using the colorimetric methylene blue method (MB-method; Fischer 1883) described in detail in the Swedish Standard Method SIS 028115. The MB-method is based on that sulphide ions together with paraaminodimetylanilin form methylene blue in the presence of $Fe^{3+}_{(aq)}$. The experimental setup for the sulphide extraction was not trivial, as the speed of the dissolution of the sulphide phases in the bentonites was unknown (particle sizes are not known of the sulphides). However, while the dissolution of sulphide particles is expected to be relatively slow, the oxidation of dissolved sulphide is expected to be fast if traces of oxygen were present. Sulphide could also escape as $H_2S(g)$ if the system was is fully closed. Bentonites increase the pH of the water compared to pure water which is expected to reduce H_2S formation. However, the optimum duration of such an experiment is far from clear. Additionally montmorillonite, the main component in bentonite, very effectively absorbs methylene blue, hence any traces of montmorillonite must be removed from the solution before the sulphide determination can performed, and this without oxidising or degassing any H_2S from the solution.

2 Experimental

2.1 Methylene Blue (MB) method for sulphide

After stabilisation the sulphide content was analysed according to the SKB MD 452.011 SULFID method (based on SIS 028115). To the sample 2 ml amine sulphuric acid was added, followed by 0.5 ml iron(III)chloride solution and the solution was mixed thoroughly, and placed dark for 1 h. In the next step 3 ml diammonium hydrogenphosphate solution was added and the solution was mixed, and placed dark for 15 minutes. The evaluation was done by measuring the absorbance at 665 nm with UV/Vis spectrometer (this corresponds to absorbance by methylene blue which is proportional to the sulphide concentration). The method was calibrated by analysing the stock solution with an independent silver electrode.

2.2 Copper Sulphate (CS) method for sulphide

The following reaction was used as an independent alternative method for sulphide detection:

Cu^{2+} (aq) + S^{2-} (aq) à CuS (s) (covellite)

This reaction can be used to determine sulphide content in water (e.g. Cord-Ruwisch 1985). The Cu^{2+}(aq) ion has a blue color, while the covellite is brown or black, and hence the reaction with S^{2-} decreases the blue color of the solution and forms initially a brown dark colloid and later after aggregation a more solid black precipitate (Figure 2-1). Evaluation can be done visually and documented by photography, or by spectrophotometric measurement of the absorption of the Cu^{2+}(aq) (the blue color). Spectrometry of Cu^{2+} however only capture the decrease in blue and not the formation of brown/black. One should remember, that CuS can only form if sulphide is present, but Cu^{2+} can be consumed by other processes, such as ion exchange with montmorillonite if not properly removed in the earlier steps.

2.3 Bentonite – sulphide experiments

2.3.1 Using the MB method

Many and several types of experiments were performed (Table 2-1). The experimental setup was refined with time, and each type of setup is denominated by MB-*n*, were *n* is I to VI (I the first and VI the last type of experimental setup used).

A solution of sulphide was prepared from Na_2S either aerobically (MB-I to V) or anaerobically (MB-VI). The sulphide solution was added to the bentonite, together with deoxygenated water and in the last experiments $CaCl_2$ was added to reach a final concentration of 0.1 M (MB-V, MB-VI). The $CaCl_2$ was added to avoid formation of montmorillonite colloids (that may go through the 0.45 µm filter).

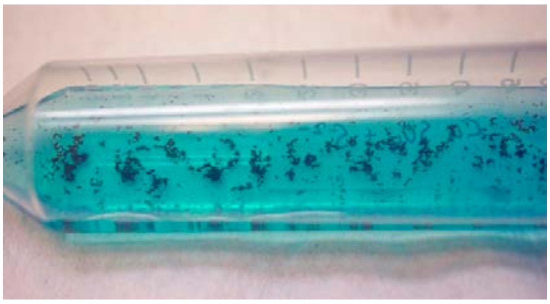

Figure 2-1. *Rapid precipitation (in seconds) of CuS formed from S^{2-} and Cu^{2+}, used for sulphide detection.*

The reaction time was from 1 day (MB-V) to approximately 1 month (MB-III). After the experiment the bentonite and liquid was separated (in all cases except MB-III) and the liquid was filtered in MB-V and MB-VI (0.45 µm filter). After the separation of the liquid from the bentonite the sulphide was stabilised with the addition of 0.5 ml 1 M NaOH and 0.5 ml 1 M Zn-acetate. After stabilisation the samples were stored dark until analysed.

Table 2-1. Overview of the MB experimental types and evolution with time. Ibeco BF is Milos bentonite (Greece), and MX80 Wyoming bentonite (USA).

Experiment type	Time	Materials	Comments
MB-I	2013	Ibeco BF	
MB-II	2014	Ibeco BF, Na_2S	
MB-III	2015	Ibeco BF, MX-80, Na_2S	Air removed. Bentonite not separated from the liquid (artefact).
MB-IV	2015	Ibeco BF, Na_2S	Air removed. No complete separation of montmorillonite from solution (artefact).
MB-V	2016	Ibeco BF, Ca-Ibeco BF, Na_2S, $CaCl_2$	Complete montmorillonite separation. Air removed.
MB-VI	2016	Ca-MX80, $CaCl_2$, Na_2S	Complete separation. Anaerobic box (< 1 ppm O_2) used. Still sulphide loss.

2.3.2 Using the CS method

The experiments (Figure 3-7) were performed in a glove box similar to MB-V, including the addition of $CaCl_2$ to reach a final concentration of 0.1 M $CaCl_2$. To minimize effects from oxidation, higher concentrations of sulphide were used in the CS-I and CS-II compared to the MB-experiments. The procedure of the experiment was: 1 or 0 gram of clay was added to a test tube, followed by the addition of water, sulphide solution and $CaCl_2$ to reach a final concentration of 0.1 M $CaCl_2$ (Figure 2-2). After a reaction time of 1–2 hours, the liquid phase was filtered (0.45 µm filter) into a new reaction flask. In the new reaction a minor amount of copper sulphate was added. If no sulphide was in the solution, the solution turned blue due to the color of the $Cu^{2+}(aq)$ complex, while in the case sulphide was present, the blue color was less pronounced, and a brown colloid formed of CuS that later aggregated to black particles. After the addition of Cu^{2+} the sample color was documented by photography (CS-I and CS-II) and quantitatively evaluated by computer software (CS-II).

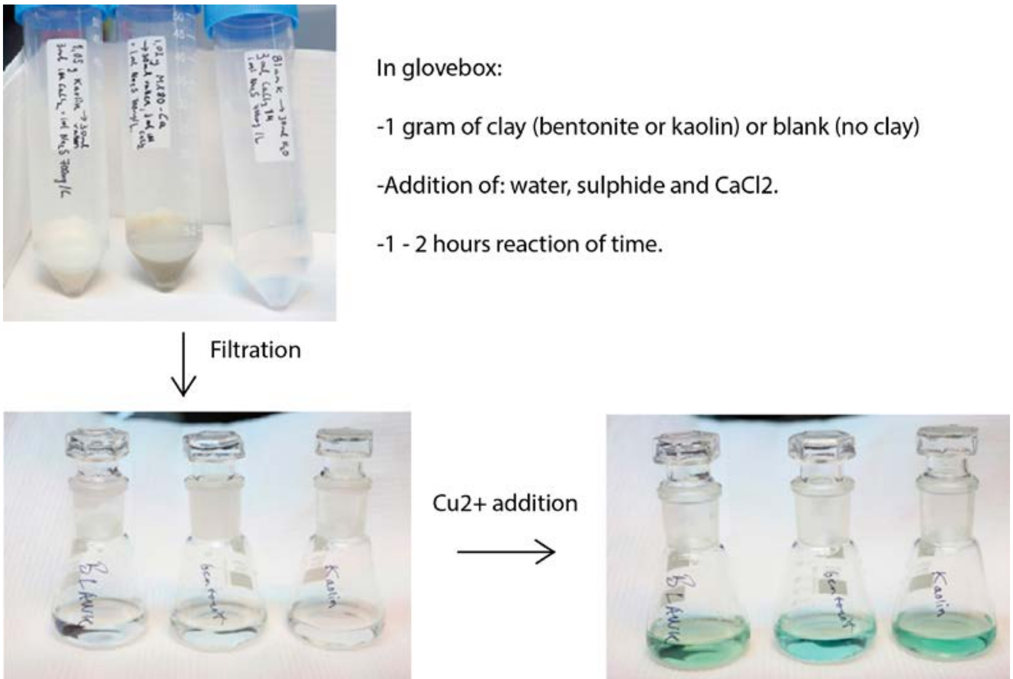

Figure 2-2. CS-I. Experiment with clay, water, sulphide and $CaCl_2$, illustrating the steps of filtration and Cu^{2+} addition. Overview of how the CS-experiments were performed.

Several experiments were made. If the amount of sulphide added was too high, no trend could be seen when adding the bentonite (the loss was to small in relation to the sulphide concentration), and when the added amount of sulphide was to small, it could not be detected in any sample afterwards. Hence, it was critical to have a high enough sulphide content, and a bentonite addition in the interesting interval (some absorption but not total). Hence, the following two experiments are reported here:

Table 2-2. Overview of the CS experimental types with details.

Type	Time	Materials
CS-I	2016	**Ca-bentonite, Kaolin, Blank** **Blank** 30 ml water + 3.5 ml 1 M $CaCl_2$ + 1 ml 700 mg/L sulphide solution. **Ca-bentonit** 1 g diluted with water until 30 ml. 3.5 ml 1 M $CaCl_2$ + 1 ml 700 mg/L sulphide solution. **Kaolin** 1 g diluted with water until 30 ml. 3.5 ml 1 M $CaCl_2$ + 1 ml 700 mg/L sulphide solution.
CS-II	2016	**MX80 bentonite different masses** **Blank-0, Blank-0** 30 ml water + 3.5 ml 1 M $CaCl_2$ + 1 ml 1 000 mg/L sulphide solution (c = 29 mg/L). **Bentonite-10, Bentonite-50, Bentonite-100, Bentonite- 500,** (10 to 500 mg of MX80 bentonite was added)

3 Results and discussion

3.1 Experiments using the Methylene Blue (MB) method

A summary of the experiments can be seen in Table 2-1.

In the first type of experiment, MB-I, only bentonites and deoxygenated water was used. No sulphide could be detected above the reporting limit. The montmorillonite was not fully separated from the supernatant, and hence, later we understood that this resulted in an artefact and the results could not be used.

To investigate if the MB method worked or not for the purpose, the bentonite samples were spiked with sulphide (Na_2S) in the next type of experiment (MB-II). In MB-II no precautions were taken to minimise the amount of oxygen except for the use of degassed oxygen free water. To the bentonite-water solution different amounts of sulphide was added and the experiment was allowed to stand for about a week. The bentonite was separated from the solution by decantation of the liquid phase prior to adding the reagents. After the reagents were added the solution was filtered through a 0.45 µm filter before they were measured by spectroscopy to quantify the MB. Due to the filtration some of the filters turned blue, this was later interpreted as that traces of blue montmorillonite were present in the solution (Figure 3-1), and where the blue colour came from the absorption of methylene blue. Hence the montmorillonite absorbed the indicator of sulphide. Potentially the bentonite or the montmorillonite was also involved in the chemical reactions forming the MB. As the amount of oxygen was not reduced in the bentonite, and as the montmorillonite absorbed created artefacts, including the absorption of methylene blue, this series of experiments was regarded to have too many artefacts to really be used for evaluation; hence none of this data is further used.

The details of the MB-montmorillonite interaction was not clear to us at the moment, and in MB-III the focus was on removing all oxygen from the bentonite, and from the bentonite-sulphide solution during the experimental time. To reduce the risk of oxidation, which was interpreted as the biggest problem at the moment, the bentonite was not removed from the solution before the addition of the reagents (Figure 3-2). Hence, in MB-III, the montmorillonite-MB interaction was even larger than in MB-II, although the oxidation and H_2S escape was minimised by the setup. Due to the pronounced influence of the bentonite no data is used for evaluation from this series.

In MB-IV the procedure was identical to MB-II, but the exposure to oxygen was minimised by the techniques introduced in MB-III. But the montmorillonite was still not successfully removed from the solution.

Figure 3-1. *MB-II. No control of oxygen and filtration after addition of reagents show montmorillonite traces. Ibeco BF bentonite used.*

Figure 3-2. MB-III. Air removal but no bentonite– water separation.

Na-montmorillonite forms colloids in water solution at low ionic strength, and some of these colloids were found to be too small to be trapped with the 0.45 µm filter. To be able to fully remove montmorillonite from the solution, the montmorillonite must be ion exchanged into the Ca, as Ca-montmorillonite does not form colloids and instead aggregate into bigger particles. The MB-V experiment included both raw bentonite and Ca-converted bentonite. The Ca-converted bentonite was done by washing with 1 M $CaCl_2$ three times followed by washing with water three times, however, later it was found to be enough to simply add 1 M $CaCl_2$ to the solution to reach a concentration of 0.1 M.

In MB-V there was no problem with bentonite in the solution during the addition of the reagents, however, still there were some problem with sulphide escape/oxidation as the total values were much lower than expected.

Although some oxidation occurred, the MB-V experiment was free from artefact and hence it provided some interesting data. In the experiment with no added sulphide, different amounts of bentonite was added to the water. Both raw bentonite and Ca-exchanged bentonite was used, and in no case sulphide was detected (Figure 3-3). However, this behaviour could still have been due to oxidation or H_2S escape.

More interesting are the two series of samples that were spiked with a lower (0.9 mg/L; Figure 3-4) and higher (1.6 mg/L; Figure 3-5) level of sulphide. In both series a clear trend could be seen that the added bentonite lowered the amount of sulphide in the solution by absorption or some kind of chemical reaction. One possibility was that the bentonite porosity contained some air (oxygen) and the more bentonite that was added, the more oxygen was introduced into the system that simply could have reacted with the added H_2S.

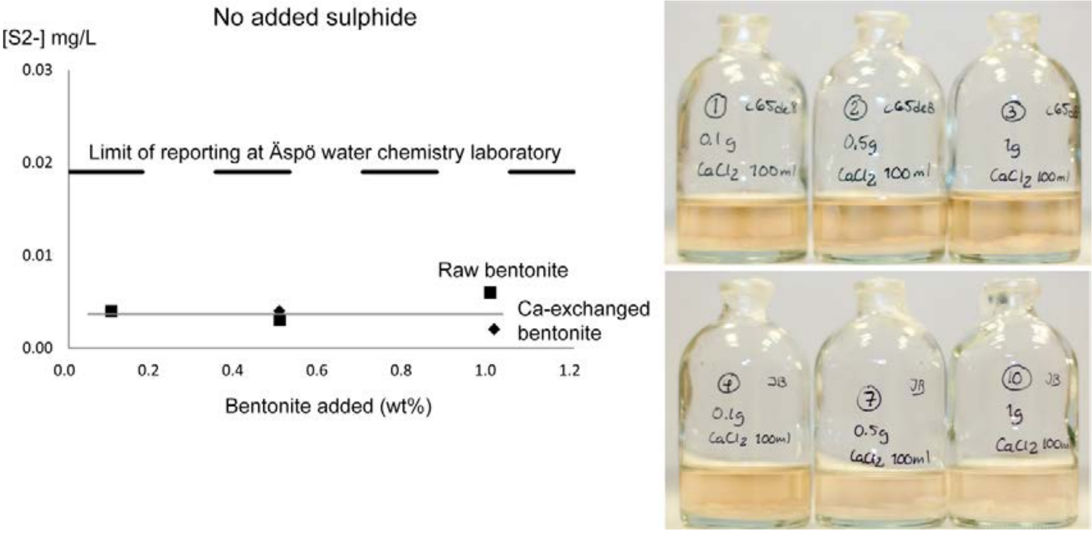

Figure 3-3. MB-V. The measured sulphide content in the solution after the addition of different amounts of Ibeco BF bentonite. All points below limit of quantification (LOQ) 0.019 mg/L. Limit of detection is 0.006 mg/L. Uncertainty in the measurements is approximately ± 32 %. Lines were added as guides for the eyes.

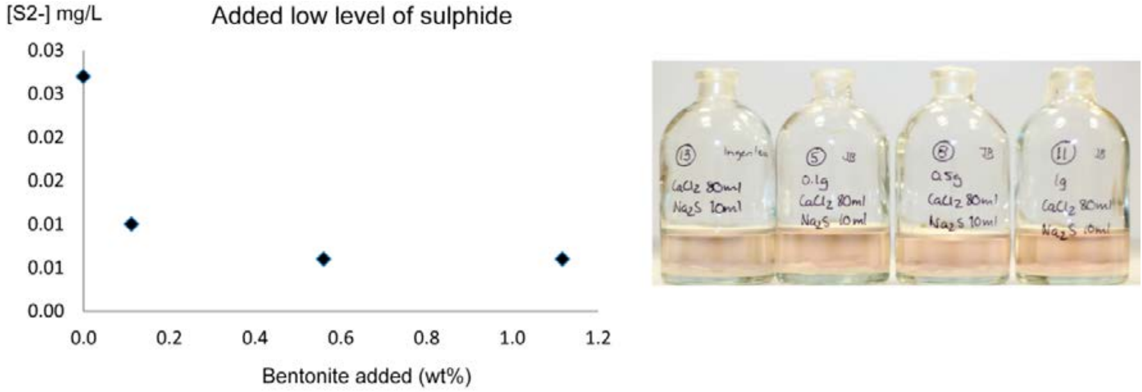

***Figure 3-4.** MB-V. Constant sulphide addition (c = 0.9 mg/L) with different amounts of Ibeco BF bentonite (wt%). Limit of quantification (LOQ) 0.019 mg/L. Limit of detection is 0.006 mg/L.*

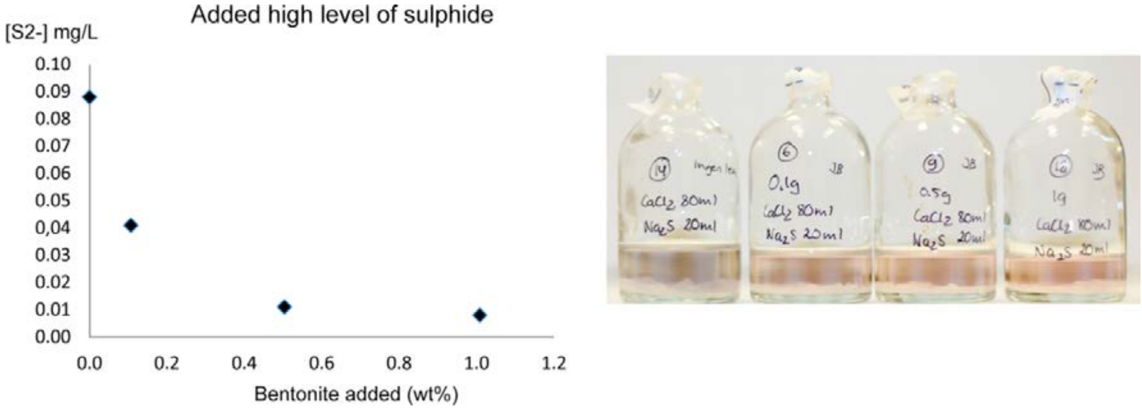

***Figure 3-5.** MB-V. Constant sulphide addition (c = 1.6 mg/L) with different amounts of Ibeco BF bentonite (wt%). Limit of quantification (LOQ) 0.019 mg/L. Limit of detection is 0.006 mg/L.*

To fully exclude the effect from oxidation by air, the MB-VI was done within an anaerobic box (< 0.1 ppm O_2). In MB-VI all solutions and reagents were prepared inside the box, based on oxygen free water that had been standing to equilibrate in the box for about 2 months.

To the bentonite in the glovebox, water, sulphide solution and calcium chloride solution was added (to reach 0.1 M $CaCl_2$). This mixture was allowed to react for about 2 hours. After the reaction the liquid was filtered (0.45 µm) into a new flask. In the new reaction flask reagents were added to conserve the sulphide (NaOH and Zn-acetate). The stabilised sample (free from montmorillonite) was taken out from the glovebox and analysed using standard method at the Äspö water chemical laboratory. In the samples contained either no bentonite or a fixed amount of bentonite (100 mg) and different amounts of added sulphide. As previous data indicated that bentonite reduced the sulphide concentrations, to the samples with bentonite more sulphide was added in order to ensure detectable sulphide levels in the end. Hence, it was a qualified guess what proportions of sulphide and bentonite that would give measurable and comparable data. It was clear from the results that the addition of bentonite lowered the sulphide content (Figure 3-6), however still for some reason a big part of the sulphide seems to be lost during the steps (artefact). However, in this case, no oxygen is present in the bentonite, and the unintentional oxidation should be equal to all samples as well as the H_2S escape (which should be even lower in samples with bentonite due to the higher pH). After adding 1 mg/L to water with no bentonite 0.4 mg/L could be detected, while in the case with bentonite to be able to detect a similar amount about 2.5 mg/L had to be added. It is clear that the bentonite lowers the sulphide content, but to what extent is from this data still unclear due to the impact from oxidation/H_2S escape and as the investigated interval of samples with and without bentonite was fairly separated.

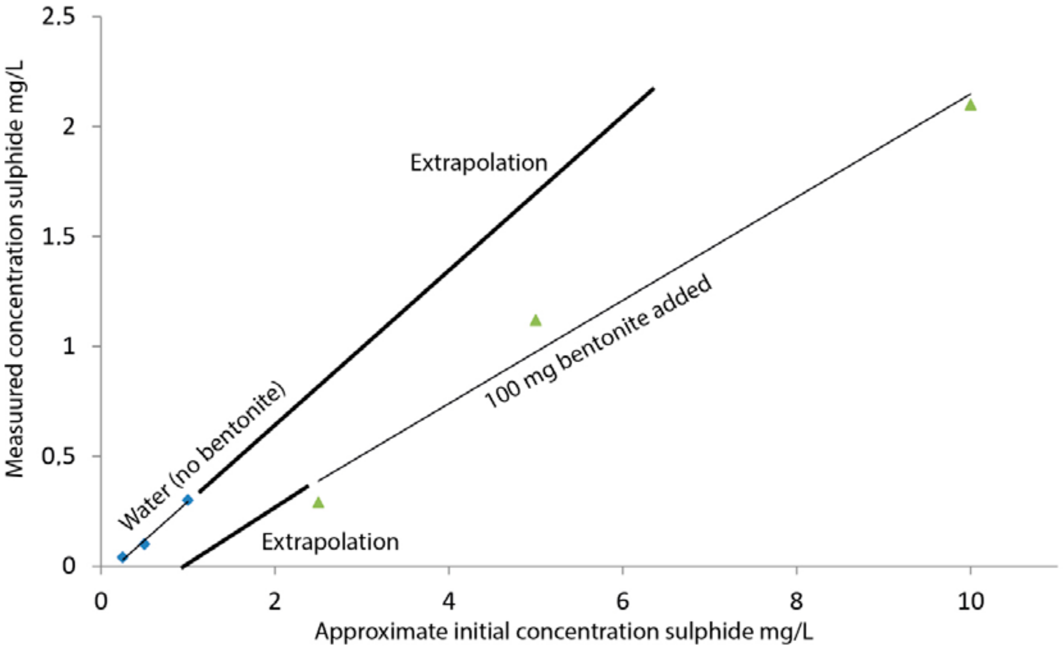

Figure 3-6. *MB-VI. Measured sulphide conentrations as a function of the theoretical value. In pure water and in water-bentonite mixture (100 mg MX-80 bentonite). Lines were added as guides for the eyes. Triangles = samples with bentonite. Squares are samples with no bentonite. Limit of quantification (LOQ) 0.019 mg/L. Limit of detection is 0.006 mg/L.*

3.2 Experiments using the Copper Sulphate (CS) method

A summary of the experiments can be seen in Table 2-2. In CS-I a fixed amount of sulphide was added to test tubes with no clay, bentonite and with kaolin clay (field sample collected at Rokle mine, Czech Republic). In the case of no bentonite and/or kaolin, sulphide could be detected (Figure 2-2) while with bentonite (MX-80) no visible signs of sulphide could be seen. Hence, CS-I confirm the sulphide loss behaviour observed in the MB-experiments.

In the next experiment, CS-II, only bentonite was used. To a fixed addition of sulphide (initial value = 29 mg/L) a series with 0, 0, 10, 50, 100 and 500 mg of bentonite was added (Figure 3-7).

Visually there was a small difference already between 0 and 10 mg bentonite, at 50 mg the difference was more significant, and at > 100 mg of bentonite the solutions were clearly blue. The dark color in the test tubes with no bentonite indicated high levels of sulphide present at the addition of Cu^{2+}, while the blue color in the test tubes with bentonite indicated Cu^{2+}. A color measurement of the amount of blue in the samples was done with Adobe Photoshop color sampling tool (5 sample points per test tube). The blue index of the RGB values represent the amount of blue present, here scaled towards the maximum value of 255 to be expressed as percent (Figure 3-7b). The curve has the same appearance as is seen in Figure 3-4 and 3-5. Comparing the 100 mg and 500 mg additions show only minor difference, indicating that a plateau has been reached (Figure 3-7b). The experiment was not designed to be quantified, hence unfortunately no zero sample was included with only Cu^{2+}. But based on the rapid decline in sulphide in MB-V (e.g. Figure 3-4) to rapidly go to values below limits of quantification, and to the plateau of the 500 tube, the 500 tube is here assumed to have a very low sulphide concentration close to zero.

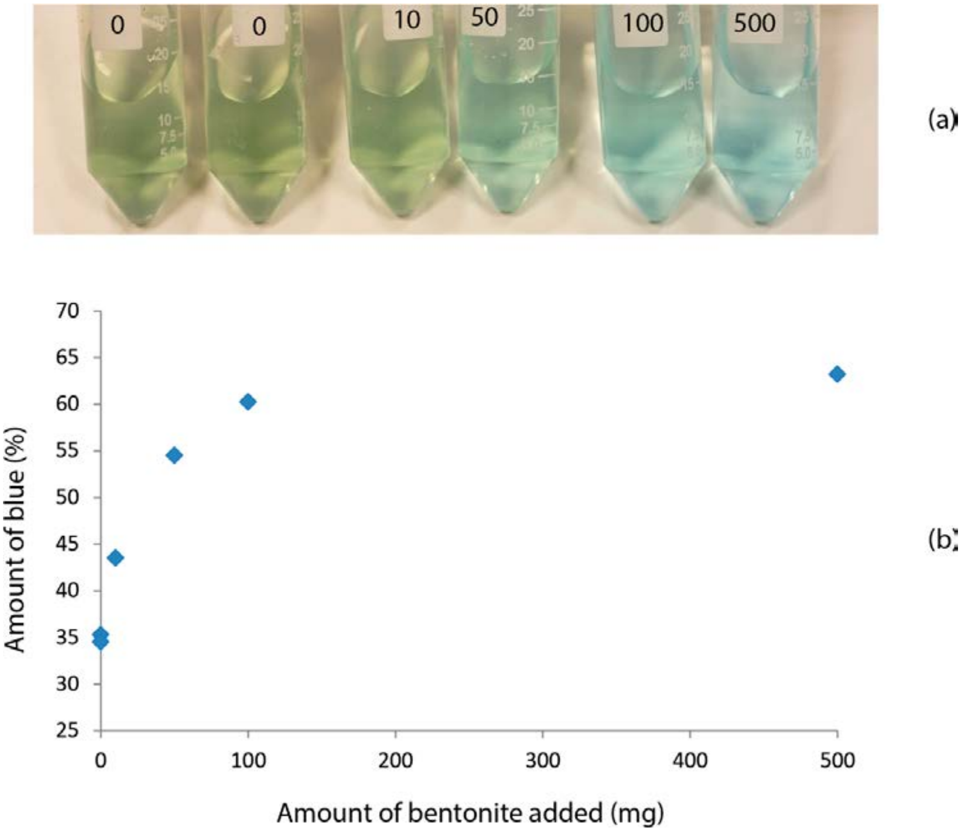

Figure 3-7. *CS-II. Constant sulphide addition (29 mg/L in 33 ml) different mass of bentonite (0–500 mg) added to each tube. In the picture (a) the filtered supernatant is shown after* Cu^{2+} *addition (1 ml 1 M/tube). From left: 0, 0, 10, 50, 100 and 500 mg. Blue color indicate* Cu^{2+}*. A color measurement by software (b) estimated the amount of blue in the samples which is proportional to* Cu^{2+} *and inversely proportional to* S^{2-}*.*

4 Discussion

The level of sulphide in the water after contact with bentonite was below the limit of quantification (Figure 3-3). Even with minor additions of sulphide to the bentonite still the level was below the limit of quantification (Figure 3-4 and 3-5). There was evidence or strong indications that the bentonite lowered the sulphide concentration in solution (Figure 3-4, 3-5, 3-6 and 3-7), this supports that the equilibrium concentration of sulphide in solution in contact with bentonite is very low.

The exact amount of sulphide that is lost by the bentonite cannot precisely be determined from these measurements due to the uncertainties. Nor is there any clear information suggesting a mechanism for the sulphide loss. It is however possible to do rough estimations based on the results obtained to make an estimate of the magnitude:

Based on Figure 3-6, about 66 % of the added sulphide is lost (when 1 mg/L was added 0.4 mg/L was detected, when no bentonite was added).

The initial sulphide concentration in the CS-II experiment was 29 mg/L (very high). The volume was 34.5 ml.

Based on Figure 3-8b 100 mg bentonite approximately removed the added amount of sulphide.

The added amount (34.5 ml × 29 mg/L) × (1/3) = 0.33 mg.

0.33 mg/100 mg bentonite = 0.3 wt%.

Hence, based on the experiments done, a very rough estimation indicate a sulphide loss in size of 0.3 wt% of the bentonite, or 1 000 kg bentonite would remove about 3 kg of sulphide (a better guess perhaps is 1–10 kg sulphide/1 000 kg bentonite).

5 Conclusions

Plenty of time was used on method development, as it was far from trivial what kind of setup, reaction times and concentrations that were suitable for the experiments. The water to be analysed for sulphide must be totally free from montmorillonite, as it strongly interfered with the methylene blue method for sulphide determination, this was more critical than first expected. The combination of an addition of $CaCl_2$ salt to 0.1 M and filtration of the clear liquid (0.45 µm filter) was able to remove all traces of montmorillonite. The $CaCl_2$ salt was added to make sure montmorillonite did not form colloids that would have been small enough to go through the filter. The $CaCl_2$ together with the MB-reagents precipitated gypsum ($CaSO_4$) in the final step that had to be filtered away, at his stage the sample was however no longer sensitive to oxygen. When no Na_2S was added no sulphide could be detected in the water that had been in contact with bentonite (the concentration was below Limit of Quantification; LOQ). It was found that the selected bentonites reduced the amount of sulphide in solution when sulphide was added, hence bentonite is not expected to by itself release enough sulphide that it can be detected using this method and procedure. The sulphide loss mechanism (absorption/transformation/reaction) is not known but was found to be fast and 1–2 hours seemed to be enough for the reaction to take place. A very rough estimation was done from the data, indicating that the bentonite seemed to reduce an added amount of sulphide in the range of 1–10 kg per 1 000 kg of bentonite.

More experiments would be needed in order to understand this behaviour further and to better quantify the sulphide loss by the bentonite. This was however outside the scope and time frame of this project. One experiment was done with kaolin clay and no similar behaviour on sulphide was seen in that case.

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SKB is responsible for managing spent nuclear fuel and radioactive waste produced by the Swedish nuclear power plants such that man and the environment are protected in the near and distant future.

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