Applicability and Suitability of Different Alkaline By-Products in Historic Mine Sites Remediation

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Abstract
Six alkaline by-products were evaluated in various experiments in order to determine their applicability and suitability in remediation of historic mine sites. The alkaline materials were either of carbonate type (lime mud, green liquor dregs, water works granules), hydroxide type (lime kiln dust, LD-slag) or mixed carbonate/hydroxide type (fresh fly ash, carbonated fly ash). Experiments were performed at the bench scale and pilot scale over a four year period. The materials were used as a neutralizing agent in reactive filters to treat acidic drainage and as an amendment blended with acidic waste rock to neutralize stored acidity in-situ to prevent acidic drainage (stabilization). In the stabilization experiments, both layering and homogeneous mixing of materials were tested. It was found that the different alkaline materials were suitable for different applications. Lime mud and lime kiln dust were more effective at preventing acidic drainage when mixed with waste rock, whereas carbonated fly ash was the most effective when layered with waste rock. Hydroxide materials were superior to carbonate materials in filter experiments for treating acidic seepage.

Key Words: LD-slag, LKD, fly ash, lime mud, green liquor dreg

Introduction
Despite their contamination potential, historic mine sites, with mineral waste heaps and old buildings, are valued for their industrial and cultural heritage (Conesa*et al*. 2008; Rieuwerts*et al*. 2009).Hence, remediation techniques that make as little visual impact as possible are important. *In situ*techniques are the preferred remediation approach to achieve this goal.

High weathering rates in sulphidic waste have resulted in acidic leachates laden with dissolved metals. Alkaline amendments are required to reduce the acidity and metal concentrations in these leachates. Various alkaline industrial by-products have been used to amend and neutralize acidic mining waste and acidic drainage. By-products are cheap alternatives to primary materials like limestone and quicklime and re-utilization of industrial process residues conserves natural resources.

Reactive (alkaline) materials can be tilled or injected into a pile containing sulphidic mine waste. This will reduce the pyrite oxidation rate, neutralize the acid produced and immobilize metals (through sorption and/or precipitation) at the source and prior to discharge. Also, reductive dissolution of secondary iron(oxy)hydroxides and iron(III) mediated weathering of pyrite can be prevented. Injecting the material as a slurry would have the advantage of preserving the landscape, ifthat is desired.

The use of alkaline slags and ashes may result in hard pan formation through pozzolanic reactions. A hardpan is an impermeable barrier, resulting from the formation of calcium-silicate-gel (CSH) and calcium-aluminate-gel (CAH) (Bertocchi*et al*. 2006; Shang *et al*. 2006; Xenidis*et al* 2002). As a hardpan makes infiltration of oxygen and water difficult, fly ash is sometimes incorporated in sealing layers for waste rock and tailings. A hardpan can, however, also consist of an accumulation of secondary precipitates (goethite, gypsum, jarosite).Hard pan formations decrease the flow rate through the pile and hence increase the contact time between water and the alkaline material.

Materials and Methods

Alkaline materials

Lime kiln dust (LKD)
Lime kiln dust (LKD) is a waste residue from the liming industry (Nordkalk, Köping, Sweden). Quicklime (CaO) is produced in a kiln by calcination of limestone ($CaCO_3$) at 1400 ºC:

$$CaCO_3(s) + \text{heat} \rightarrow CaO(s) + CO_2(g) \quad (1)$$

LKD is a fine-grained material with low permeability which could make it suitable as an impermeable cover; the hydraulic conductivity is estimated to be $2\text{-}3 \cdot 10^{-10}$ m/s. The material consists mainly of quicklime and various oxides and carbonates ($CaCO_3$ content 66 %). The pH is approximately 12.4 (close to the $Ca(OH)_2$ equilibrium pH).
The reaction between quicklime and water as well as the neutralization reaction between the hydrated lime and acid are shown in reactions 2 and 3.

$$CaO(s) + H_2O \rightarrow Ca(OH)_2(s) \quad (2)$$
$$Ca(OH)_2(s) + H_2SO_4 \rightarrow Ca^{2+} + SO_4^{2-} + 2\ H_2O \quad (3)$$

LD-slag (LD)
LD-slag is a by-product from the steel industry, the "Linz-Donawitz" process (SSAB Merox AB, Oxelösund). Additives to the process, such as quicklime and dolomite, and oxidizing substances from the rock (iron ore) (e.g. silica, iron and phosphorous) are components in the LD-slag. It contains some unreacted lime (CaO) resulting in approximately pH 12.3 for the final product. The $CaCO_3$ content of LD-slag is 5 %. It also contains several heavy metals, for example vanadium and chromium. Minor quantities of LD-slag are used as an amendment in asphalt and as a gravel substitute.

Lime mud (LM)
Lime mud is a waste residue from the pulp and paper industry (KorsnäsFrövi, Sweden) and consists mainly of calcite (97 %) and small amounts of quicklime and hydrated lime (Martins *et al.* 2007). The pH is around 11.4. In the Kraft process, hydrated lime reacts with the green liquor containing sodium carbonate (Na_2CO_3) to regenerate white liquor, which is a solution containing sodium hydroxide (NaOH) used as a pulping chemical. The solid by-product separated from white liquor is denoted *lime mud*, which is then washed and thickened to about 70%.The fine-grained lime mud residue could be a potential neutralizing cover component.

Green liquor dreg (GLD)
Green liquor dreg (GLD) is a waste residue from the pulp and paper industry (KorsnäsFrövi, Sweden). It is generated before the lime mud in the Kraft process. GLD consists of sodium carbonate (Na_2CO_3)/ calcite(70 %) and sodium sulphide (Na_2S), and is approximately pH 11.3. The material could be effective as a neutralizing as well as a reducing agent due to its sulphide content.

Fly ash (FAF and FAE)
Two fly ash samples were tested in this study. One sample (FAF) came from the pulp and paper industry (KorsnäsFrövi, Sweden) with a bubbling fluidized bed boiler (BFB), bio fuel and electro filter. The other sample (FAE) came from an incineration plant used to generate district heating (E.ON, Örebro, Sweden) with a circulated fluidized bed boiler (CFB), bio fuel and electro filter. The $CaCO_3$ content of FAF and FAE were 30 and 23 %, respectively.

Fly ash (FA) generally consists of silicon, aluminum, iron, magnesium and calcium oxides. The fly ash is more suitable for remediation purposes than bottom ash, due to its homogeneity and lower concentrations of heavy metals compared to bottom ash. The calcium and magnesium oxides of the FA react with water generating hydroxyl ions that can neutralize the hydrogen ions in ARD. Pore fluid pH increases and dissolved metals are precipitated (Polat*et al.* 2002).

Processes producing CSH- (calcium silicate gel) and CAH- (calcium aluminate gel) can lead to the formation of an impermeable hard pan that would hinder the transport of water and air. Formation of the mineral ettringite from a reaction between calcium aluminate monosulphate and gypsum is an example of such a process (reaction 4). The formation of ettringite and similar minerals in sealing layers is, however, not always advantageous since ettringite is an expanding mineral. Hence the formation of ettringite can cause physical and chemical instability.

$$6\ Ca^{2+} + 2\ Al(OH)_4^- + 3\ SO_4^{2-} + 4\ OH^- + 26\ H_2O \rightarrow Ca_6[Al(OH)_6]_2(SO_4)_3 \cdot 26\ H_2O(s) \quad (4)$$

Other materials

Waste rock
Ljusnarsberg historic mine site is situated in the centre of the town of Kopparberg in the Bergslagen region. The mine field was discovered in 1624 and the latest mining period ceased in 1975. Approximately 65 large or small pitheads have been found within the central parts of the field. It is estimated that a total area of 500×400 m^2 is affected by mining, ore and waste rock dumps.

The mine site was abandoned in 1975. Several waste rock piles remain in the area. The waste rock is heavily oxidized and covered with secondary precipitates. The material was crushed and sieved, with the <13 mm material used for the experiments.

Prevention of ARD Test Method (Stabilization)

Layered Alkali Experiments
The stabilization experiments demonstrate the long-term changes (both chemical and hydraulic) taking place when a weathered and acidic historic mine waste is layered with an alkaline material. Waste rock from the Ljusnarsberg mine site, Kopparberg, was used in this experiment. Waste rock and alkaline materials were layered inside PE-plastic barrels. Eight different layering configurations were evaluated, as specified in Table 1 and shown in Figure 1. Expanded clay aggregates (Leca®) were added to each layer (25%) in order to minimize the risk of clogging and increase the hydraulic conductivity. The barrels were 1.5 m high with a diameter of 0.8 m (total volume 0.7 m^3).

Table 1.Alkaline materials and number of alkaline layers in the eight test barrels.

System	Alkaline material (%)	Alkaline material	No of alkaline layers
T1	50	FAF	3
T2	10	LM	11
T3	0	None	0
T4	10	FAF	7
T5	25	GLD	3
T6	10	GLD	11
T7	10	FAE	11
T8	25	FAE	10

Tap water is added to the top of the barrels and allowed to percolate by gravity through the layers of alkaline material and waste rock. Approximately 50 L water is added each month. Since the start of the

experiment in December 2006, some 820 litres of water has passed through each barrel, corresponding to an L/S ratio of around 1. Samples are taken from the outlet at the bottom of the barrels, as well as from suction lysimeters installed inside the barrels (L1, L2 and L3 in Figure 1). Samples are analyzed for pH, Eh, electrical conductivity, alkalinity/acidity, inorganic anions and major and trace elements. Variable flow rates have been used in order to study how different amendments may develop a hard pan. Since the start of the experiment, the barrels have been kept indoors at approximately 10°C.

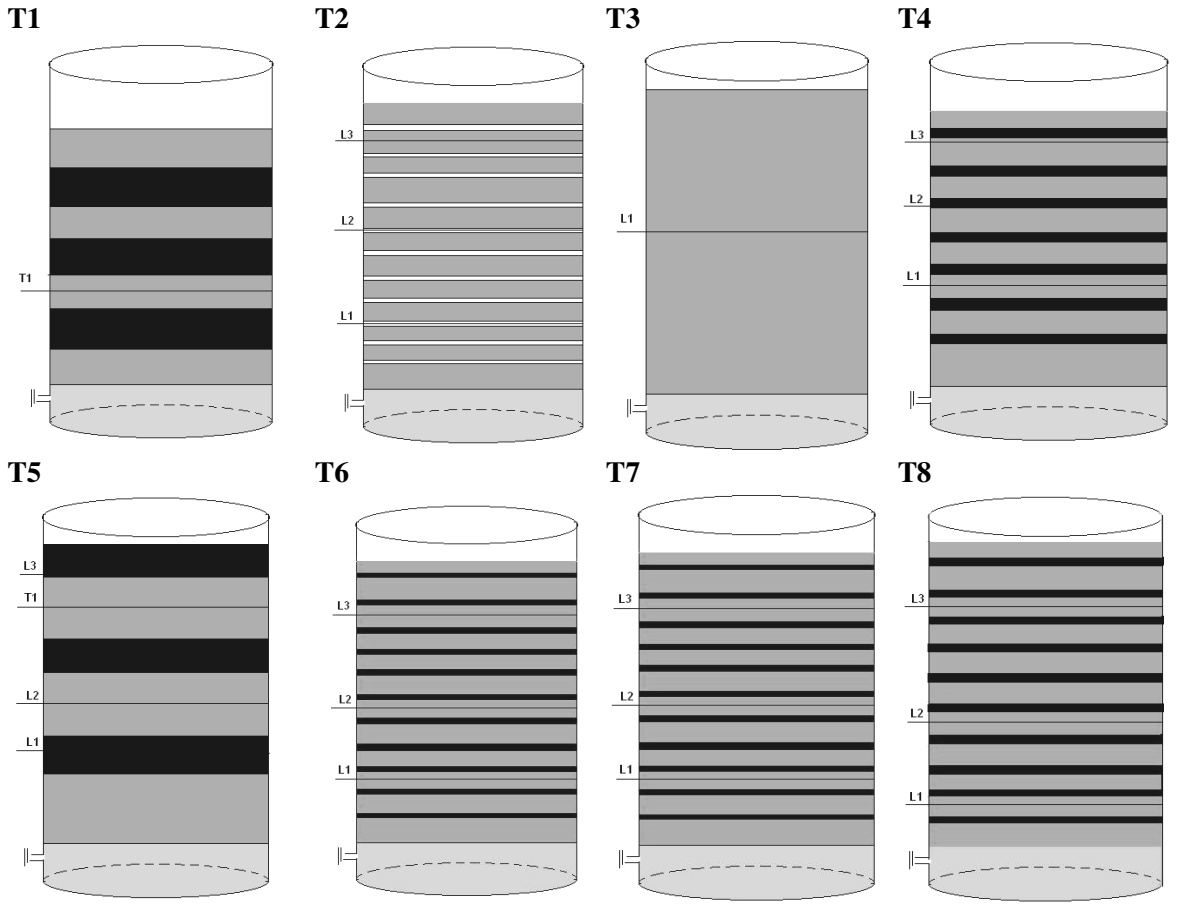

Figure 1.Schematic of the layers in each barrel test. L1-L3 in the figure stands for lysimeters 1 to 3, T stands for thermometer.

Alkali/Waste Rock Mixing Experiments

The interaction between the alkaline by-products and acid-producing waste rock was studied by mixing mine waste and the alkaline product in batches. Experiments were set up in 50 ml Sarstedt tubes, with eight tubes for each alkaline material (Figure 2). Five grams of a mixture of mine waste (crushed, homogenized) and the alkaline material was added to each tube in different proportions (0, 5, 10, 15, 20, 30, 50 and 100 %, respectively). Fifty mL of deionized water (18.2 MΩ) was then added, giving a liquid/solid ratio (L/S) of 10. The tubes were sealed and agitated (end-over-end shaker). A 10mL sample was taken after 24 hrs, 3 days, 8 days, 28 days and 65 days for analysis of pH, electrical conductivity, alkalinity, inorganic anions and major and trace elements. Each sample was replaced with deionized 18 MΩ water to maintain a constant liquid/solid ratio in the tube.

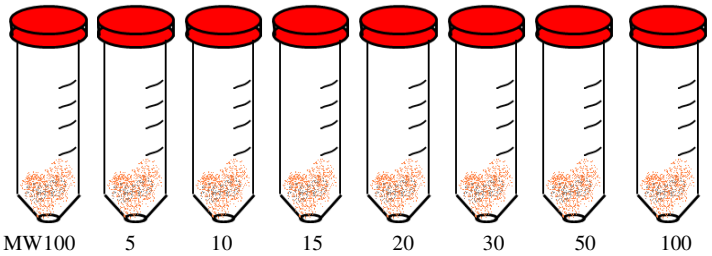

Figure 2.For each alkaline material, series of eight Sarstedt tubes containing the mine waste and the alkaline material were set up. The first and the last tube contained 5 g of only mine waste (MW100) and the alkaline material (100), respectively. Tubes 5 to 50 contained a 5 g mixture of mine waste and the alkaline material (5 to 50 weight-%) .

Treatment of ARD Test Method

Pilot-scale filters

Meso-scale filter sections for treatment of ARD were constructed in 2007, and experiments started in May 2008. Eight filter lines were evaluated, where each line consisted of three barrels in sequence connected with 2" diameter rubber tubing. Each barrel (polyethylene) had a total volume of 0.4 m^3 (length 1.5 m, diameter 0.6 m) and contained baffles that prevented channelling of the water flow. The coupled 3-barrel lines are placed outdoors on a gravel bed with approximately 10° inclineto allow water to flow by gravity through the line (Figure 3). ARD for the filter lines was created in two reactors (R1 and R2). Each reactor was a 1 m^3 plastic container filled to 75% with minus 13 mm waste rock. Water pumped from a nearby mine shaft was added to each reactor and the resulting ARD flowed through the filter line.The residence time before the resultant ARD went into the filter lines was 6 hours. ARD quality was measured each day the filters were in operation.The flow rate was controlled by valves on the inlet tubing to each filter line.

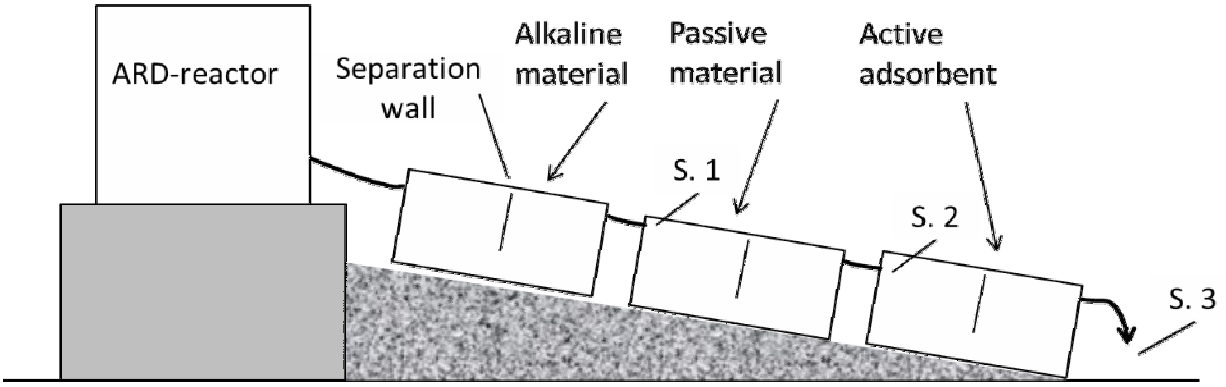

Figure 3.Schematic of the sequential filter lines.ARD from the reactor passes through the filter sections by gravity (the filter sections lay on a gravel bed with approximately 10° incline). Sampling locations S.1, S.2 and S.3, after the first, second and third filter section, respectively, are marked in the figure.

The composition of each filter line is listed in Table 2. The first barrel in each filter line contained an alkaline material. In order to facilitate precipitation and adsorption the test lines also contained passive materials at a suitable size range (5-25 mm). One passive material was lightly expanded clay (Leca®). The other material was crushed clay brick (Table 2). The purpose of the passive materials was to provide surface sites for precipitation of Fe- and Al-hydroxides. Active adsorbents were included in the final barrel. These were peat granules (*sphagnum spp*.) and water works granules (WWG).

ARD produced from reactors R1 and R2 were fed to filter lines 1-3 and 4-6, respectively. Filter lines 7 and 8 received ARD directly from the nearby shaft (T). The flow rate was in the range of 10-15 L/hour, giving a water residence time of around 24 hours. ARD was added in 100 L increments to the filter lines 3-4 times every week from May to October. The sampling frequency was typically every 50 L while ARD was added to the lines.

Table 2.Alkaline materials, passive materials and active adsorbents in the filter lines (F1-F8). W-P stands for water works granules and peat.

Filter	**F1**	**F2**	**F3**	**F4**	**F5**	**F6**	**F7**	**F8**
Reactor	R1	R1	R1	R2	R2	R2	T	T
Section 1	FAF	LM/FAF	FAE	LKD	GLD	LD	LKD	LM/FAF
Section 2	Brick	Leca®	Brick	Leca®	Brick	Brick	Leca®	Leca®
Section 3	Peat	Peat	Peat	Peat	W-P	W-P	W-P	W-P

Smaller treatment experiments

Sequential batch experiments, based on the method described by Bäverman*et al.* (1997), were used to demonstrate the processes taking place in a sequential filter receiving ARD. Water was passed through a sequence of batch units (Figure 4). Samples were collected from each batch unit before the leachate was moved to the next batch unit. The migration of the acid plume, and associated metals, can be monitored and used to predict break-through and retention times in field-scale filter experiments.

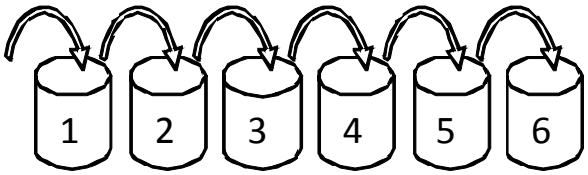

Figure 4.Fluid shifts in a sequential batch experiment, simulating continuous addition of acidic water to an alkaline material. Each ARD portion is in contact with the alkaline material for 24 hours and then the tubes are centrifuged and the water phase is transferred to the next tube. As the L/S ratio cumulatively increases, the acid plume moves forward.

Two sequential batch tests were set up, each consisting of six 250 ml centrifuge tubes, to evaluate lime kiln dust (LKD) and LD-slag (LD). The alkaline materialwas homogenized and 20 grams put into each of the six centrifuge tubes (LKD into one set of 6 tubes, LD into the other set). ARD collected from the Bersbo copper mine, Åtvidaberg municipality, Sweden, wasaerated to oxidize iron, filtered through 0.4 µm polycarbonate filtersand 200 mL added to the first tube of each 6 tube series.The tubes were put on an end-over-end shaker for 24 hours and then centrifuged for 30 minutes at 20 000 g. The leachate was removed: 3 mL of the total 200 mL for analysis and the rest was added to centrifuge tube 2 in each of the two series (LKD and LD). A new 200 mL portion of ARD was added to tube 1 in each of the two series and the procedure was repeated. Thus, after the sixth experiment day, the first ARD portion had passed all six tubes.

Analytical

Electrical conductivity, pH and redox potential were determined immediately after sampling using the relevant electrodes. Alkalinity (end-point pH 5.4) was determined through titration with HCl. Inorganic anions (chloride, fluoride and sulphate) were analysed with ion chromatography. Elemental analysis was performed using ICP-MS. All analysis was done at Kopparberg except for the ICP-MS (in a clean room at Örebro University).

Results and Discussion

Stabilization experiments

Alkali/waste rock mixing experiments with mixtures of various alkaline materials revealed large differences in pH and alkalinity, depending on the nature of the source – oxide/hydroxide or carbonate.The proportion of alkali in the mixtures ranged from 0-50%; however, pH in the carbonate amended mixtures (LM, GLD, LM/FAF) was almost independent of the amount added.The alkalinity decreased in the carbonate mixtures with increasing alkali additions due to the lower carbonate dissolution rates at higher pH (Figure 5, top right).When the surface acidity had been neutralized, indicated by the solid vertical line in Figure 5, calcite would be present in excess, leading to an increasing pH.This leads to substantially reduced dissolution of calcite and lower levels of dissolved alkalinity (HCO_3^-) (Figure 5, FAF).Addition of 5% of the carbonate materials LM and GLD was enough to reduce trace element concentrations by more than 97%. An alkaline addition of 5% was, however, not sufficient for trace element reduction (notably Zn and Pb) in the fly ash amended systems (FAE and FAF). Desorption of lead and copper at high pH (>11) was found for the LKD and LD amended systems.

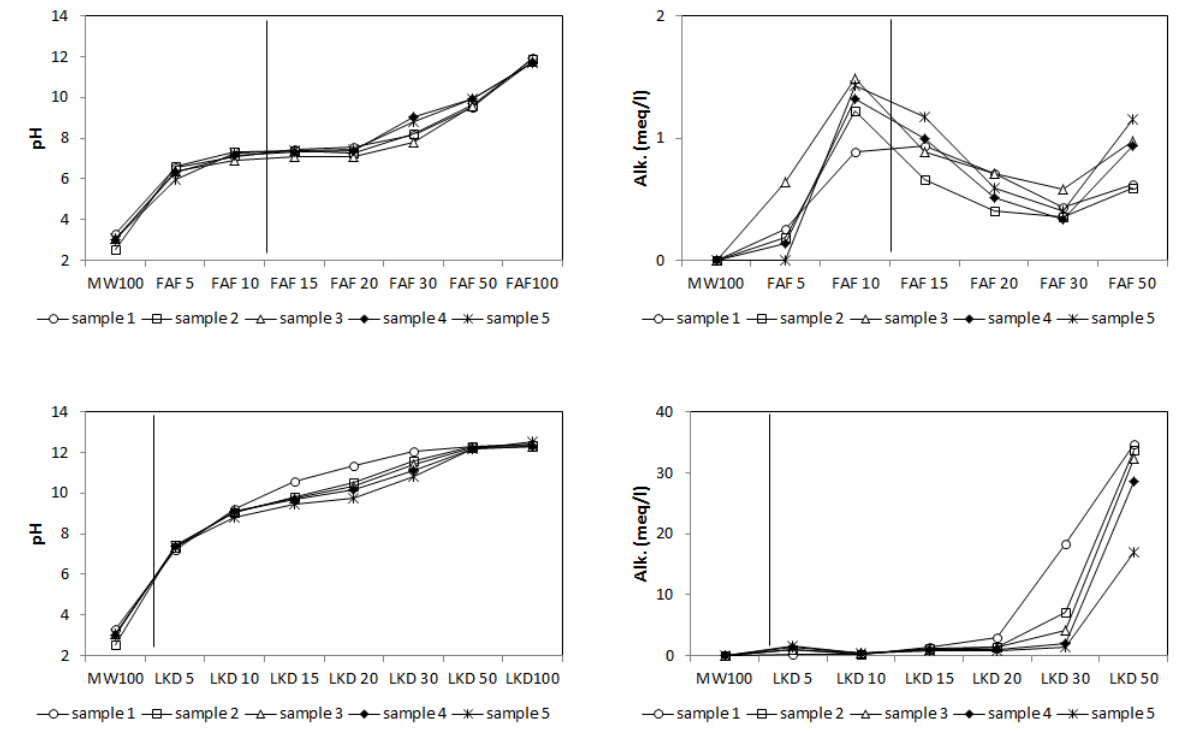

Figure 5.pH(left figures) and alkalinity (right figures) in mixing experiments with waste rock and alkaline materials (FAF and LKDmixtures). Top figures carbonate system, bottom figures oxide/hydroxide system.Total mass in each mixture was 5 grams and the alkaline additions ranged from 0 to 50 % (by weight). MW100 is waste rock without alkali addition (control). Each mixture (in %) was sampled five times (after 1, 3, 8, 28 and 65 days). Solid lines in the diagrams indicate sufficient alkaline addition for neutralization of surface acidity.

Concentrations of lead and zinc from the layered alkali experiments are shown in Figure 6.The average concentration reduction (compared to the reference) of all trace elements (lead, cadmium, copper and zinc) in all test barrels was approximately 96 % after 44 months.

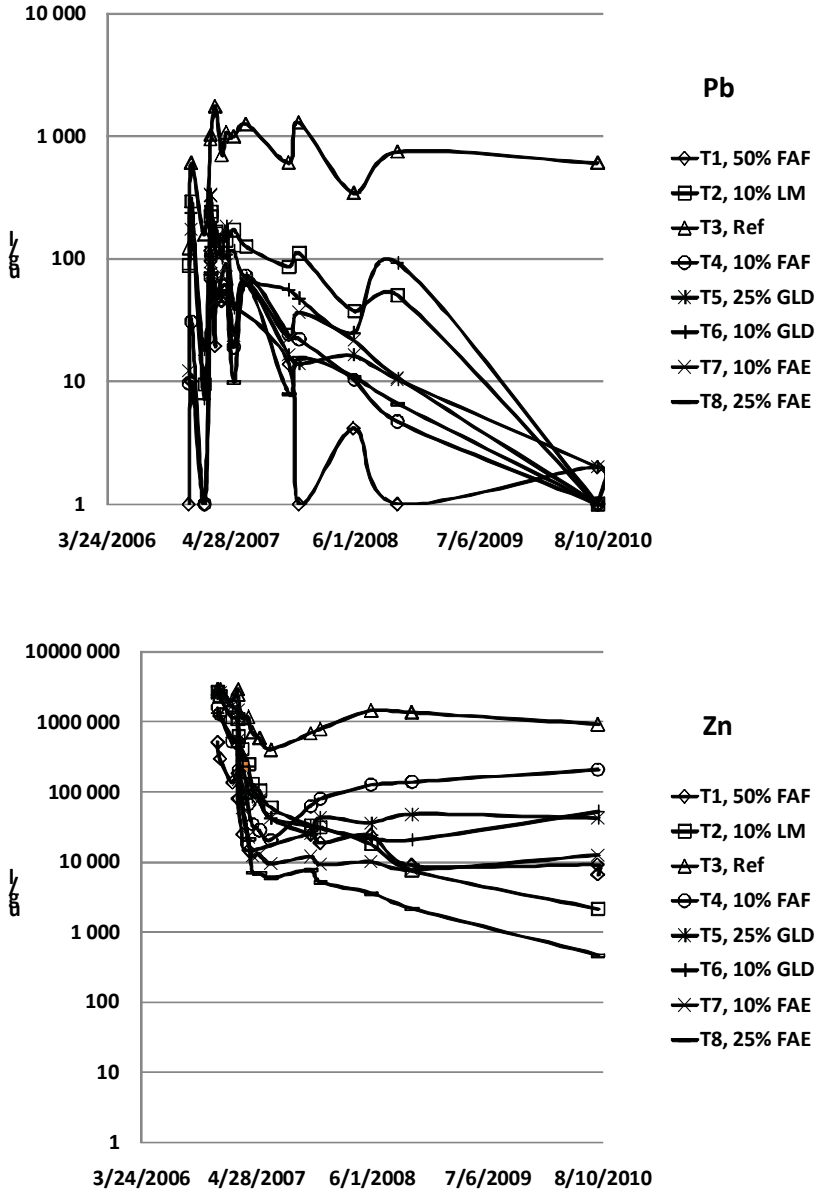

Figure 6.Trace element concentrations in the reference system (T3) and the layered systems.

Three of the test barrels treated with fly ash (T1, T7 and T8) and the test barrel with 10 % lime mud (T2) produced leachates with higher pH and/or lower trace element concentrations than the other test barrels (Table 3). The higher pH in the fly ash test barrels may be caused by longer contact time between the alkaline material and the acid leachate due to hard pan formation. The performance of barrel T4 (10 % FAF) was poor regarding Zn and Cd reduction, despite a higher pH than for example T2 (10 % LM). This may be explained by a rather high concentration of Zn and Cd in the FAF- ash from the beginning.

Table3.pH and reduction (%) in trace element concentrations at the last sampling occasion compared with the reference (T3). Worst performing system for each element is shaded.

	pH	Pb		Zn		Cd		Cu	
		Conc	%	Conc	%	Conc	%	Conc	%
T1	6.68	1.0	99.8	6 450	99.3	21	98.7	6.0	100.0
T2	5.50	1.0	99.8	2 140	99.8	<0.1	100.0	48	99.6
T3	4.44	606	-	918 000	-	1 600	-	13 900	-
T4	5.72	1.0	99.8	208 000	77.3	305	80.9	312	97.8
T5	5.64	2.0	99.7	41 400	95.5	78	95.1	237	98.3
T6	5.89	1.0	99.8	52 200	94.3	90	94.4	383	97.2
T7	5.96	1.0	99.8	12 500	98.6	23	98.6	77	99.4
T8	6.21	1.0	99.8	460	99.9	3.0	99.8	19	99.9

Treatment experiments

Figure 7 shows the pH and alkalinity measured in the sequential batch experiments. A large portion of the available alkalinity from the alkaline materials was flushed out of the mixtures before it could be utilized as a buffering agent. Around cycle 14 the LKD mixture alkalinity increased after dropping two orders of magnitude from the starting value. This was most likely due to the dissolution of calcite in the LKD. A similar increase in alkalinity was not observed in the LD mixture, the result of minimal calcite present in LD. The LKD mixture maintains alkalinity and pH above 7 throughout the experiment (L/S=1000) indicating a significant buffering capacity.

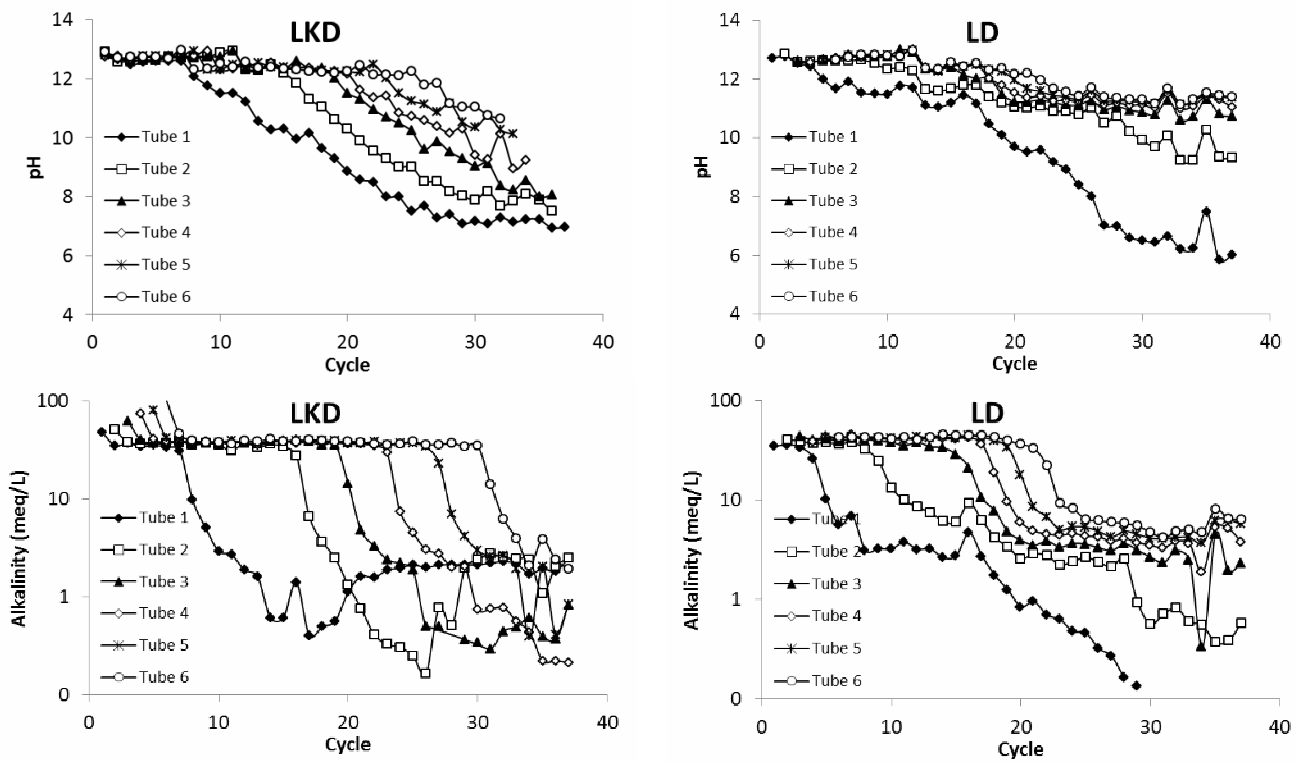

Figure 7.pH and alkalinity for LKD (left) and LD (right) in all six tubes from the sequential batch experiments.

The already immobilized trace elements from the added ARD started to be remobilized around pH 8. Concentrations increased rapidly below this pH, mainly due to desorption, and was soon found to be

higher than in the added ARD (Figure 8). This means that some of the trace elements were released also from the LD-material.

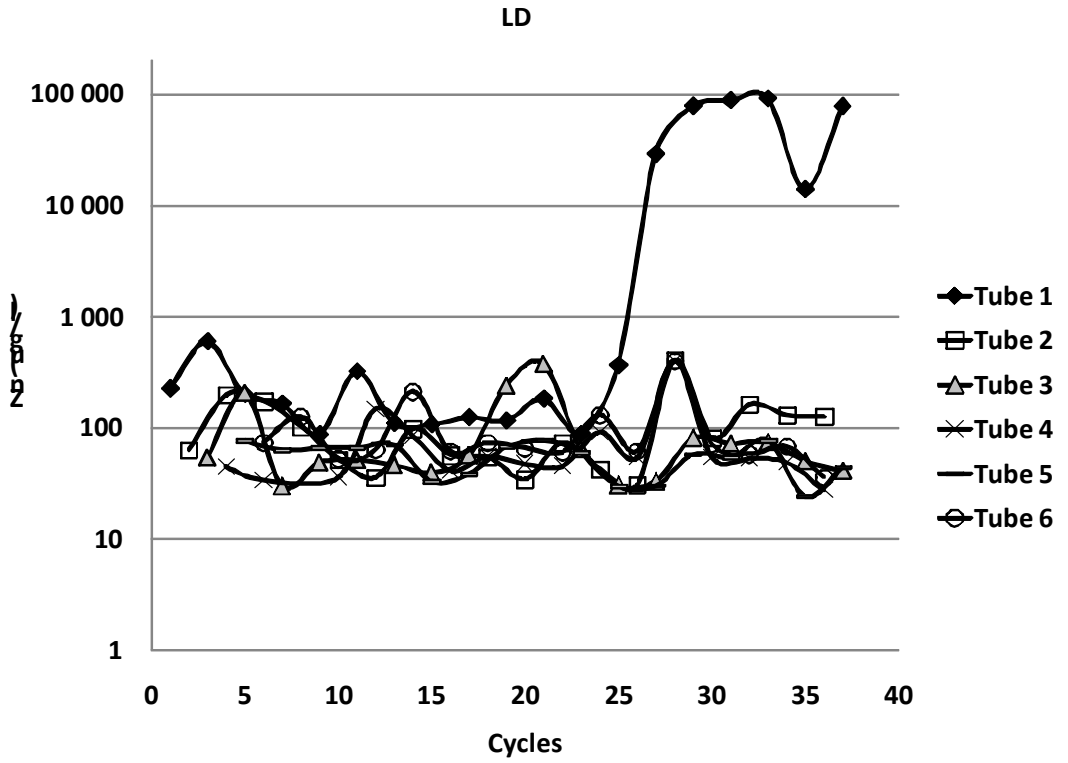

Figure 8.Release of zinc at cycle 20 (pH 8.5) from LD in the sequential batch experiment. Zinc concentration in the added ARD was 33 mg/L.

The outdoor sequential filter lines experiments were run in 2008, 2009 and 2010. The total buffering capacity had only minor influence on the overall performance of the filter lines. Other chemical and physical parameters were more important, namely passivation of the alkaline material surfaces by coatings of fresh precipitates (c.f. Cravotta III 2008) and carbonation of the oxides in fly ashes and lime kiln dust (Pérez-López*et al.* 2010). Also kinetic constraints could limit the efficiency of the carbonate materials (e.g. GLD, LM/FAF and WWG), due to slow dissolution rates (Cravotta III and Trahan 1999). Table 4 shows median pH-values achieved during the years 2008-2010.

Table 4.pH (median values) in filter sections 2008-2010.

Filter	**F1**	**F2**	**F3**	**F4**	**F5**	**F6**	**F7**	**F8**
Reactor	3.43	3.43	3.43	3.41	3.41	3.41	4.07	4.07
Section 1	5.40	4.11	4.68	10.8	5.14	8.88	6.95	6.43
Section 2	6.49	3.64	4.39	10.5	5.56	8.82	7.61	6.47
Section 3	4.82	4.18	4.28	5.17	5.05	6.16	5.99	5.81

The amount of trace elements removed from solution varied widely between filter lines. Zinc concentrations measured after the first barrel in the filter lines are shown in Figure 9 as a representative element. The best overall trace element reduction was obtained in the FAF filter during the first year. Despite a rather low pH (Table 4), a significant amount of iron and aluminum precipitation occurred with the associated sorption and/or coprecipitation of the trace elements. However, the overall performance of the FAF filter did decrease with ARD loading, either due to carbonation of the fly ash or armouring of neutralizing surfaces by ferric and aluminum hydroxides.

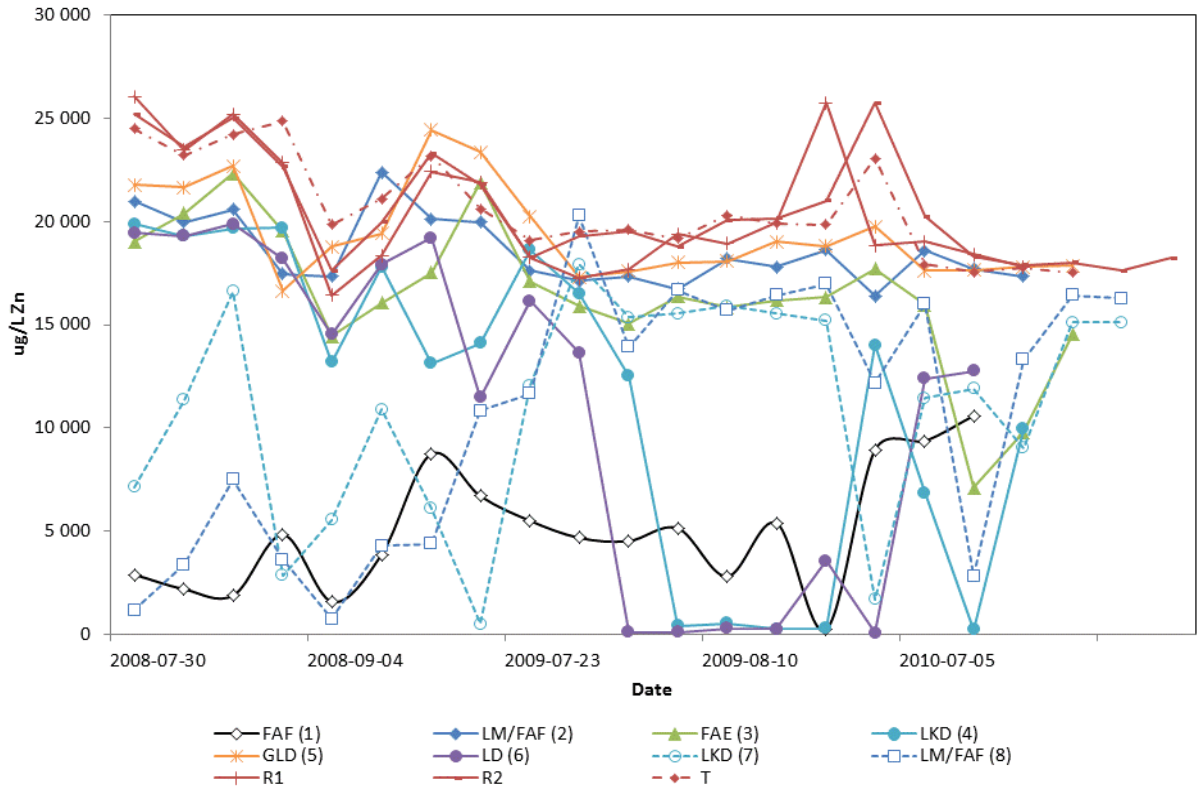

Figure 9.Zinc concentrations (in µg/l) in the first barrel (containing alkaline material) of the filter lines and in the ARD-reactors (R1, R2) and ARD-tank (T). Filter lines 1-6 receive water from R1 and R2, and filter lines 7-8 receive water from T (see Table 2).

The filter lines with a combination of fly ash and lime mud (F2: LM/FAF) and with carbonated fly ash (F3: FAE)had a similar chemical response. These filter lines gave the poorest immobilization of trace metals. The limited neutralization in these lines lead to low pH and hence resulted in a modest formation of sorbent phases of fresh iron and aluminum precipitates. This explains the low trace element reduction. In summary, filters with hydroxide materials (LKD, LD) were superior to filters with carbonate materials.

In July 2010, new chemical conditions were induced in filter lines F2 (LM/FAF) and F3 (FAE) because of their low performance. About 1/3 of the material in the first barrel of the filter line was replaced with horse manure. This was done to create more reducing conditions, which would enhance the neutralization and stabilization of Fe(II)(aq). It was also expected that the reducing conditions would induce reductive dissolution of the ferric precipitates that were believed to coat the surfaces of the alkaline materials. Preliminary results indicate that the manure had the desired effect. The pH increased from 3 to above 5.5.

Conclusions

It has been demonstrated that there are several alkaline by-products from various industrial processes that can be used as amendments to historic (sulphidic) waste rock, and as components in chemical barriers (filters) for the

- *Prevention* of the onset of acidic drainage and subsequent releases of trace metals associated with the sulphide phases, as well as the
- *Treatment* of metal-rich acidic leachates and effluents from waste rock heaps and mining areas.

The most important parameter is pH – it determines the weathering process, as well as the mobility and distribution of trace metals, and the formation of precipitates (primarily iron and aluminum), which serve as sorption and co-precipitation agents. The choice of alkaline neutralizing agent is crucial, taking into consideration capacity and pH-buffering ability (as well as alkalinity source – hydroxide or carbonate).Controlling the chemical state of iron is also of crucial importance.

The following lists conclusions regarding specific processes:

- Alkaline by-products like fly ash, lime mud and lime kiln dust can be used in both prevention of ARD as an amendment to waste rock, and in treatment of ARD in engineered filters and barriers.
- The proportion of hydroxide material to waste rock must be high enough to provide sufficient neutralizing capacity, but low enough to prevent dissolution of metal hydroxidesat high pH. The proportion of carbonate to waste rock in amendments is not as crucial (in the short term perspective).
- A significant fraction of the alkalinity is washed out from filters using solely hydroxides as an alkaline additive (e.g. LD and LKD). This reduces the effective neutralizing capacity.
- Carbonated fly ash and mixtures of fly ash and carbonate material are not suitable for treatment of iron-rich ARD because of passivation by surface coatings of iron and aluminum.
- Iron-rich ARD is preferably treated with filters containing hydroxide materials.

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