Phosphate Mining Waste Reduces Microbial Oxidation of Sulphidic Minerals: A Proposed Mechanism

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Abstract

A critical detailed review of the use of phosphate-containing materials to reduce sulphide oxidation revealed that the proposed iron-phosphate coating is short-lived and thus the application had been dismissed. We have field tested below grade natural phosphate rock (NPR) from the Aurora Mine in North Carolina since 1992 and have documented encouraging results due to the formation of a biofilm integrating inorganic precipitates. This biofilm, which is not the previously proposed iron-phosphate coating, led to a decrease in oxidation. The NPR is carried throughout the drums by rain and snowmelt and is lodged onto the mineral surfaces, which are often iron stained similar to the iron staining on the sulphidic waste rock. Leaching the NPR with 0.1N H_2SO_4 or distilled water revealed that water released mainly calcium, but the sulphidic acids released all major and minor nutrients needed for growth of biota. Integrating the findings of the acid leaching experiments and the physical distribution of the NPR particles in the waste rock drums, it is suggested that the elements released from an NPR particle along with the neutralization they provide induces an environmental change on the mineral surface, which reduces microbial oxidation as heterotrophic biofilm growth is initiated. The hypothesis has yet to be tested in the field and the laboratory.

Keywords: Biogeochemistry, biofilm, phosphate, mining wastes, sustainability, decommissioning, sulphidic oxidation, microbes

Introduction

In the nineteen eighties, experiments to reduce sulphide oxidation on pyritic waste rock using industrial phosphate wastes were carried out in the field and laboratory with varying degrees of success (Meek 1983, Renton 1988 a,b). The noted reductions in sulphide oxidation were thought to be the result of the formation of iron-phosphate (Evangelou 1994), phosphate oxides (Nicholson *et al.* 1990) or phosphate hydroxide-silica (Zhang and Evangelou 1998) coatings over the rock surfaces. Further reductions in oxidation rate were thought to be due to the removal of ferric iron (a strong leaching agent) and the reduction of oxygen access to the rock surface (Meek. 1991, Belzile *et al.* 1997, Hart *et al.* 1990 and Hart and Stiller 1991, Evangelou 1995, Ziemkiewicz 1990). The phosphate and ferric iron reportedly resulted in the formation of vivianite ($Fe_3(PO_4)_2.8H_2O$) and strengite ($FePO_4.2H_2O$), respectively. Ziemkiewicz (1990) reported a physical isolation of the pyrite surface. In 2003, Jones *et al.* refuted the effectiveness of an iron phosphate coating and confirmed the presence of microbes underneath the coating on arsenopyrite. In discussing the implications of these findings for remediation he stated that phosphate additions could be detrimental as they might stimulate growth of sulphide-oxidizing bacteria in a phosphate-limited environment. However, other microbes and fungi will also be stimulated to grow and potentially outcompete the slow-growing sulphide oxidizing group. In 2005, Mudder *et al.* summarized a 5 year laboratory and field study in collaboration with several large mining companies testing thiocyanate

as a biocide together with phosphate rock and commercial fertilizers. They proposed a combined biotic–abiotic treatment for reducing sulphide oxidation. Their laboratory and field tests were initially successful, but failed in the longer run. Similar results were reported by Mauric and Lottermoser (2011), testing local phosphate rock and commercial fertilizer. They concluded that phosphate was not effective in reducing sulphide oxidation in the field.

We reviewed in detail more of the published work on oxidation reduction, including many phosphate-containing materials, but also phosphate ore from the Aurora mine in North Carolina U.S.A. Why did we not find the expected iron phosphate coating, but an organic coating? A coating was formed when water soluble phosphate fertilizer was added, but mostly in laboratory experiments. Reductions in oxidation rates were reported when natural phosphate rock (NPR) from the Aurora Mine was added in outdoor coal waste rock piles and base metal mining wastes (Meek 1983, Renton 1988a,b, and Mudder *et al.* 2005). The fertilizer worked briefly in outdoor experiments, but due to its water solubility, it was likely easily washed out. The prevailing expectation was that an iron-phosphate coating had to be formed, given probable inorganic chemical reactions. When such a coating was not found, the experiments were terminated.

The involvement of biofilms in the corrosion process is well documented (Characklis and Wilderer 1989), but the similarity between corrosion and acid-generating processes was not widely recognized, nor was the idea of the formation of biofilms on rocks accepted. The presence and activities of microbes in the acid-generating process was ignored along with the other chemical and physical actions of phosphate in the interpretation of the results. The importance of microbial involvement is easily missed, especially when short-term experiments (less than one whole year) are performed, as microbial effects on the acid-generating process take longer to establish than effects of first order chemical reactions. Further, iron phosphate coatings formed when water soluble phosphate fertilizers or phosphate-rich media were used. NPR, on the other hand, is very weakly water soluble, but dissolves readily in acid. So, the conclusions of Meek (1983), Renton (1988a,b) and Mudder *et al.* (2005) were correct, based on the prevailing hypothesis. VNPR did not lead to the formation of a phosphate iron coating over the sulphide minerals, but sulphide oxidation inhibitory effects were evident particularly in field test(s). Thus, the evaluation criteria were not appropriate for this amendment.

We also added phosphate mining wastes to various sulphidic mining wastes with effluents low in acidity with circum-neutral pH values (Kalin and Harris 2005). However, we could not find the expected coating which would have reduced the oxidation of sulphides, when surfaces of rocks from the experiment were examined. Instead, with SEM/EDX, we found an organic cover or a biofilm, the dimensions and coverage of which remain undefined. After investigating many rock surfaces, only traces of phosphate were identified. In further studies, the rocks from the experiment were immersed in growth media, which revived and grew the persistent biofilms (Ueshima *et al.* 2003 and 2004). All rocks from the experiment had been stored indoors while the investigation of the coatings by Ueshima *et al.* (2003 and 2004) took place at the University of Ottawa. Having successfully revived the biofilm on the mineral surface, we re-exposed the rocks for one more complete season outdoors without further NPR addition. Initially, for a brief period, acid effluent was generated, but the pH improved to a neutral value after about 2 months (Kalin and Harris 2005).

The persistence of the organic covering was extraordinary and stimulated the interest of R. Smart, leader of a group of scientists working for AMIRA International at the University of South Australia performing an evaluation of ARD passivation treatments. Rocks from the first outdoor exposure, now stored for 11 years in buckets outdoors, exposed to heat and freezing, were investigated as part of the AMIRA project. The presence of an organic coating containing mainly inorganic iron precipitates but only traces of iron-phosphate was confirmed on the mineral surfaces. These findings lead to a re-examination of all stored rock surfaces of the experiment. Rocks exposed outdoors with and without NPR between 1992 and 1995

(first exposure), as well as those stored indoors until 2000 and re-exposed outdoors without NPR were re-examined by SEM/EDX. In addition, outdoor control rocks (no NPR) and indoor control rocks (kept in sterile bags immediately after collection from the mine site in Northern Quebec) were examined. Organic coatings were still present on rocks exposed to NPR, albeit the coatings were changed in appearance. No traces of an organic coating could be found on either type of control, indoor or outdoor (Kalin *et al*. 2010).

Our field experiments with NPR approached the sulphide oxidation reduction problem from a practical, pragmatic and empirical perspective. This approach is called for given the complexity of phosphate chemistry in the presence of microbial activity. Further complexities stem from the large gap between the bio-geochemical processes on the microscopic scale *i.e*. nanometers to micrometers, and those processes which take place on the scale of the waste rock dump. These scale-up problems are often a major hurdle to success when promising results are tested in the field.

The experiments with NPR (from the same mine in North Carolina) were also applied to sulphidic tailings, ranging in sulphide content from 1 to 90% and waste rocks with sulphides up to 15 %. Only after we had documented inhibitory effects on the field samples, did we proceed to investigate the process, which was in disagreement with the previously postulated iron-phosphate coating. We had used a dosage of NPR that was based on economic considerations, dictated by the mining operation which supplied the waste rocks, not on a stoichiometric chemical reaction basis. We waited 3.1-3.75 years before we sampled the tailings test plots and collected effluent from the waste rock drums for 2.7 years before dismantling them. These very long outdoor exposures provided adequate time for chemical and microbial oxidation reactions to take place and any short-lived inhibitory effect of neutralisation to diminish. We obtained further confirmation of the biofilm's existence, and persistence (Kalin *et al.* 2009 and 2010) when we re-exposed rocks outdoors without NPR for a year, which should have destroyed the coating.

The next step was to characterize the NPR, by focusing on properties of the material and its distribution within the drums. The objective of this work is to present the relevant characteristics of the NPR used in all the field tests and address those characteristics in light of a possible contribution to biofilm initiation and growth.

Materials and Methods

Ontario phosphate mine material

Mine tailings and low grade ore from the Agrium phosphate mine in Kapuskasing (Ontario) were included in our study to compare their characteristics with those of the NPR mining wastes of the Aurora Mine in North Carolina. Large samples (up to 10 kg) received from the Ontario mines were sub-sampled by the quarter method after intensive mixing. This material was included here only because it was considered for use in future field tests.

NPR application method

The NPR used in these experiments was below-grade phosphate rock from the North Carolina Aurora phosphate mine, which was washed free of clay particles prior to shipment. A one time application of 5 L or 8.2 kg of the NPR gravel (>0.04 but <4 mm) was dumped onto the top of the 70 L drums containing about 61-79 kg of waste rocks ranging in size from 0.01 to 0.25 m diameter. A total of 14 drums were set up: 5 controls, 8 NPR variations, and 1 drum which remained covered. At the end of the experiment each drum was divided into 5 layers (top = 1, bottom = 5) and the distribution of NPR throughout the drums was recorded photographically for each layer. The remaining NPR was collected and weighed. Details of the mining wastes used are presented in Kalin and Harris (2005).

Fractionation
The particle fractionation of all materials was carried out using Taylor sieves with equipment available at Queens University and University of Windsor. Fractions were air-dried and weighed.

Elemental composition
Fractionated and un-fractionated NPR samples were air-dried, digested in nitric/perchloric acid, and sent to a certified laboratory (Saskatchewan Research Council, Saskatoon) for ICP analysis.

Leaching by decanting
The phosphate-containing materials were leached in distilled water and in 0.1 N sulphuric acid. The mixture was stirred on a magnetic stirrer for up to 1 min in 100 mL of acid with 10 g of material allowed to settle for a minimum of 0.5 h at which point the leachate was decanted. The total reaction time was increased after the 3rd decanting to up to 17 h. A stable, but increased pH value in the leachate after addition of the acid (pH 1.4) was used to signal the decanting. The experiment was terminated when the pH no longer increased in the supernatant. The electrical conductivity, pH and acidity values were determined in the leachate for each decanted cycle, the last determination allowed for a reaction time of 642 h before decanting. These data are not shown.

Instrumentation and Measurements
A Sauter top-loading balance, with a precision of 0.01 g, was used to weigh samples. The electrical conductivity and temperature were determined with an Oakton Con 400 series EC, which adjusted the EC measurement automatically to 25°C. A Corning 315 pH/Ion meter equipped with a Corning #33221-034 combination electrode was used, calibrating between pH 4 and 7. A Corning M103 redox inert platinum electrode with a standard Calomel probe was used to determine the measured redox potential (E_m). E_m was converted to standard redox potential at 25°C (E_H) by using the following formula: $E_H = E_m + (241 - (0.66*(\text{Sample Temperature in } ^oC - 25)))$. All measurements were obtained in 15 mL of supernatant collected in the decanted leachate each time up to the 8th and final decanting. Acidities were determined using a 702 SM Titrino (Brinkmann; Metrohm). Alkalinities were determined for the supernatant of the distilled water extractions.

Results and Discussion
The distribution of the NPR within the waste rock drums was documented with photographs as the drums were dismantled at the end of the experiment. Figure 1a shows the top layer (1) rocks in a control (no NPR) drum and 1b the layer below. Figure 1c is the top layer of a drum which received NPR and 1d is the bottom layer of the same drum. It is important to note that after 2.7 years outdoors in Toronto, NPR gravel was no longer visible on the surface of the drums, the gravel had all been dispersed throughout the drum. All layers below showed unevenly distributed NPR particles lodged onto the rock surfaces. Often both rock surfaces and NPR particles were iron stained (Figures 1b, 1c and 1d). This suggests that acid was generated, *i.e.* oxidation took place and acid did contact the NPR particles. As it is reasonable to assume that the NPR particles made the same path through the drums as rain and snow melt, it follows that the water acted as the sole agent to transport particles. The drums were neither shaken nor moved for the period of the experiment. Hence, the water path through the drums was determined by the physical rock arrangements and by the intensity and direction of the rain. The acid generation was not only evidenced by the acidity of the effluent from the untreated drums (Kalin and Harris 2005), but also by the iron staining of the rocks and on the NPR gravel (Figure 1).

On dismantling of the drums, it became evident that only 50 to 61% of the added NPR had reacted. It was therefore relevant to determine the particle size distribution and the elemental content of the NPR and any future test material such as phosphate-containing tailings from the Agrium phosphate mine in north Ontario. Smaller particle sizes should have greater mobility within the drums and could have different elemental compositions. The particle size distribution of the NPR and the elemental content of each size

fraction are given in Table 1. Particles of NPR with a diameter between 6 and 2 mm (fine gravel) constituted 45% , those between 2 and 0.6 mm (coarse sand) constituted 52% of the mass, and the remaining 3% of the total NPR were finer grained, classified as medium to fine sand or silt. The tailings particles of the Agrium phosphate mine were nearly completely fine sand or silt particles, whereas Agrium ore particles adhered to the sieve, preventing accurate particle fractionation with the available methodology.

The elements determined in the solid material were grouped in Table 1 to reflect their importance in relation to the growth of biota. The major nutrients are Ca, P, Fe, Mg and K and the micro-nutrients or co-factors for growth are Zn, Mn, Cu, Mo, Pb, Co. Minor micro-nutrients were also present in very low concentrations and are not listed. For NPR, the major constituents are Ca and P, and they are distributed throughout all particle sizes, although decreasing in concentration in fine sand and silt.

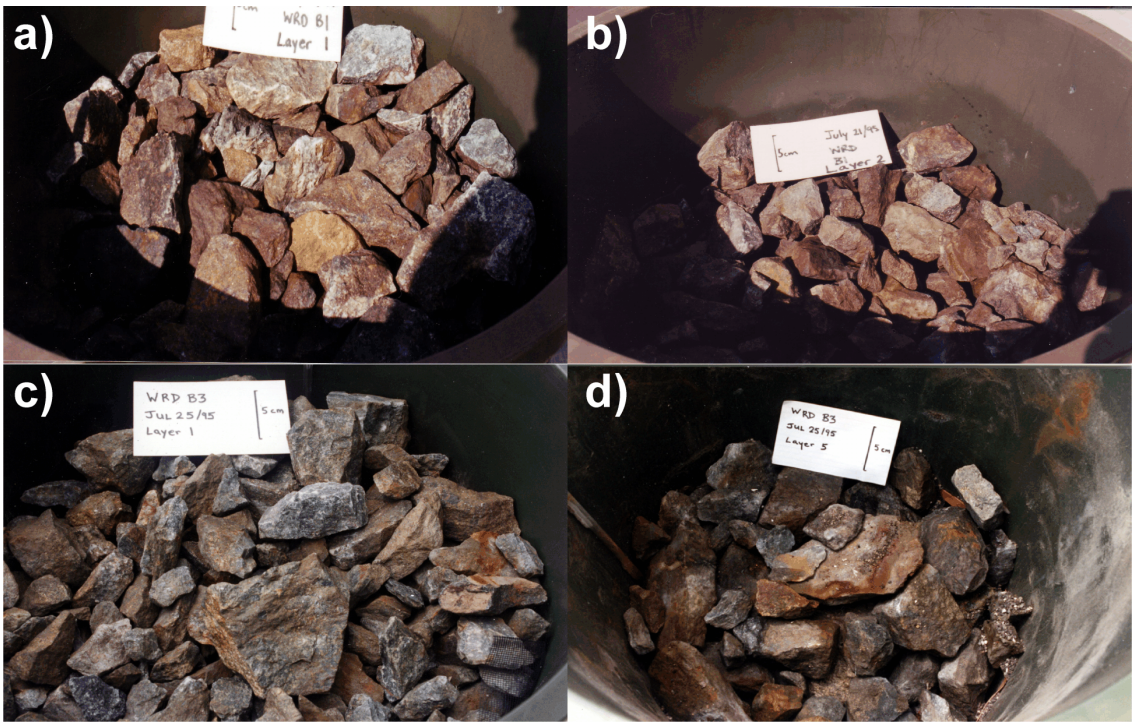

Figure 1: a) Level 1 of control no NPR waste rock drum. b) Level 2 of control waste rock drum; c) level 1 of NPR waste rock drum; d) Level 5 or bottom of NPR waste rock drum.

The results of the decant experiment are presented in Table 2, where we leached 10 g of NPR with 0.1N sulphuric acid and distilled water. The cumulated supernatant from 8 decant cycles (0-8) showed that, with the exception of Pb, all major nutrients are released when NPR is stirred (stirring time 0.02 to 1 h, cumulative total = 6 h) and allowed to settle (settling time 0.5 to 1.0, last decant 642 h, cumulative settling time = 681 h). This 'stirred' scenario simulated a brief, initial contact with a waste rock surface, and then becoming stuck in a sulphuric acid environment. This scenario is contrasted to a 'stagnant' solution' in which 10 g of NPR was not stirred, but remained in acid for 10 days or 1486 h. In the first scenario, water was 'washed' through the NPR particles, while in the second scenario the particles remained near the waste rock for extended time periods. Under acidic conditions, it is evident that the elemental concentrations of the 'stirred' and 'still' leaching solutions, were similar, releasing equivalent concentrations of each element (Table 2). This is contrasted to leaching in distilled and rain water, where the only similarity is in the leaching of calcium. All other elements are significantly lower, usually by

Table 1: Particle size distribution and elemental content of NPR (mg/g)

	Ca	P	Fe	Mg	K	Zn	Mn	Cu	Mo	Pb	Co
Fine Gravel 45%	68400	24400	1550	600	400	15.7	4.49	1.84	2.47	0.45	0.11
Coarse Sand 52%	37400	13300	1230	650	200	8.74	3.05	1	1.39	0.27	0.07
Medium Sand 2%	1600	433	300	0.2	0.08	1.1	1.02	1.55	0.04	0.04	0.01
Fine Sand 0.7%	533	200	100	0.23	0.12	0.41	0.36	0.49	0.02	0.02	0
Silt 0.3%	150	3	4.3	1	0.16	0.24	0.16	0.84	0.01	0.02	0
Un Fractionated	304000	47100	5900	3630	158	388	28.6	29.7	n.a.	n.a.	n.a.
Agrium Tailings	275000	112000	125000	920	190	260	1780	17	>0.5.	35	83
Agrium high Grade ore	256000	93800	44300	1600	60	340	610	64	1.4.	25	140

n.a. = not analyzed. Agrium tailings: fine sand 95 %, silt 5 % Agrium ore: Fine gravel 8%, coarse –medium sand 36 %, fine sand 56 %

several orders of magnitude. Along with the outdoor waste rock drums, a drum containing only NPR was also run. Effluent was collected, analyzed by ICP and compared to the rain water which was collected in a nearby rain gauge (Table 2). Only calcium seemed to dissolve in the NPR drum. All other elements were found only in trace amounts. Since the NPR from N. Carolina is mined from a marine sedimentary phosphate deposit, we expected the carbonates to leach.

Table 2: Release of elements from NPR in sulphuric acid and distilled water.

	mg/L	**Stirred H_2SO_4**	**Still H_2SO_4**	**Still DH_2O**	**NPR Drum rain**	**Rain gauge**
Major Nutrients	Ca	620	680	510	33.6	6.65
	P	400	490	9.8	0.09	>0.06
	Fe	5.37	11.9	0.21	0.043	0.052
	Mg	18	31	0.6	1.6	1.23
	K	3.7	8.9	0.7	-4	8
Co-Factors	Zn	0.25	0.5	0.026	0.075	0.04
	Mn	0.094	0.16	0.003	>0.005	0.015
	Cu	0.017	0.09	>0.001	0.022	>0.003
	Mo	0.015	0.029	0.018	>0.01	>0.01
	Pb	>0.002	>0.002	>0.002	>0.025	>0.025
	Co	1.58	0.035	-0.001	>0.005	<0.005

Conclusion

The dismantling of the drums has shown that NPR particles are distributed throughout the drums. There is no difference in the elemental composition of the larger and smaller particles. Major nutrients and co-factors are released from NPR gravel under acidic conditions, but not released to any degree in distilled water. Rain water in contact with NPR alone contains higher concentrations of Ca but all other elements are similar to rain collected in a rain gauge. Therefore, NPR gravel in contact with the oxidizing mineral surface, probably provides sufficient nutrients for the stimulation of biofilm growth.

Bacterial oxidation has been shown to dramatically increase the oxidation of pyrite by as much as 10^6 times (Singer and Stumm 1970). It is also well-documented that most free-floating microbes *i.e.* those which would be present in rain water, possess the ability of 'quorum sensing'. Quorum sensing triggers normally motile, free-floating organisms to settle onto surfaces and form biofilms (Ruiz *et al*. 2008). Quorum sensing has been linked to specific molecules released by microbes when environmental conditions change (Ruiz *et al*. 2008). Hence, environmental change would occur when an NPR particle is lodged onto the oxidizing mineral surface. This may, in turn, induce biofilm formation or alter the monolayer biofilm of the oxidizing microbes (Harneit *et al.* 2006). Oxidizing bacteria and the oxidation of pyrite require an acidic interface (Mielke *et al.* 2003). This interface might be neutralized by the NPR, stressing the existing oxidizing microbial community, making some of their organic carbon and extracellular polymers available for heterotrophic microbe growth.

We propose that particles of the NPR (or other material with similar elemental composition like Agrium tailings) added to the mining wastes are carried with the rain to the oxidizing sulphides where they induce change through neutralization and the release of essential nutrients. This might lead to the production of the observed organic biofilm that restricts oxidation of the pyrite in our NPR-treated sulphidic wastes. These biofilms have been shown to be persistent over long periods of time, surviving desiccation, re-wetting, and extreme temperatures. The extensive literature on biofilm formation and development further supports our idea that beneficial biofilms can be formed and developed on sulphidic wastes. In light of the economic and environmental costs associated with sulphide oxidation, this idea may lead to a potential large-scale reduction in AMD and ARD. It is certainly worth testing both in the field and laboratory.

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Acronyms

AMIRA	Australian Mining Industry Research Association
AMD	Acid Mine Drainage
ARD	Acid Rock Drainage
ICP	Inductively-coupled Plasma Spectroscopy
NPR	Natural Phosphate Rock
SEM/EDX	Scanning Electron Microscopy/ Energy-dispersive X-ray spectroscopy