

METAL BINDING, ACID NEUTRALIZATION AND MINIMIZATION OF SULFIDE OXIDATION IN DREDGE SPOIL MATERIAL USING VIROSOIL™ TECHNOLOGY

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ABSTRACT

Material removed during dredging at ports typically is characterized by high sulfide concentrations, high metal loadings from upstream industrial activities, or both. Sulfide oxidation associated with sub-aerial disposal commonly produces acidic pore water; the acidity mobilizes metals that may have serious ecological impacts. When this water leaves dredge spoil disposal areas, it is hazardous to fish and other aquatic organisms. The acid conditions also adversely affect plant growth during disposal site restoration and damage engineered containment structures. ViroSoil™ Technology can be used to alter the geochemistry of dredged material to reduce sulfide oxidation rates, neutralize acidity, and precipitate heavy metals, resulting in a soil capable of sustaining plant growth and runoff that is not toxic to aquatic biota.

ViroSoil™ Technology incorporates ViroBind™ reagent, which has a high metal binding and acid neutralizing capacity and good phosphate retention for plant growth; the reagent also promotes healthy populations of soil micro-organisms. The reagent is extremely fine grained (>90% is < 10 µm) and has a very high reactive surface area but is largely insoluble and non-dispersive. This makes it ideal for blending with suction dredge discharge, or during transfer from clamshell dredging. ViroBind™ reagent is ideal for treating sulfidic dredge spoil because it not only neutralizes acidity, but also greatly reduces sulfide oxidation rates. The reagent can also be formed into granules or pellets, for use in permeable reactive barriers, or enclosed in gabions, constructed around dredge spoil disposal sites. Analysis of ViroBind™ treated material shows that metals are bound into non-leachable crystalline forms. The metal-binding strength increases with time and plants growing on treated material and earthworms living in it do not exhibit increased metal uptake.

This paper presents dredged material treatment case histories, and suggests alternative methods for non-toxic upland disposal, or beneficial use of the material.

Keywords: Contaminated sediments, beneficial uses, pollution prevention, heavy metals, sulfide oxidation

INTRODUCTION

Material removed during maintenance and infrastructure dredging at ports typically is characterized by high concentrations of sulfide minerals resulting from microbial sulfate reduction in anaerobic sediment, high trace metal loadings from upstream industrial activities, or both. Moreover, a high proportion (often all) of the sulfide minerals are likely to be fine, poorly crystalline black iron monosulfides (e.g. hydrotroilite, greigite/melnikovite, mackinawite) that are much less stable than common well crystalline metal sulfides such as pyrite. As a result, these black iron monosulfides are highly reactive and substantial quantities can oxidize within minutes or hours of exposure to atmospheric oxygen. In contrast, more crystalline metal sulfides usually take months or years to oxidize to any great extent under natural conditions, even if the pH becomes low enough (less than about 3.0) to allow oxidation by the ferric iron pathway, or if they become infected with sulfur oxidizing bacteria (e.g. *thiobacillus ferrooxidans*). Consequently, sulfidic marine or harbour sediment exposed to atmospheric oxygen can potentially

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generate metal-contaminated acidic leachates much more quickly than the more coarsely crystalline metal sulfides that are responsible for the widely recognized problems associated with acid rock drainage.

Marine and harbour sediments often also contain calcium carbonate (calcite or aragonite) in the form of shell fragments or foraminiferal tests that could potentially neutralize acid produced as a consequence of sub-aerial exposure of the sulfidic sediment and, for some sediments, the available acid neutralizing capacity can equal or exceed the acid generation potential. However, many sediments have little or no acid neutralizing capacity and even those that do have a high carbonate mineral content may not be able to prevent the production of metal contaminated acidic leachates. There are several possible reasons why acid neutralization may be insufficient, even where the carbonate mineral concentration is high: a) the sulfides and the potentially acid neutralizing carbonate minerals in the sediment may be irregularly distributed (e.g. in mineralogically distinct layers) such that acid generated when the sulfides oxidize may leach out without ever coming in contact with the carbonate minerals; b) the rate of acid generation may exceed the rate at which it can be neutralized by the available (often coarser grained and hence, less reactive) carbonate minerals; c) coarse carbonate mineral particles often become coated by gypsum and iron- and aluminium-oxyhydroxide precipitates, which form as a result of some acid neutralization, and thus become unavailable to neutralize any additional acid that may be produced; d) under certain geochemical conditions bicarbonate ions produced by partial reaction of carbonate minerals can catalyse the decomposition of sulfide minerals thereby accelerating acid generation; or e) a combination of these processes may be operating (e.g. Evangelou, 1995). For these reasons, even if a sediment has an acid neutralizing capacity that exceeds its acid generation potential, it may still be able to produce acidic metal-contaminated leachates.

Because these near-shore marine and harbour sediments frequently contain abundant labile metal contaminants, hazardous organic contaminants, or both, there is limited potential for open-water disposal and many countries have legislation that prevents, or strictly limits any dumping in offshore marine environments. This means that most dredge spoil has to be placed in confined disposal facilities (CDF) or used in coastal land reclamation. However, sulfide oxidation associated with sub-aerial disposal commonly produces acidic pore water and leachate (pH values < 3) and the acidity mobilizes metals and metalloids that may have serious ecological impacts. The combination of high acidity, high trace metal and metalloid loads, and low dissolved oxygen concentrations, common in water near dredge spoil disposal areas, is hazardous to fish and other aquatic organisms. Furthermore, the acid and elevated concentrations of elements (e.g. aluminium) adversely affect plant growth and can make it extremely difficult to revegetate former dredge spoil disposal sites and acidic pore fluids can seriously damage engineered containment structures and the foundations of other structures that may be placed on reclaimed land. Clearly, an alternative treatment is needed to reduce acid formation and metal mobilization from dredged material. This paper provides examples where ViroSoil™ Technology and the ViroBind™ reagent have been used to alter the geochemistry of dredged material to inhibit sulfide oxidation, neutralize acidity, and precipitate heavy metals in non-leachable forms, resulting in a soil capable of sustaining plant growth and runoff that is not toxic to aquatic biota.

VIROSOIL™ TECHNOLOGY AND VIROBIND™ REAGENTS

ViroSoil™ Technology incorporates ViroBind™ reagent, which has a high metal binding and acid neutralizing capacity and good phosphate retention for plant growth; the reagent also promotes the development of essential populations of soil micro-organisms. The reagent is extremely fine grained (typically, 90% is < 10 µm in diameter) and has a very high reactive surface area but is largely insoluble and non-dispersive. This makes it ideal for blending with suction dredge discharge during pumping, or mixing into material during transfer from clamshell dredging. ViroBind™ reagent, available as a powder or slurry, treats sulfidic dredge spoil not only by neutralizing acidity, but also by reducing the rate of sulfide oxidation and directly removing dissolved iron and aluminum before these metals undergo hydrolysis, thereby eliminating the additional acid formation. Although the reasons why are not entirely clear at this stage, both field and laboratory studies consistently show that the ViroBind™ reagent does more than just treat any acid generated within the sediment, with the result that much less reagent is required to prevent the formation acidic pore water or leachates than would be expected based on conventional acid-base accounting. The reagent can also be formed into granules or pellets, for use in permeable reactive barriers, or enclosed in gabions, constructed around dredge spoil disposal sites to prevent the escape of any acidic metal-contaminated leachates from untreated or inadequately treated dredge spoil.

Analysis of ViroBind™ -treatment material (Table 1) shows a complex mix of primarily iron and aluminum oxyhydroxides and alumino-hydroxy-carbonates. The iron oxides are mostly in the form of para-magnetic hematite (72%) and super-para-magnetic iron oxides (Clark, 2000). The freshwater washed reagents, used where the presence of soluble salts could cause degradation of ground- or surface-water resources or inhibit site revegetation, contain

much less soluble components such as sodium and chloride ions; almost all sodium and chloride present in the washed material is part of the stable silicate mineral sodalite.

The acid neutralising capacity of the cocktail of minerals in ViroBind™ (about 5 – 7 moles H^+ /kg of reagent at pH \approx 7 and up to 14 moles H^+ /kg of reagent at pH \leq 5) is largely provided by low solubility carbonate, hydroxide and hydroxycarbonate minerals, many of which react very slowly (McConchie *et al.*, 1999, 2000) and, at low pH, by surface adsorption onto iron and aluminium oxides. Hence, over 48 hours are required for reactions to go to completion; tests involving the addition of standardised sulphuric acid to a pH of 7 show that about 40% of the acid neutralising capacity is used in 5 minutes, about 70% in 4 hrs and about 95% is used in 24 hrs (McConchie *et al.*, 1996). The ability of the minerals to trap trace metals is also strongly time dependent and although most of the initial metal trapping is complete within 24 hrs, metal trapping will continue, albeit more slowly, for many months and the longer the material is left the more tightly the metals become bound.

Although the exact mechanisms by which trace metals are trapped when the ViroSoil™ technology is used are still being investigated, presently available data suggest that many trace metals may initially be trapped by adsorption, which is particularly efficient because the mineral assemblage is dominated by particles with a high surface area/volume ratio and a high charge/mass ratio. However, adsorption alone can not explain the metal binding process because the cation exchange capacity of the mineral assemblage is only about 3.7 meq/100g and because when desorption tests (using compulsive exchange reagents) are carried out on residue that has been saturated with metals and aged for 7 days, less than 20% of the metals (less than 5% for As, Cd, Cr, Cu & Zn) can be recovered; recovery decreases as the time of ageing increases. Similarly, TCLP tests involving leaching at a pH of 2.88 are able to remove only a small proportion of the bound metals and this proportion decreases the longer the metal-enriched residue is left after use (Clark *et al.*, In Press). Furthermore, if the metal enriched residue is left to age for a few weeks after use, it develops some new capacity to trap trace metals even if all available metal binding sites were occupied before it was left to age. The marked effect of aging is most clearly demonstrated by the fact that if acidic water with a high iron and aluminium content, and minor amounts of other trace metals, is passed through a bed of ViroBind™ for about twelve months the reagent can bind up to about 20% of its own weight in metals; this could be achieved only if the metals are ultimately bound by crystal growth because otherwise the precipitation of metal hydroxides would have blocked off all fluid flow paths. Consequently, it is clear that elements, which may initially be bound by adsorption, are redistributed during ageing to become structural components of low solubility minerals; part of the time dependence of metal uptake probably reflects the freeing-up of adsorption sites as initially adsorbed metals are redistributed. During treatment using ViroSoil™ technology, trace metals are also removed by direct precipitation and co-precipitation as the metals react with minerals in the treating reagent to form low solubility precipitates (particularly sulphates, hydroxysulphates, carbonates, hydroxides and hydroxycarbonates). There is also evidence to show that some elements (particularly arsenic, cadmium and copper) can undergo solid-state diffusion into minerals in the ViroBind™ reagent (c.f. Axe & Anderson, 1997, 1998). Because the metal-binding strength increases with time, plants growing on treated material and organisms living in it do not exhibit increased metal uptake (e.g. Maddocks *et al.*, 2003, 2005).

Table 1
Mineral composition of typical ViroBind™.

Mineral	(%) Unwashed			(%) Washed ¹		
	Min	Max	Mean	Min	Max	Mean
Iron oxides & oxyhydroxides	30	35	31.6	30	35	33.2
Hydrated alumina	15	20	17.9	15	20	18.1
Sodalite	15	20	17.3	15	20	17.8
Quartz	5	8	6.8	5	8	7.0
Cancrinite	5	8	6.5	5	8	6.5
Titanium oxides	4	6	4.9	4	6	5.0
Ca(Al) hydroxides & hydroxycarbonates	4	6	4.5	4	6	4.6
Mg(Al) hydroxides & hydroxycarbonates	3	5	3.8	3	5	3.9
Calcium carbonates	2	4	2.3	2	4	2.2
Halite	2	3	2.7	0	0.1	0.03
Others ²	1	2	1.7	1	2	1.7

¹Washing with freshwater removes soluble salts generated in the Basecon™ transformation.

²Others include: diaspore, lepidocrocite, chromite, monazite, zircon, fluorite, euxinite, gypsum, anhydrite, bassanite, whewellite; Iron oxides & oxyhydroxides include: hematite & ferrihydrite; Ca(Al) hydroxides & hydroxycarbonates include: hydrocalumite & p-aluminohydrocalcite; Mg(Al) hydroxides & hydroxycarbonates include: brucite & hydrotalcite; hydrated alumina includes: boehmite & gibbsite (mainly boehmite); titanium oxides include: anatase & rutile; calcium carbonates include: calcite & aragonite.

In experimental trials, artificial dredge spoils (containing pyrite at 6.5, 5.2, 2.8, 2.3, 1.05, 0.89, 0.59, & 0.15% S²⁻, and a 2:1 quartz, kaolinite mix) were fully oxidised using 33% hydrogen peroxide in the presence of a ViroBind™ reagent to obtain a target pH of 5.5 (Clark, 2000). The data from these experiments indicate that as the acid generating capacity of the soil decreases, the amount of ViroBind™ reagent required to achieve neutralization (pH 5.5) decreases at an even faster rate than expected (Figure 1). Plotting experimental data against the theoretical acid-base neutralization curve shows that suppression of acid generation by the ViroBind™ reagent exceeds expectations based on simple acid-base reactions. It is possible that the ViroBind™ reagent leads to the formation of mineral coatings (e.g. jarosites or similar) on the sulfides causing micro-encapsulation of the pyrite thereby inhibiting further oxidation. An electron microscopic study (conducted for the US EPA by Dr John Drexler, University of Colorado at Boulder) of sulfidic mine wastes that had been treated using ViroSoil™ Technology at the former Gilt Edge Mine, South Dakota, found that well developed mineral coatings had selectively formed on sulphide mineral grains, but currently there is no proof that such coatings could explain the need to use much less reagent than theoretically expected. Furthermore, Lin et al. (2002) found that if the target pH for the soil solution was lowered from 5.5 to 5.0, the effective acid neutralizing capacity (ANC) of the ViroBind™ reagent tested increased from 3.7 to about 14 mol H⁺/kg, indicating that the reagent contains abundant colloidal minerals that have a strong pH controlled tendency to form variably charged particles. The application of these findings to dredge spoil from the Brisbane River estuary shows that the same effects occur in natural sediment and are not an artifact of the use of a artificial sediment in experimental work (Clark, 2000).

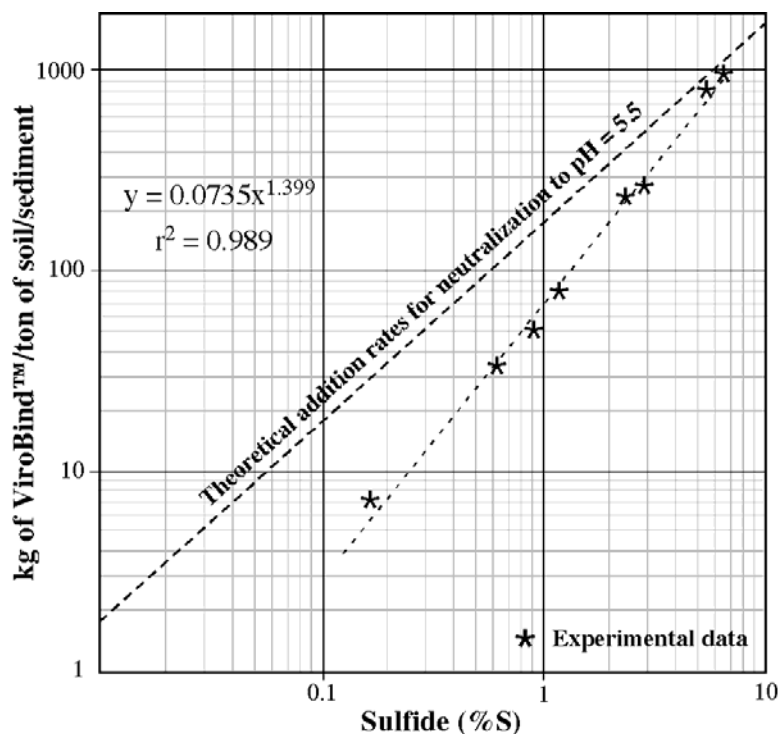


Figure 1. Log/log plot of soil sulphide content vs. ViroBind™ reagent addition rates required to neutralize a fully oxidised soil to a pH of 5.5. This plot involved a ViroBind™ reagent with an ANC of 3.5 mol H⁺/kg; almost identical relationships are found for ViroBind™ reagents with an ANC of 5 mol H⁺/kg.

GEOCHEMICAL CHARACTERISTICS OF VIROBIND™

The ability of ViroBind™ to serve as a conditioner for dredged material depends on two geochemical conditions:

- The ability to neutralize acidity and to reduce the rate of sulfide oxidation, thereby preventing the formation of additional acidity, and
- The ability to remove metals and metalloids from solution in pore water within the dredged material.

Titration of ViroBind™ reagent with acid reveals that two neutralization processes occur:

- Rapid neutralization involving reaction with the hydroxides, hydroxycarbonates and carbonates in the reagent, and
- A slower protonation reaction as iron and aluminum oxides react with hydrogen ions (this process is most effective at lower pH conditions)

The acid neutralizing capacity of ViroBind™ ranges from 3.5 to 9 moles H⁺/kg (usually 5 – 7 moles H⁺/kg), but can be further increased by blending it with additional neutralizing agents if required.

ViroBind™ reagent has the ability to remove almost all metals and metalloids from solution and to bind them in a non-leachable solid form. Metal trapping efficiencies for ViroBind™ show a preference as Pb > Fe ≈ Al > Cr > Cu > Zn > Cd ≈ Ni > Co ≈ Mn (Clark et al. 2007). The most tightly bound metals are least susceptible to leaching, but even less tightly bound metals, such as cadmium and manganese, are still very difficult to remove by leaching and the susceptibility of all metals to leaching decreases with time. This, and the ability to load more than 1500 meq metals per kg of ViroBind™ indicates the loading mechanism is a form of chemi-sorption and not mere cation exchange.

Another interesting aspect of the ViroBind™ reagent is that it has an excellent ability to bind phosphate in such a way that it is not leachable by water, but remains plant-available (citrate soluble in standard agronomy tests). In many soils, particularly sandy soils, about 50% of applied phosphate fertilizer can be lost by leaching following rainfall, whereas less than 1 % can be leached from ViroBind™-treated soils under similar rainfall conditions. This assists the growth of plants on disposal areas during reclamation (Maddocks et al., 2003).

APPLICATION METHODS

The ViroBind™ reagent is available either as a fine-grained powder, a slurry, or as agglomerated pellets or granules. The powder can be mixed (either dry or as a slurry) into dredged material in the discharge line or milled into the dredged material at transfer points, for active dredging sites. In the case of acid neutralization and revegetation of existing upland CDF, the powder could also be spread on the surface and mixed into the upper layer using conventional agricultural equipment, thereby neutralizing acidity and binding metals (e.g. aluminium that could be toxic to plants at elevated concentrations) in the upper root zone, to enhance plant growth. Such a treatment has been demonstrated to be far superior to conventional liming of the surface because the ViroBind™ reagent has a very low solubility and does not flush from the waste with time. Even if the ViroBind™ reagent is applied only to the upper 0.5 m of the soil or dredge spoil the depth that is effectively treated will more than double over about twelve months, but if it is necessary to treat the material to a greater depth, a slurry of reagent can be injected under pressure on a grid basis.

Runoff and dewatering water can be treated by placing pelletised or agglomerated reagent into permeable, flow-through discharge structures, to provide passive treatment of any contaminated water prior to discharge.

CASE HISTORIES

Much of the existing experience with the application of ViroBind™ comes from Australia, an area with extensive areas of acidic soils and dredged material. It is estimated that Australia has over 50,000 square kilometers of coastal acid sulfate soils, containing over a billion tonnes of pyrite. Two example cases of the applications are listed below.

Gladstone Port Authority, Queensland

The Gladstone Port Authority had a problem with acid producing dredged material stored on land. The soil had a pH of about 4, a high exchangeable sodium content, a high net acid generating potential and the potential for leachable metals to adversely affect the nearby environment. During construction of railway lines, approximately 30,000 cubic meters of this material was excavated and stockpiled, resulting in an acidic runoff problem. No vegetation could be grown on the material.

Virotec contracted to remediate the stockpiled material. The objective was to neutralize the pH from the 4.0 present in the stockpile to between 6.5 and 7.0, and to reduce the Net Acid Generating Potential to 0.02 moles H^+ /kg. This was accomplished by determining how much ViroBind™ reagent would be required to neutralize all acid that could be produced if all the sulfides were oxidised and then mixing that amount of ViroBind™ reagent and some fertilizer with the stockpiled sediment, using common construction equipment. As a result, the soil pH was raised from 4 prior to treatment to a range of 6.5 to 7.5 a month after treatment and it has remained in that range over the five years since the treatment was carried out. The treated material has been successfully revegetated and the site now supports a rich cover of grasses and flowering shrubs and trees. Ground-water monitoring efforts show that the acid conditions have been remediated and dissolved metal concentrations have been lowered to acceptable levels.

Action Sands, NSW Australia

Action Sands operates one of Australia's largest sand mining and processing facilities in the northeast corner of NSW, and supplies much of the aggregate for the construction industry on the Gold Coast. During the processing, Action Sands segregated a black sulfidic waste sediment (potentially acid generating), with low nutrient value that would not support plant growth. The material was characterized by:

- a) A high sulfide mineral content and associated Total Potential Acidity (TPA),
- b) A low cation exchange capacity,
- c) A substantial nutrient deficiency, with low levels of potassium, nitrogen, and phosphorus, and
- d) A low moisture retention capacity.

Action Sands wanted to use the waste to produce a conditioned soil mix, to aid in landscaping of the rapidly developing Gold Coast region. They contracted with Virotec to conduct laboratory and field trials, leading to the treatment of about 7,000 cubic meters of stockpiled waste. The field and laboratory tests demonstrated that when the waste material was mixed with ViroBind™ and fertilizer it would support significant growth of grass, flowers,

vegetables, shrubs and trees. The acidity was reduced to near-neutral, the cation exchange capacity increased to an acceptable level, the material had much better moisture retention, and the nutrient levels were suitable for a soil conditioning agent. The waste stockpile was treated and sold, and Action Sands now utilizes all the acidic waste in the manufacture of a high-value soil conditioning mix.

CONCLUSIONS

Mixing ViroBind™ reagent with dredged material as it is removed, or as stored in CDF, has the advantages of:

- * Neutralizing actual acidity resulting from sulfide mineral oxidation,
- * Preventing or reducing the rate of future sulfide mineral oxidation and acid formation,
- * Binding metals and metalloids into a non-leachable form, and
- * Supporting revegetation efforts.

The treated material is no longer environmentally hazardous and can be readily revegetated; in some cases it can be sold as a high quality topsoil. The available forms of the reagent allow mixing into dredge streams, tilling into the surface of CDF, or forming permeable, flow-through structures to allow passive treatment of runoff.

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