

Screening Protocols for Beneficial Utilization of Solid Waste Residuals as Soil Amendments and Conditioners

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Cooperator: Don DeLorme – VDACS Richmond

Objectives

- To *describe* the Virginia cooperative program for state labeling of recycled mine wastes and other residuals when used for various beneficial uses.
- To *detail* the procedures used by Virginia Tech and VDACS to provide reasonable analysis and screening for any residual proposed for land application or soil blended use.

Objectives

- To *discuss* a wide range of industrial that we have successfully developed labels and major markets for in Virginia.

Cooperating Agencies

- **Virginia Tech** – Screening and “Advice”
- **Virginia DEQ** – Their waste definition allows for wastes that are validly recycled or labeled by VDACS to be excluded from designation as “waste”. However, waste must pass a TCLP!
- **Virginia Dept. of Agric. & Consumer Services (VDACS)** – Labels and regulates fertilizers, limes, soil amendments, potting soils, etc.

History of Cooperation

- **As Virginia's Land Grant University, VT has long supported VDACS in a wide array of research, extension and outreach activities.**
- **In the early 1990's, VDEQ developed new beneficial use guidelines for coal combustion by-products that specifically included labeling by VDACS as one way to "de-list" fly ash etc. as solid waste.**

History of Cooperation

- **VDACS was immediately contacted to accept a wide range of CCB's, wood ash and other residuals for soil applied uses. Landfill costs were also obviously driving this trend.**
- **In 1995, VDACS requested formal guidance from VT on what appropriate testing and screening protocols should be employed for industrial residuals.**

March 1995
memo to
VDACS
establishing
minimal
screening
protocols and
requirements
for labeling of
industrial
residuals such
as fly ash or
other XYZ
products as
proposed.

March 30, 1995

Mr. Jay Crane
Program Manager, Office of Product Regulation
VDACS
P.O. Box 1163
Richmond, VA 23209

Dear Jay:

I am sorry that I have been delayed in formally responding to you regarding our stance on the application of coal combustion by-products (CCB's) and other industrial by-products as soil amendments. However, over the past several months I have taken the opportunity to meet with several of our soil chemistry and fertility faculty here, and with this letter I would like to lay out our proposed approach to working with you on dealing with these materials. While the beneficial reuse of waste materials such as fly ash, FGD scrubber sludges, etc. is certainly in the best interest of the Commonwealth, we must insure that our soils will not be damaged by land application practices and that the food chain is not endangered. Hence, we will take a conservative approach to evaluating any material as a potential soil amendment for agricultural lands. Therefore, I propose that we take the multi-step approach described below to dealing with these various materials as they are presented to you for consideration.

We are willing to perform sequential laboratory, greenhouse, and field plot screening of soil amendment materials for you (or the supplier) on a fixed-cost basis. Due to the severe budget cuts imposed upon us over the past several years, we simply cannot supply this work as a public service, even though we would certainly like to. We are experienced and skilled in conducting these sorts of analyses and trials, and would be happy to serve as a "third party" evaluator of these products. First of all, we will assume that the material of interest (e.g. fly ash) is eligible for beneficial re-use consideration and meets the standards for such as set by DEQ. This typically involves a TCLP test and/or other certifications. Secondly, we must assume that the use of the material as a soil amendment must represent a *beneficial* addition to the plant/soil environment and not just a "legal" disposal option. If we (VPI and VDACS) concur on these questions, then we would propose the following stepwise process. Please note that the costs of each "step" given below assume that we would be working with a number of materials over time. If we are forced to do the greenhouse and field plot work on a single material, the costs would be higher. Also please be aware that these are informal estimates only and any contract work such as this would need formal University approval. I feel confident that the estimates are reasonable, since they are derived from actual work that we have completed or is currently active.

VDACS Labeling

- **Originally developed for mandatory and necessary labeling of N-P-K fertilizers and liming materials for content, solubility and efficacy. All fertilizers and limes sold in Virginia must be tested and labeled.**
- **Standard AOAC lab testing and reporting protocols available and used.**

VDACS Labeling

- Also has regulatory language empowering them to label and set inspection fees for:
 - A. Specialty Fertilizers*
 - B. Soil Conditioners*
 - C. Off-grade liming materials*
 - C. Soil Amendments*
 - D. Horticultural Growing Media*
- VDACS does not vigorously pursue labeling of all these material in the marketplace, but does selectively enforce label requirements where it feels indicated.

VIRGINIA DEPARTMENT OF AGRICULTURE AND CONSUMER SERVICES

2010 Rev.

GUIDELINES FOR APPROVING INDUSTRIAL CO-PRODUCTS FOR AGRICULTURAL USE UNDER THE VIRGINIA FERTILIZER AND AGRICULTURAL LIMING MATERIALS LAWS

PURPOSE

The Virginia Department of Agriculture and Consumer Services (VDACS) recognized in 1991 that many industries were interested in identifying agricultural uses for waste products and process residuals, including coal combustion products (CCPs), paper mill sludge, wood ash, kiln dust, tobacco dust and foundry sand, that chemically contained plant nutrients. In 1994, the Virginia Fertilizer Law and Agricultural Liming Materials Law were amended to allow waste products, referred to as industrial co-products, to be used as a fertilizer, soil amendment, soil conditioner, horticultural growing medium or liming material. Businesses were required to demonstrate that these waste products provided a clearly observable benefit to plants and/or soils, were safe to use and apply, and met the definition and criteria for one of these regulated products as defined in the Virginia Fertilizer and Agricultural Liming Materials Laws. Currently, industrial co-products may include, but are not limited to coal combustion products (CCP), exceptional quality biosolids (wastewater sewage sludge), and other organic and inorganic matrices including designer mixtures of many such wastes.

REQUIREMENTS OF THE LAWS

The following sections of the Virginia Fertilizer Law, Virginia Agricultural Liming Materials Law and regulations promulgated under these laws describe the authority by which VDACS can request and review additional data before allowing an industrial co-product to be registered and distributed in the Commonwealth.

§3.2-3613.A.1. of the Virginia Fertilizer Law prohibits distribution of any regulated product if it contains any deleterious or harmful ingredient, in sufficient amount to render it injurious to beneficial plant life, when applied in accordance with directions for use on the label.

§3.2-3607.D. of the Fertilizer Law states, “The commissioner may require verification of any labeling claims for any regulated product.”

Sections 3.C.3 and 3.C.4 of 2VAC5-400-30 “Rules and Regulations for the Enforcement of the Virginia Fertilizer Law” state that the Commissioner may require proof of any claims made for any soil conditioner or soil amendment or one of its labeled ingredients. If no claims are made, the Commissioner may require proof of usefulness and value. For evidence of proof, the commissioner may rely on experimental data, evaluations, including evaluations of data submitted or advice from such sources as the Agricultural Experiment Station and Extension Service of VPI & SU.

Underlying Assumptions for Screening XYZ Residuals

- Utilization of any residual as a soil amendment or in blended soil products must be **presumptive beneficial** use, not simple co-disposal or low cost alternative to land-filling.
- Virginia Tech **can** perform screening analyses as indicated by VDACS for a fee, but any other qualified lab or organization is also fully acceptable.

GUIDELINES FOR APPROVAL

Any person requesting approval of an industrial co-product to be used as a regulated product under the requirements of the Virginia Fertilizer Law or the Virginia Agricultural Liming Materials Law shall, at the request of the Commissioner of the Virginia Department of Agriculture and Consumer Services, provide product data as outlined in the following four steps. Product data shall be used to assess the product's benefit and potential deleterious effects necessary to allow any product to be distributed in the Commonwealth:

1. Initial characterization demonstrating the waste product (i) is non-toxic and non-hazardous with respect to RCRA subtitle C criteria via the appropriate tests and (ii) provides a defined benefit as a reusable product as required for "delisting" by the Virginia Department of Environmental Quality (DEQ) as a regulated solid waste.

Underlying Assumptions for Screening XYZ Residuals

- **VDACS remains the final arbiter of quality and labeling for these materials. VT or other labs simply run tests and make recommendations.**
- **Virginia Tech will review other laboratory supporting data upon request by VDACS and offer opinions.**

2. A complete chemical, physical, mineralogical analysis (as appropriate) of the product, conducted by an independent laboratory recognized and approved by the Commissioner (Appendix A).
3. Greenhouse pot studies of the product utilizing soil and plant materials from the proposed utilization area, conducted by or under the direction of an independent research facility recognized and approved by the Commissioner such as a Land Grant University.
4. Outdoor field trials to confirm the actual effectiveness of the product on soil properties, plant growth, and leachate quality. The field trials shall be run for a minimum of one full growing season and shall be conducted by or under the direction of an independent research facility recognized and approved by the Commissioner such as a Land Grant University.

VT/VDACS Waste Screening Protocols

- **The supplying industry or mine must provide evidence such as TCLP and total elemental analysis results that the product is not hazardous/toxic per DEQ and EPA criteria.**
- **Depending on material properties, part or all of a prescribed three-step screening procedure must be followed and reported to VDACS.**

VT/VDACS Waste

Screening Protocols – Step 1.

- A full analysis of the basic physical and chemical analysis of the proposed material must be provided to include pH, soluble salts, organic matter content, nutrients and extractable cations, total heavy metals, particle size/texture, etc.
- If the proposed material is a well-documented material like wood ash or gypsum, this level of analysis is usually sufficient for label development.

Baseline Characterization

The following is a brief summary of the tests required for review and determination of an industrial co-product as a fertilizer, soil amendment, soil conditioner, horticultural growing medium or liming material before the product may be registered and distributed within the Commonwealth. However, based on the uniqueness of the waste and the extent to which the relevant properties and characteristics of the waste have been previously studied, additional tests and analysis may be required to fully characterize the waste product and its suitability for distribution and application to agricultural lands and homeowners' properties.

For predominately inorganic wastes such as coal combustion products the following are required:

1. pH and calcium carbonate equivalent (CCE).
2. Extractable P, Ca, Mg, K, Na and soluble salts.
3. Total As, Al, B, Ba, C, Ca, Co, Cr, Cu, Fe, Hg, K, Mg, Mn, Mo, Na, Ni, P, Pb, S, Se, Si, Ti, Zn.

For predominately organic waste, including water treatment residuals (WTR) and exceptional quality biosolids the following are required:

1. Complete nutrient and metal elemental analysis including solids content, pH, calcium carbonate equivalent (CCE), total organic C, TKN, NO₃-N, NO₂-N, and NH₄-N.
2. Total P, K, Ca, Mg, S, As, Cd, Cu, Hg, Mo, Ni, Pb, Se and Zn.
3. In addition to the above, for exceptional quality biosolids a detail description of the processes employed to meet the Class A pathogen requirements in 40 CFR 503.32(a) and the vector reduction requirements in 40 CFR 503.33(b)(1) through (b)(8).

Organic wastes and sludges must be tested for EPA designated Priority Pollutants and shown to be under any current risk-based action levels. Water treatment sludges must meet the EPA 503 guidelines for heavy metals content for land application. The 503 guidance levels for heavy metals in biosolids are commonly used as a general screening tool for all organic wastes applied to soils, but are not proof-positive within the decision criteria for land-applied products that are not biosolids.

Table 3. Lab analysis, C:N ratio, and estimated N mineralization for Georgia Pacific mixed (primary + secondary), secondary, and pond sludge samples. Analyses are by A&L Labs, Richmond, VA, except for total C and N and saturated paste EC, which were run at Virginia Tech, and estimated organic-N mineralization, which was calculated by Greg Evanylo.

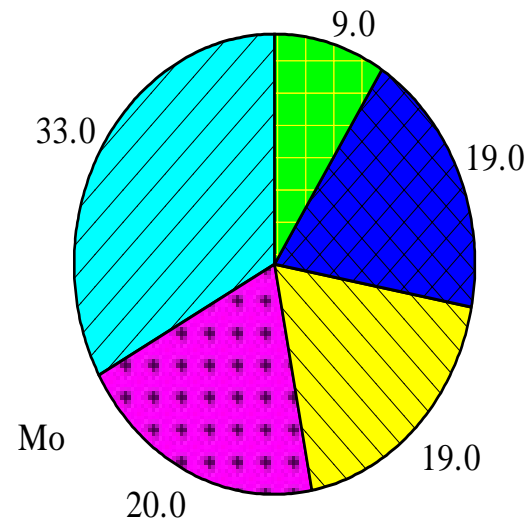
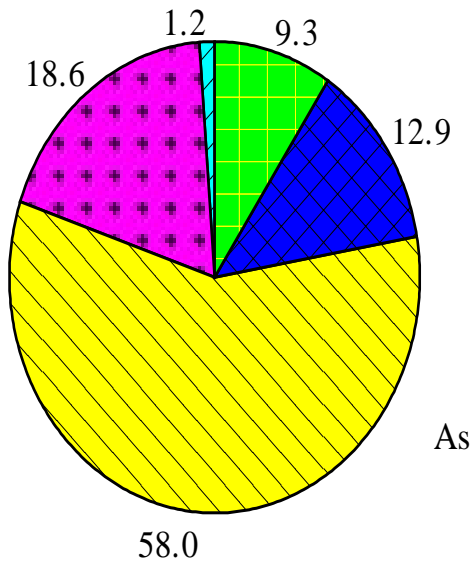
Parameter	Mixed Sludge	Secondary Sludge	Pond Sludge
Total solids (%)	21.17	16.18	7.81**
Total volatile solids (%)	85.40	86.43	73.81
pH	7.58	7.97	7.58
Saturated paste EC (dS/m)	3.61	6.21	7.75
CCE (%)	3.11	2.12	4.21
Total Kjehldahl N (%)	2.89	4.51	1.32
Ammonia N (%)	0.15	0.24	0.22
Organic N (%)	2.74	4.27	1.10
Nitrate + Nitrite N (mg/kg)	nd (<2.0)	64.9	nd* (<2.0)
C:N	18:1	12:1	32:1
Estimated N mineralization (%)	15	25	5

Typical lab characterization data set for waste/residuals. In this case, the materials are three different papermill sludge products.






Total P (%)	0.34	0.51	0.14
Total K (%)	0.19	0.25	0.11
Total S (%)	0.60	0.82	1.03
Total Ca (%)	2.00	2.17	2.15
Total Mg (%)	0.25	0.27	0.16
Total Na (%)	0.17	0.22	0.40
Total Fe (mg/kg)	2520	2580	6280
Total Al (mg/kg)	6200	4700	7500
Total Mn (mg/kg)	885	1520	610
Total Cu (mg/kg)	47	49	89
Total Zn (mg/kg)	125	116	1150
Total Cd (mg/kg)	nd (<2.0)	nd (<2.0)	nd (<2.0)
Total Cr (mg/kg)	32	27	45
Total Ni (mg/kg)	15	13	26
Total Pb (mg/kg)	18	13	35
Total As (mg/kg)	nd (<3.0)	3.0	7.0
Total Hg (mg/kg)	nd (<0.4)	nd (<0.4)	nd (<0.4)
Total Se (mg/kg)	nd (<5.0)	nd (<5.0)	nd (<5.0)
Total Mo (mg/kg)	nd (<5)	nd (<5)	5

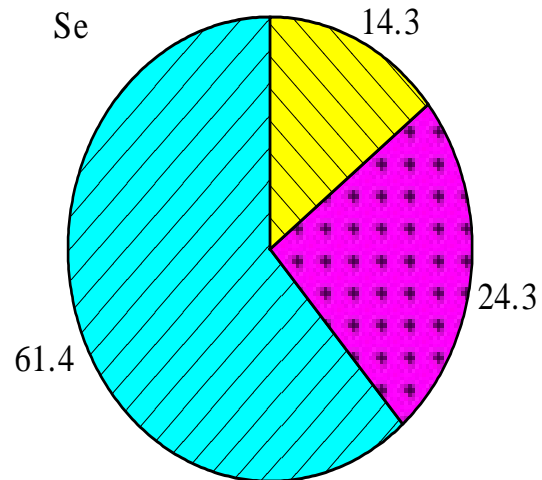
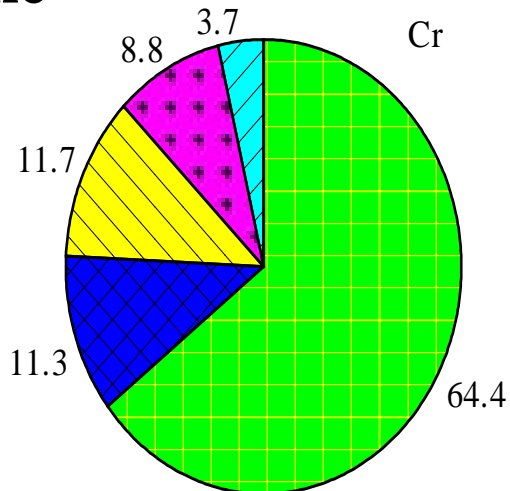
*nd=below limit of detection

**before dewatering



**Sequential
fractionation
data for a fly
ash product.
Not a routine
analysis!**

-  Fraction 1: Exchangeable
-  Fraction 2: Carbonates
-  Fraction 3: Amorph. Fe & Mn
-  Fraction 4: Crystalline Fe & Mn
-  Fraction 5: Residual



VT/VDACS Waste

Screening Protocols – Step 2.

- If the basic analytical data is not clear cut “clean” and/or the material does not have a well-documented history of land application, then a greenhouse screening bioassay is required.
- The bioassay is run with tall fescue (tolerant) and soybeans (sensitive) in a standard Virginia topsoil at either the proposed material loading rates or at a range of rates.



12. 1. 2005

Table 4. Number of soybean seeds, out of 8, germinated after laboratory incubation with saturated paste extract of sludge for 14 days.

Treatment	Mean # soybeans germinated
Control (deionized water)	7a*
Mixed (primary+secondary) sludge	6a
Secondary sludge	8a
Pond sludge	7a

*means followed by the same letter within columns are not significantly different ($P \leq 0.05$; Fisher's LSD).



Figure 1. Soybean plants growing in soil amended with Georgia Pacific sludge on 9/22/13 (12 days after planting). Plants in the secondary sludge treatments appear to be smaller than those in other treatments.



Figure 2. Soybean plants growing in soil amended with Georgia Pacific sludge on 10/15/13 (2 days before harvest). Plants in the secondary sludge treatments now appear to be of similar size to those in the control treatment, while plants in the pond sludge treatments appear to be smaller than those in the other sludge treatments, and have marginal chlorosis on lower trifoliates.



Figure 3. Close-ups of the marginal chlorosis that appeared on lower trifoliates of all soybean plants growing in the pond sludge treatments.

**Soluble salt/B damage
on soybean plants
grown in soil
amended with 10%
coal fly ash.**

**Most legumes are
very sensitive to salt
damage, so seeding
should be delayed
until after salts leach
where possible. But if
the stuff is this salty,
what's the
groundwater effect?**





Soybean toxicity from unknown organic compound in a steam/ pyrolysis treated biosolids product. All conventional lab analyses indicated this product was highly suitable for use as a soil amendment. Fescue, corn and wheat showed no negative effects. *We like soybeans for this test!*



Figure 4. Tall fescue growing in soil amended with Georgia Pacific sludge just before harvest on 10/29/13, showing apparent nitrogen deficiency in pond sludge treatments, and spotty germination in control treatment.

VT/VDACS Waste

Screening Protocols – Step 2.

- **If the bioassay results are conclusive and (A) no overt toxicity is noted and (B) some beneficial plant growth or soil quality response is noted, a positive recommendation is made to VDACS.**
- **That recommendation includes label guidance, loading rate max, and other application restrictions.**

VT/VDACS Waste

Screening Protocols – Step 3.

- **If the bioassay results are mixed, then a full replicated field trial is necessary to confirm field response in the “real world”.**
- **We have had experience with certain products that due to the greenhouse environment did not exhibit a positive response, but did quite well in the field.**

Corn established in June 2002. "Thicker plot" in middle ground is on 100 tons per acre rate with untreated alleys to either side. N applications were minimal (40 lbs/Ac) over the season. Wheat crop in background.



What if field results are negative?

- **Results are reported back to client; they may or may not continue pursuit of labeling with VDACS.**
- **We usually isolate what the issues may be in a given product (e.g. high salts in a compost product), and offer recommendations to modify the product.**

Materials

Screened to Date by VT

- **FGD by-product gypsum (5) (+)**
- **Soybean processing residues (2) (-)**
- **Wood ash (4) (+)**
- **Foundry mold sands (+)**
- **Foundry dust (-)**
- **Papermill sludge or compost (7) (-/+)**
- **Ground/screened construction soil + wood debris (-)**
- **Many other “crazies”, e.g. entire ground demolished buildings.**

Recent Interesting Stuff

- Ground “virgin” wallboard – Good material, also certified in GA and other states
- Spent peat from septic filtration – Nice material; short term pathogen risk, must meet EPA 503 Class A; other “complications”
- GatorAde/Propel Wastewater – Low but sig. N+P; variable solids content over time.
- Ground Ceiling Tiles – Certain formulations phytotoxic; glues?

High Volume Inorganic Materials

- Dredge Spoils – Fresh water, saline, clean or contaminated?
- Fly Ash/CCP's – Vary widely; limited by salts, B, soluble oxyanions of As, Se, Mo etc.
- Waste Limes & Gypsum – Secondary contaminants
- Cement Kiln Dust – Very alkaline; what fires the kiln?
- Wood Ash – Safer/cleaner than most if only wood fired.

Success Stories with Mining Residuals

- **Luck Stone Inc.** has one labeled manufactured topsoil to date and a second product under final development. They market over 30,000 yards per year and *good topsoil sells for \$10 to \$25 per yard FOB.*
- **Hoover Color Inc.** (Fe-oxides for pigments) has developed a marketable soil product from overburden saprolites and waste soil.

Green Quarry granite gneiss saprolites in cut



2. 10. 2004

Composted papermill sludge used as organic amendment.



Mineral fines from air classifier used to blend with saprolites



Mineral mix and composted mill sludge being fed into asphalt batch plant. Current operation uses 2 of 6 blending hoppers.



**Mineral
blend and
composted
mill sludge
traveling
down belt
line to pug
mill mixer
and load out.**



2.10.2004

Final product ready for market.



Advantages of Labeling

- **Offers a clear marketing advantage against non-labeled and more variable materials.**
- **Required by DEQ/EPA for certain waste streams to be exempted from solid waste regulations.**
- **Projects a positive image with the public that you actually are “recycling”.**

Important Themes:

- Beneficial use vs. disposal
- Non-degradation of soil & water
- Economic benefits as soil amendments, limes and fertilizers
- Alternatives? Where does it go if I don't land-apply or use it as soil amendment?
- Unknowns: What's in this material?
- Public perception: Will my neighbors like this stuff?

Conclusions

- **The three-step mechanism outlined here is unique to Virginia, but could readily be implemented elsewhere.**
- **The standard bioassay approach has been proven across a range of materials and is much cheaper to implement than analyzing a waste stream for all known organic and inorganic toxics.**
- **Industry, regulators and the public all benefit and are very positive about the varied benefits of the program.**

2007 EPA “White Paper Report” on how to match use of soil amendments to stabilize and remediate the full range of mining wastes and sites.

This document has the most up-to-date and easy to understand approach to understanding what metals/toxicities must be remediated by mine type and what treatment interactions will be.



The Use of Soil Amendments for Remediation, Revitalization, and Reuse



Amendment	Availability	Uses	Public Acceptance	Cost	Advantages	Disadvantages	Links
Wood Ash	Locally available	Increase pH; Source of mineral nutrients, Ca, Mg, K; Can work for odor control.	Accepted.	Materials generally free; Locally variable cover and transport costs.	Acceptance; Cost; Multi-purpose; Can limit odor of organic soil amendments.	Highly variable; Lime equivalent will vary by burn temperature and age of material; Dioxins should not be a problem but tests should be conducted to verify.	
Coal Combustion Products	Most available in eastern U.S.	Increase pH; Source of mineral nutrients (e.g., Ca).	Variable.	Materials generally free; Transport and application fee.	Regulated ² ; Well characterized; Soil aggregation; Light color reduces surface temperature for seedlings; Increases moisture-holding capacity; Reduces odor of organic soil amendments.	Varies plant to plant; can be high B and salts; can leach Se and As.	American Coal Ash Association (http://fp.acaa-usa.org/CCP.htm) The Fly Ash Resource Center (http://www.geocities.com/capecanaveral/launchpad/2095/mar_index.html)
Sugar Beet Lime	Locally available - primarily in west	Increase pH.	Accepted.	Materials generally free; Transport and application fee.	More reactive than agricultural limestone.	Potential fugitive dust.	
Cement Kiln; Lime Kiln	Locally available	Increase pH; High Ca.	Variable.	Materials can have associated cost; Transport and application fee.	Highly soluble and reactive.	Potential fugitive dust; Highly caustic; Variable content; May contain contaminants.	

Amendment	Availability	Uses	Public Acceptance	Cost	Advantages	Disadvantages	Links
Red Mud	Locally available in TX and AR in U.S.	Increase pH; Sorbent.	Variable.	Commercial product from a residual under development.	Demonstrated effective in limited testing in Australia and other sites at moderating pH and sorbing metals.	Potentially costly, High salt content; Variable CCE.	<p>I-99 ARD Remediation Status, June 8, 2005 (http://www.dep.state.pa.us/dep/deputate/fieldops/nc/I_99/Reports_Documentation/5_PennDOT_Acid_Rock_Remediation_Plan/I-99_ARD_Pres_Tran_Sub_Final.ppt#274_8,Interim Remediation Measures)</p> <p>International Aluminum Institute (http://www.world-aluminium.org/environment/challenges/residue.html)</p> <p>Red Mud Project (http://www.redmud.org/home.html)</p>
Lime-stabilized Biosolids	Locally available	Increase pH; OM and nutrient source; Potential sorbent.	See biosolids.	See biosolids.	See biosolids; Potential multi-purpose soil amendment.	Can have high odor; Lower N content than conventional biosolids; Variable lime content.	National Lime Association (http://www.lime.org/ENV02/ENV802.htm#BioS)
Mineral							
Foundry Sand	Large quantities locally available	Modifies texture; Sorbent.	Variable.	Materials generally free; transport and handling fee.	Good filler; Sand replacement.	Can have trace metals, Significant Na; Only Fe and steel sands currently acceptable.	