

FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION

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May 16, 2013

Via Electronic Mail

rossabi@redox-tech.com

Joe Rossabi, Ph.D., P.E.
Redox Tech LLC
200 Quade Drive
Cary, North Carolina 27513

Re: **Oxygen BioChem (OBC)™**

Dear Dr. Rossabi:

The Florida Department of Environmental Protection's Division of Waste Management, which includes the Bureau of Petroleum Storage Systems, hereby reaffirms and updates its original November 7, 2006 acceptance of Oxygen BioChem (OBC)™ for the remediation of petroleum and other suitable contaminants in groundwater and soil, in situ and ex situ. Other suitable contaminants include but are not limited to chlorinated ethenes, chlorinated ethanes, chlorinated methanes, methyl tertiary butyl ether and 1,4-dioxane. OBC™ is a proprietary blend of sodium persulfate and calcium peroxide. This reaffirmation and update supersedes the original acceptance, and applies to the jurisdiction of the Division of Waste Management. Regulatory information is provided in Enclosure 1, and supplemental information is provided in Enclosure 2.

The sodium persulfate component of OBC™ provides a fast initial destruction of contaminants via chemical oxidation, and the calcium peroxide component provides oxygen for a subsequent long-term aerobic bioremediation process. Residual sulfate from the sodium persulfate is also used by indigenous or added microorganisms during the bioremediation step. OBC™ itself does not contain any microorganisms.

For vadose remediation where the underlying groundwater will not be affected by the leaching of OBC™, there are no special concerns beyond those that would normally be addressed in preparing a Remedial Action Plan and conducting a cleanup to the target levels of Chapter 62-777, F.A.C. The soil cleanup target levels set forth by this chapter apply to both the contaminants of concern and any residual chemical species from the OBC™ itself that are regulated as soil contaminants. However, for in situ groundwater remediation applications that involve the direct injection of OBC™ into an aquifer, there are underground injection control regulations that must be observed.

There are no objections to the use of OBC TM provided: (a) the considerations of this letter are taken into account; (b) a site-specific Remedial Action Plan is submitted for review and approval by the Department; (c) the Remedial Action Plan specifies the size and duration of a temporary injection zone of discharge for sodium, sulfate, total dissolved solids and pH, and proposes adequate groundwater monitoring of them pursuant to Rule 62-520.310(8)(c), F.A.C. [formerly numbered 62-522.300(2)(c)]. Chloride, if it will be liberated in appreciable amounts as a degradation by-product during the remediation of chlorinated contaminants may have to be added to this list.

While the Department of Environmental Protection does not provide endorsement of specific or brand name remediation products or processes, it does recognize the need to determine their acceptability from an environmental standpoint with respect to applicable rules and regulations, and the interests of public health and safety. Vendors, upon receipt of an acceptance, must market the products and processes on their own merits regarding performance, cost and safety in comparison to competing alternatives in the marketplace. This acceptance provides regulatory compliance advice that shall not be construed as either an approval of the product or a certification of its performance.

It is not a requirement that a remediation product or process obtain an acceptance from the Department in order to be proposed for use in a site-specific Remedial Action Plan, but the plan must contain sufficient information to show that all applicable and appropriate rules and regulations have been met. For OBC TM, a copy of this acceptance letter should be included in the appendix of each site-specific Remedial Action Plan that proposes its use.

The Department reserves the right to revoke its acceptance of a product or process if it has been falsely represented. Additionally, Department acceptance of any product or process does not imply it has been deemed applicable for all cleanup situations, or that it is preferred over other treatment or cleanup techniques in any particular case. A site-specific evaluation of applicability and cost-effectiveness must be considered for any product or process, whether conventional or innovative, and adequate site-specific design details must be provided in a Remedial Action Plan. You may contact me at (850) 877-1133, extension 3722, if there are any questions.

Sincerely,


Rick Ruscito, P.E.
Ecology and Environment, Inc.
Bureau of Petroleum Storage Systems
Petroleum Cleanup Section 6


Rebecca S. Lockenbach
FDEP Section Leader
Bureau of Petroleum Storage Systems
Petroleum Cleanup Section 6

Enclosures: (1) Regulatory Information
(2) Supplemental Information

c: Tom Conrardy - FDEP/Tallahassee

History

PPL 307 – INN 149 – 11/7/2006

PPL 466 – INN 201 – 5/16/2013

REGULATORY INFORMATION

- a. Groundwater cleanup standards: The onus shall be on users of OBC™ to ensure that all applicable groundwater contaminant standards will be met at the time of project completion, for petroleum and other contaminants that may be present, any residuals associated with the ingredients of OBC™ and any by-products produced as a result of chemical or biochemical reactions involving those ingredients. The following chapters of the Florida Administrative Code (F.A.C.) are cited: Chapter 62-550, F.A.C., for primary and secondary water quality standards; Chapter 62-520, F.A.C. for groundwater classes and standards, and groundwater permitting and monitoring requirements; Chapter 62-528, F.A.C., for Underground Injection Control (UIC), particularly Part V, for Class V, Group 4 aquifer remediation projects; Chapters 62-770, 62-780, 62-782, and 62-785 F.A.C., for cleanup criteria; and Chapter 62-777, F.A.C., for cleanup target levels.
- b. Ingredients: The proprietary blend ratio of sodium persulfate and calcium peroxide in OBC™ was provided in a confidential disclosure to the Bureau of Petroleum Storage Systems on November 6, 2006, as part of the original submittal of information about this product.
- c. Injection well permit: The issuance of a site-specific Remedial Action Plan Approval Order by the Department for injection of OBC™ into an aquifer constitutes the granting of a Class V injection well permit. [62-528.630(2)(c) and 62-528.640(1)(c), F.A.C.]
- d. How to obtain permission for a temporary injection zone of discharge: For injection-type, in situ aquifer remediation, the composition of an injected fluid must meet the drinking water standards set forth in Chapter 62-550, F.A.C., and the minimum general groundwater criteria of Chapter 62-520, F.A.C., pursuant to underground injection control Rule 62-528.600(2)(d), F.A.C. Chapter 62-777, F.A.C., provides additional groundwater criteria for specific chemicals or regulatory interest. Aquifer remediation fluids that do not meet these requirements must seek permission for a temporary injection zone of discharge. In the case of OBC™, the following four parameters do not meet the requirements and must therefore seek permission for a temporary injection zone of discharge pursuant to Rule 62-520.310(8)(c), F.A.C.: sodium, sulfate, total dissolved solids and pH.

In order to obtain permission for a temporary injection zone of discharge by way of Rule 62-520.310(8)(c), F.A.C., a site-specific Remedial Action Plan proposing the use of OBC™ must: (a) indicate that the concentrations of sodium, sulfate, and total dissolved solids and the pH of OBC™ may cause a temporary exceedance of their respective groundwater standards; (b) specify the size of the injection zone of discharge in which the temporary exceedance could occur; (c) specify the period of time that the temporary injection zone of discharge will be needed; and (d) propose groundwater monitoring of the parameters that could cause an exceedance. Chloride, a by-product when chlorinated contaminants are degraded, should be added to the list of chemical species to be monitored in the groundwater if it will be produced in significant amounts. Department approval of a site-specific Remedial Action Plan that meets the requirements of Rule 62-520.310(8)(c), F.A.C., constitutes the granting of permission for a temporary injection zone of discharge for aquifer remediation purposes.

The current groundwater standards for the OBC™ parameters that require a temporary zone of discharge are sodium (160 milligrams per liter, mg/L), sulfate (250 mg/L), total dissolved

- solids (500 mg/L), and pH (range 6.5 to 8.5). The current groundwater standard for chloride is 250 mg/L. If the groundwater's natural-occurring background level of any of these parameters at a specific remediation site does not meet the established groundwater standard, then the residual level of that parameter by the time the temporary zone of discharge period ends shall be no worse than the pre-existing, pre-injection background level.
- e. Utilization of wells: If a remediation site happens to have an abundance of monitoring wells, then the Department has no objection to the use of some wells for the application of OBC™. However, no "designated" monitoring well, dedicated to the tracking of remediation progress (by sampling) shall be used to apply OBC™. This will avoid premature conclusions that the entire site meets cleanup goals. By making sure that designated tracking wells are not also used for treatment, there will be more assurance that the treatment process has permeated the entire site and that it did not remain localized to the area immediately surrounding each injection well.
- f. Groundwater monitoring:
1. Active remediation petroleum monitoring: During the period of active remediation, groundwater shall be monitored in accordance with the requirements set forth in Section 62-770.700, F.A.C., for the petroleum contaminants of concern, or in accordance with an approved Remedial Action Plan for non-petroleum cleanup contaminants of concern.
 2. Post active remediation petroleum monitoring: At least one (1) year of quarterly post remediation groundwater monitoring for the petroleum contaminants of concern shall be conducted at a minimum of two (2) wells, one located in the area of maximum petroleum contamination, the other downgradient of the area of maximum petroleum contamination, pursuant to Section 62-770.750, F.A.C. For non-petroleum cleanups, the monitoring shall be in accordance with an approved Remedial Action Plan.
 3. Underground injection control monitoring: For Underground Injection Control purposes, when OBC™ is injected, the groundwater must be monitored pursuant to Rule 62-520.310(8)(c), F.A.C., for the parameters discussed above in paragraph *d*.
- g. Underground Injection Control (UIC) inventory: Remedial Action Plans proposing injection-type aquifer remediation shall include information pursuant to Rules 62-528.630(2)(c)1 through 6, F.A.C., for the inventory purposes of the UIC program. Reviewers of those plans, upon issuance of an enforceable Remedial Action Plan Approval Order by the Department, must submit a completed copy of the "UIC Notification" to the UIC program in Tallahassee. The notification is currently located on the Internet at www.dep.state.fl.us/waste/categories/pcp/pages/innovative.htm.
- h. Operation:
1. Avoidance of migration: For injection-type, in situ aquifer remediation projects, injection of the OBC™ shall be performed in such a way, and at such a rate and volume that no

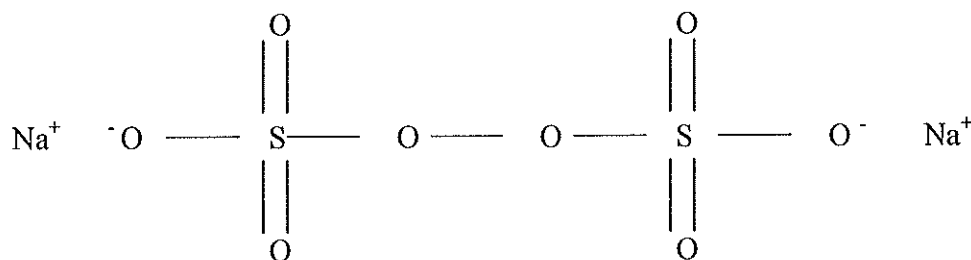
undesirable migration of either the reagent or the contaminants in the aquifer results, pursuant to Rule 62-528.630(3), F.A.C.

2. Underground injection control operating permit: Although an operating permit is not required for aquifer remediation wells pursuant to Rule 62-528.640(1)(b), and 62-528.640(1)(c), F.A.C., since no movement of the contamination plume is expected to accompany the treatment process, the Department requests that the information items listed in Rule 62-528.640(1)(b), F.A.C., be considered and included in Remedial Action Plan proposals as a matter of good and thorough design practice. Briefly summarized, they are: quality of water in the aquifer; quality of the injected fluid; existing and potential uses of the affected aquifer; and well construction details. Additionally, each Remedial Action Plan should clearly indicate the total volume of OBC TM that will be injected.
- i. Abandonment of wells: Upon issuance of a Site Rehabilitation Completion Order, or a declaration of "No Further Action", injection wells shall be abandoned pursuant to Section 62-528.645, F.A.C. The Underground Injection Control Section of the Department shall be notified so that the injection wells can be removed from the inventory-tracking list.
- j. Open-pit applications: While open-pit application of OBC TM is not an injection-type application, and notification of the Underground Injection Control Section therefore not required, the user of OBC TM must still be mindful of groundwater quality. For open-pit applications, the Division of Waste Management suggests that groundwater in the application area be monitored for the same parameters that would be monitored had the application been an injection. These parameters are listed above in paragraph *d*.

SUPPLEMENTAL INFORMATION

- a. Pilot study: For petroleum cleanup projects, per rule 62-770.700(2), Florida Administrative Code (F.A.C.), a pilot study proposal shall be submitted for review, and a pilot test shall be performed prior to designing a treatment system. If conditions or the situation at a site do not warrant a pilot study, then a proposal explaining the rationale for the decision not to perform a pilot study shall be submitted for review.
- b. Application rates: The amount of OBC TM to be applied should be determined on a site-specific basis, but for information purposes the following rules-of-thumb may be helpful:
- For the treatment of groundwater: 1 to 50 grams of OBC TM per liter of groundwater
 - For the treatment of soil: 1 to 100 grams of OBC TM per kilogram of soil
 - As an additive to an open excavation pit 1 to 10 pounds of OBC TM per square foot of treatment area
- c. Chemical processes at work: The sodium persulfate component of OBC TM destroys contaminants both by direct oxidation and by free radical mechanisms involving sulfate radicals. The lifetime of these radicals is estimated to be on the order of several months. The calcium peroxide component of OBC TM can release oxygen for up to 9 months.
- d. Stoichiometry: It has been indicated that a stoichiometric ratio for radical destruction is rather difficult to present, but that a ratio for direct oxidation can be shown by example for the complete mineralization of benzene. The ratio is 15 moles of sodium persulfate [Na₂S₂O₈] at 238 grams per mole to 1 mole of benzene [C₆H₆] at 78 grams per mole. On a weight basis, this is equivalent to 45.8 grams of sodium persulfate to 1 gram of benzene.

Molecular structure of sodium persulfate:



- e. Activation: The production of sulfate radicals from the persulfate anion can be activated in several ways: by heat, metals, hydrogen peroxide or high pH. In the case of OBC TM, the activator is the high pH of the calcium peroxide component.

- f. Oxidation potential: The persulfate anion produced by OBC TM is a relatively powerful oxidant, and the sulfate radical produced is more powerful still. Their oxidation potentials, in volts (V), relative to some other oxidants are shown below.

Highest Oxidation Potential	2.7 V	Hydroxyl Radical ($\bullet\text{OH}$)
	2.6 V	Sulfate Radical ($\text{SO}_4\bullet$)
	2.2 V	Ozone (O_3)
	2.1 V	Persulfate anion ($\text{S}_2\text{O}_8^{-2}$)
	1.8 V	Hydrogen Peroxide (H_2O_2)
	1.7 V	Permanganate Anion (MnO_4^-)
Lowest Oxidation Potential	1.4 V	Peroxymonosulfate Anion (HSO_5^-)

- g. Comparison to other oxidant chemistries: In addition to oxidizing strength, these other advantages of persulfate and sulfate radical oxidation have been indicated.
- Radical oxidation is kinetically fast.
 - The persulfate is more stable than hydrogen or the hydroxyl radical, so there is more opportunity for it to last longer and travel further in the subsurface.
 - The persulfate has less affinity for natural soil organics than permanganate, thereby making it more efficient in applications in high organic soils.
- h. Other contaminants: In addition to the cleanup of petroleum, OBC TM may be of use for the remediation of other suitable contaminants including but not limited to chlorinated ethenes (PCE, TCE, DCE), chlorinated ethanes (TCA, DCA, vinyl chloride), chlorinated methanes (carbon tetrachloride, chloroform, methylene chloride) and 1,4-dioxane.
- i. Safety reminders:
- Use of proper protective clothing.
 - Oxidation is an exothermic reaction.
 - Use storage containers, tanks and piping constructed of 304 or 316 stainless steel, polyvinyl chloride, polyethylene, Plexiglas, Teflon or glass for sodium persulfate. Do not use soft metals such as copper, brass or carbon steel.
 - Store OBC TM in a cool, dry place. See the manufacturer's Material Safety Data Sheet for additional information.
 - Dispose of OBC TM as a hazardous waste.