GREENCLEAN PRODUCT EVALUATION AND RECOMMENDATION (October 2010)

Table of Contents

This document addresses the human health and ecotoxicity review conducted by the Massachusetts Department of Environmental Protection (MassDEP) Office of Research and Standards (ORS) of two herbicide products relative to their addition to the list of herbicides associated with the Eutrophication and Aquatic Plant Management in Massachusetts: Generic Environmental Impact Report (hereafter referred to as the GEIR). This document also summarizes the conclusions and recommendations regarding use of these products in Massachusetts waterways. The summary of toxicity and fate information for the active ingredients in these products is contained in the toxicological and environmental fate profile for these compounds entitled "Hydrogen Peroxide, Peracetic Acid and Sodium Percarbonate (October 2010)" (hereafter referred to as the H_2O_2 tox/fate profile).

The products reviewed in this document include **GreenClean Pro Granular Algaecide/Fungicide** and **GreenClean Liquid Broad Spectrum Algaecide/Bactericide** manufactured by BioSafe Systems. Both of these products contain hydrogen peroxide, a strong oxidizing agent, as either an active ingredient or a component of one or more key ingredients of the product formulation. GreenClean Liquid also contains peracetic acid as an active ingredient. GreenClean Pro contains sodium percarbonate (i.e., sodium carbonate hydrogen peroxide) as its active ingredient. The Liquid and Pro products are composed of 27% and 27.6% hydrogen peroxide respectively. The Liquid contains 2% peracetic acid (Knox, 2009).

The conclusions and recommendations contained in this document are also extended to any other product of similar composition, including **PAK-27** manufactured by Solvay, Inc., which is identical in composition to GreenClean Pro.

Since both GreenClean products contain hydrogen peroxide, both products were evaluated based on consideration of hydrogen peroxide toxicity. In addition, the toxicity of peracetic acid was also evaluated for the GreenClean Liquid and the toxicity of percarbonate was addressed for GreenClean Pro.

PRODUCT FORMULATION

Hydrogen peroxide is a reactive oxidizing substance that may form a number of addition compounds with different physical and chemical properties. Peracetic acid and sodium percarbonate are organic addition compounds that are also reactive oxidants and/or break down to hydrogen peroxide. Table 1 provides a listing of ingredients for each product.

Table 1. List of Ingredients for GreenClean Products

PRODUCT	INGREDIENT	PERCENT	NOTE
GreenClean Liquid	hydrogen peroxide	27	
	peracetic acid	2	
	other	71	
GreenClean Pro (and PAK-27)	sodium carbonate peroxyhydrate	85	contains 27.60% H_2O_2 by weight
	other	15	

GreenClean Liquid is a liquid formulation that contains 27% hydrogen chloride and 2% peracetic acid as active ingredients as well as 71% other ingredients, including peracetic acid (Knox, 2009). Peracetic acid is made by combining acetic acid and hydrogen peroxide. It is available commercially as either a solution in which peracetic acid is in equilibrium with glacial acetic acid, water and hydrogen peroxide or as a distilled non-equilibrium solution of mostly peracetic acid and water with minimal residual acetic acid or hydrogen peroxide. Commercial formulations may range from 0.3% to 40% peracetic acid by weight and will often contain stabilizers to prevent decomposition.

GreenClean Pro is a granular formulation that contains 85% sodium carbonate peroxyhydrate as the active ingredient and 15% other ingredients. The active ingredient is a granular substance made by combining sodium carbonate and hydrogen peroxide. 27.60% by weight of the formulated product is hydrogen peroxide.

PAK-27 is a granular formulation identical in composition to GreenClean Pro.

EXPOSURE ISSUES

These products work on the mechanism of chemical oxidation. They exert their effect on contact. Most mammalian and ecotoxic effects produced by these compounds are due to their irritant and corrosive nature. Any dermal, ingestion and/or inhalation exposure to these products by humans in a lake or pond would most probably occur during a swimming or wading scenario. Given that application of these products would likely not be made while people were present and the products are expected to degrade relatively quickly, it is unlikely that use of these products as specified on the product labels would result in significant exposures to humans using the waterbodies recreationally. As the product label specifies, undiluted granules should not be allowed to remain in an area where humans or animals may be exposed.

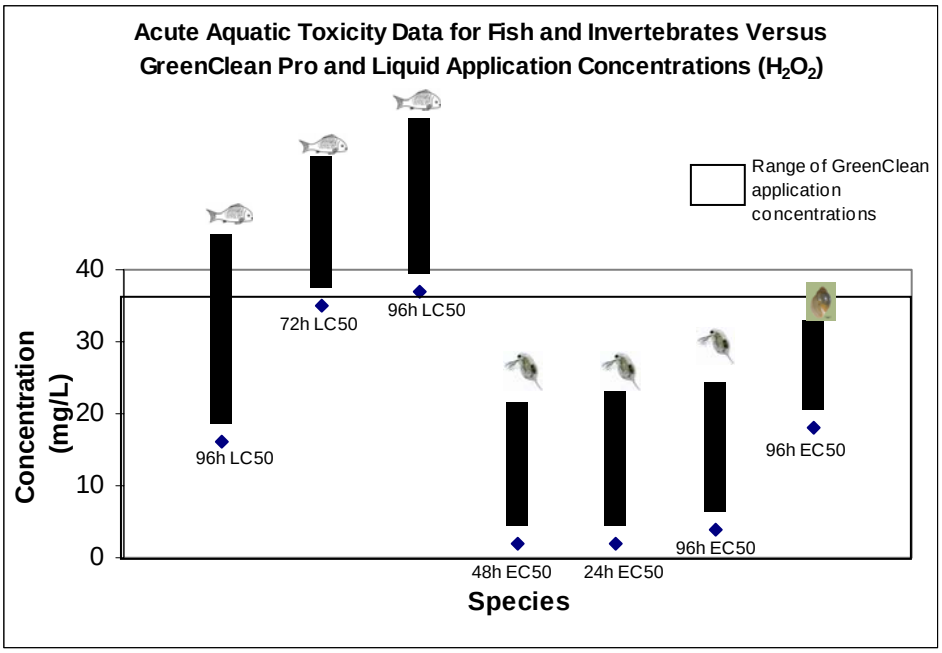
However, exposure of aquatic organisms cannot be restricted in the same way since application is being made directly to their habitat. A review of ecotoxicological data as presented in the tables in the H_2O_2 tox/fate profile indicates reported toxicity levels in several species of *Daphnia* (water fleas) as well as *Gammarus* (shrimp) and (exposed to peracetic acid) *Brachydanio rerio* (zebrafish) are within the range of predicted environmental concentrations of hydrogen peroxide associated with recommended application rates. Although these products break down relatively quickly, they produce their oxidizing effect acutely on contact. It appears that the toxic effect of these products is greater in smaller organisms than large organisms. However, if an application made to a waterbody results in a mass die-off of small invertebrates representing a lower trophic level in the aquatic food chain, there may also be an impact to larger predatory species that depend on these species for food.

AQUATIC ORGANISM TOXICITY

Figure 1 summarizes the range of hydrogen peroxide concentrations (approximately 0.9 – 36.5 mg/L) associated with the range of recommended application rates for the two GreenClean products under review.[1] Acute toxicity concentrations for invertebrates and fish from Table 2 of the H_2O_2 tox/fate profile document are also included in the figure for comparison. The toxicity information represents LC50s (i.e., concentration which is lethal to 50% of a test population) for the three fish species and EC50s (i.e., concentration which produces an effect in 50% of a test population) for the four invertebrate species. Information on the specific EC50 endpoints was not available. Figure 1 illustrates that these products are toxic to invertebrates and, at higher concentrations are toxic to fish. When applied at lower rates, the resulting concentrations, especially for the GreenClean Liquid, are particularly toxic to *Daphnia*.

In marked contrast to this information on fish are the experiences from the fish farming community and hatchery operations. Hydrogen peroxide concentrations up to two orders of magnitude greater than the LC50's reported in Figure 1 are used to treat hatchery and pond fish for fungal and parasitic diseases, with no adverse effects upon the fish themselves, except for one poorly supported documentation of several salmonid mortalities at treatment temperatures greater than 14 ^{o}C (See H_2O_2 tox/fate profile).

Figure 1.



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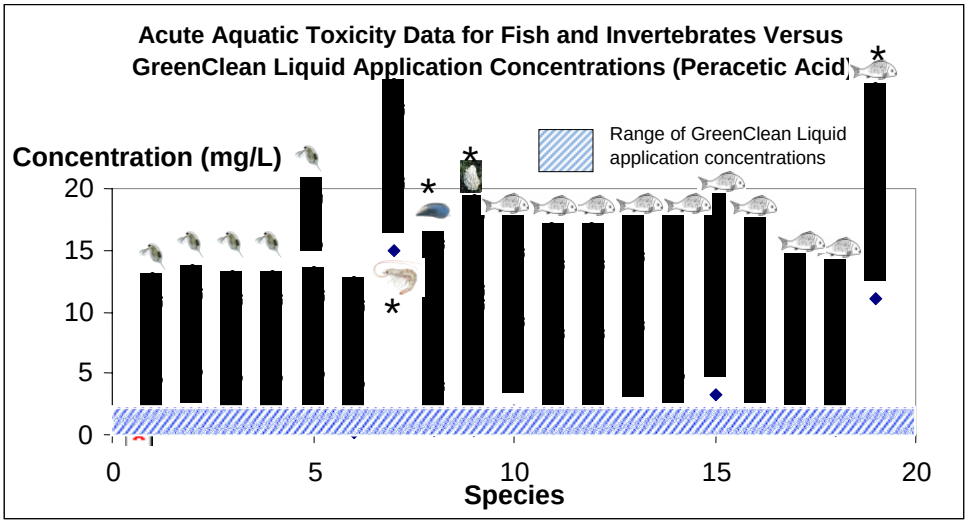
Details of the acute toxicity tests are presented in Table 2 in the H_2O_2 tox/fate profile. The tests were either static or semi-static in design. The most sensitive effect was identified in a static test using *Daphnia*. The static results particularly demonstrate that a one-time application of hydrogen peroxide at application concentrations is acutely toxic to these organisms. Furthermore, GreenClean products are formulated to prolong the oxidation effect. While GreenClean Pro is a concentrated granular formulation that only releases hydrogen peroxide as it comes into contact with water, GreenClean Liquid is formulated with an added stabilizer that retards breakdown.

[1] These concentrations were calculated as described in Appendix 1. It is not clear why the "PPM AI" concentrations presented in the label for GreenClean Liquid differ from these estimated concentrations.

Figure 2 presents similar information for peracetic acid in GreenClean Liquid. The range of peracetic acid concentrations (i.e., approximately 0.08 – 2 mg/L) associated with recommended application rates is illustrated below. The toxicity information for peracetic acid that is included for comparison is derived from Tables 5 and 6 in the H_2O_2 tox/fate profile. These tables present the concentration of peracetic acid for a range of formulations (that vary with respect to their percent weight peracetic acid, hydrogen peroxide and acetic acid). While peracetic acid typically also contains hydrogen peroxide and acetic acid, the toxicity of peracetic acid is due mainly to the toxicity of the peracetic acid component itself (ECJRC, 2003). Therefore, the range of peracetic acid concentrations, given in mg/L, represents acute toxicity to freshwater organisms for this compound.

Figure 2.



* these four species are marine. Two of them have toxicity values greater than the application concentrations, which are not relevant to our freshwater evaluation.
Note: all *Daphnia* values are 48 h EC50s; Crangon crangon is a 48h LC50; the rest are 96h LC50s

The review of these products included a search for relevant information from EPA, particularly toxicity and fate information associated with registration. The 1993 EPA Registration Eligibility Decision RED) document for peroxy compounds indicates that these compounds were registered as microbiocides for use as disinfectants, sanitizers and sterilants in the indoor environment. While the RED states that peroxy compounds are highly toxic to rainbow trout and moderately toxic to bluegill sunfish, impacts to the outdoor environment and organisms are considered minimal since the use patterns reviewed for the RED are all indoors (EPA, 1993a,b).

Since the RED was published, additional use patterns have been requested and added for these compounds. A number of these applications were located on the EPA website. In conjunction with some of these applications, the acute toxicity data presented for invertebrates and fish appear to fall into the same range as that presented in the Appendix and Figure 1. In addition to their use as microbiocides, the peroxy compounds are also being marketed as algaecides, as is the case with the GreenClean products. These applications clearly extend usage of these products to the outdoor environment. However, no documentation was found that explained the basis upon which these products could be registered for direct application to lakes and ponds as algaecides.

The EPA product manager for these products was contacted several times for this information and was not able to provide an explanation other than to state that " . . .all of the necessary data requirements were met when the product was registered, including data to support uses on the label . . .". However, he did not provide or discuss any information supporting this statement. A number of weeks ago, the product manager stated that he would be addressing a GreenClean product review that day and would try to find more information on these issues. However, to date, no information has been received.

Ultimately, the acute ecotoxicity database for invertebrates and fish is very limited and the data that are available indicate that these products have high acute ecotoxicity, but this information is tempered for fish by the

knowledge that hydrogen peroxide is used in the hatchery and fish farming industry to treat fungal and parasitic fish diseases with no adverse effects at much higher concentrations than those associated with lake algal treatments.

For large volume application (i.e., ponds, lakes and lagoons), the moderate and/or heavy algae growth application rates for both products result in estimated water concentrations of hydrogen peroxide exceeding toxic effect levels for invertebrates, and possibly some fish. In addition, for GreenClean Liquid, most of the range of application rates results in a range of peracetic acid concentrations associated with toxicity to invertebrates and possibly, but to a lesser degree, to fish. In general, *Daphnia* appear to be the most sensitive species.

The product label for GreenClean Pro warns that use of this product should be avoided near shallow waterbody margins during amphibian breeding seasons. Such a warning was not provided on the product label for the GreenClean Liquid. Specific information on amphibian toxicity was not found and the toxicological basis upon which this recommendation was made for amphibians was not obtained for this review. It is possible that the concern stems from the potential for contact burns with GreenClean Pro crystals. While the particular contact burn scenario may not be a concern with the GreenClean Liquid, water concentrations after application may potentially be much higher than they would be for a GreenClean Pro application. The GreenClean Liquid also contains a stabilizer that retards breakdown, thus prolonging the oxidation effect. In addition, based on calculated aquatic concentrations associated with recommended application rates, the aquatic concentration of hydrogen peroxide following the maximum recommended GreenClean Pro application rate of H_2O_2 (i.e., 25 mg/L according to the label or 36.5 mg/L based upon a scaling of concentrations as calculated in the Appendix of this document) may be up to four times higher than the aquatic H_2O_2 concentration (9.1 mg/L, based on the Appendix calculations) resulting from the maximum recommended GreenClean Liquid application rate. A review of EPA's ECOTOX database identified a single LC50 value for the frog *Xenopus laevis* of 598.82 μM (or, approximately 20 mg/L).[2] This value falls into the same hydrogen peroxide toxicity range identified for fish and within the range of application concentrations for GreenClean Liquid. In the early stages of amphibian development, there are similarities between fish and the fish-like tadpole stage (i.e., tadpoles have gills, swim via lateral undulation, etc.) of frogs and toads. Given the above information indicating amphibian and some fish toxicity of both GreenClean Liquid and GreenClean Pro products, especially at the moderate and high application rates, it would perhaps also be prudent to extend the amphibian precaution to the GreenClean Liquid.

The product label for GreenClean Liquid warns that this product is toxic to fish. It is unclear why this warning is not included on the GreenClean Pro label. As discussed above and calculated in the Appendix, application concentrations of GreenClean Pro, once dissolved, are in the same range as application concentrations of the GreenClean Liquid.

Another possible concern with regard to GreenClean Pro and ecosystem effects is the potential for sodium percarbonate to produce localized pH changes. Decomposition of sodium percabonate in water forms carbonate, which has a tendency to raise pH. The degree to which pH may change in a waterbody is dependent on the buffering capacity (i.e., generally, the bicarbonate concentration) of that waterbody. Table 1 in the H_2O_2 tox/fate profile provides a range of carbonate concentrations that would raise pH from an initial pH of 8.3 to 9, 10 or 11 for a range of buffering capacities. This range is within the range of application concentrations for these products and also within the range of acute toxicity values identified for fish and invertebrates. Thus, depending on the buffering capacity of the waterbody, localized pH changes are also possible. The impact of these localized changes to aquatic organisms and/or their susceptibility to hydrogen peroxide is unknown.

SUMMARY OF APPLICATION CONCENTRATIONS VERSUS TOXICITY

Table 2 summarizes the range of recommended label application rates and their resulting water concentration for each of the active ingredients in each GreenClean product. In addition, the Table provides the application rate associated with the lowest effect concentrations for invertebrates and fish. This information was used as a basis for the recommendations in the following section.

[2] Since 1 M H_2O_2 is equal to 34 g/L, then 0.000059882 moles/L x 34 g/mole equals 0.002036 g/L, or a water concentration of 20.4 mg/L.

Table 2. Application Rates and Associated Toxicity

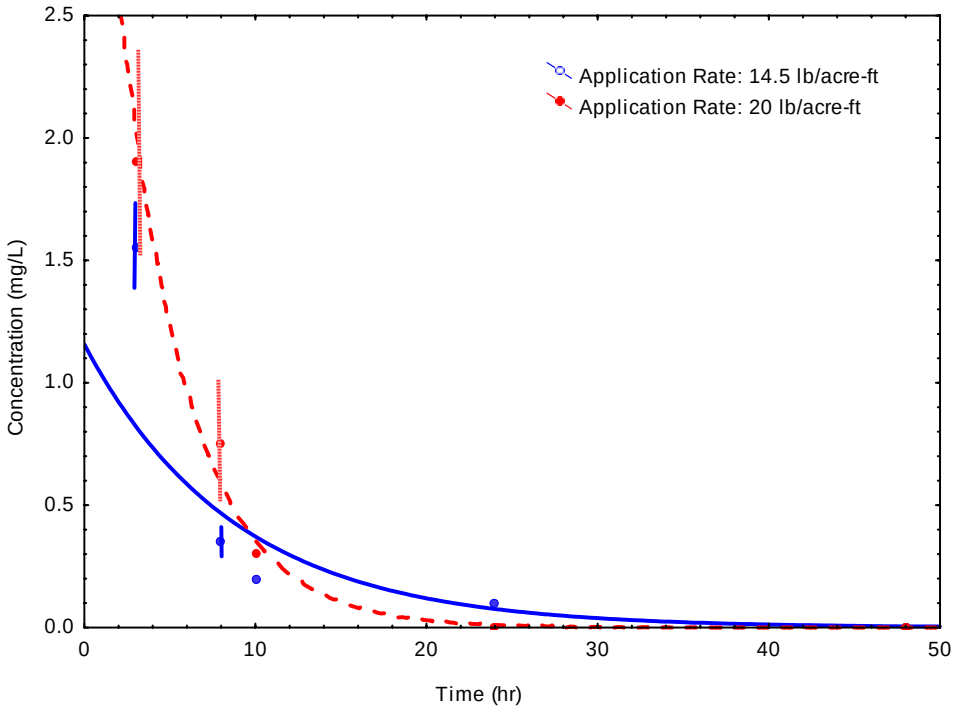
Active Ingredient	Label Application Rate (per acre-ft)	Estimated Water Concentration (mg/L)	Toxicity Range (mg/L) (NOTE: lowest effect concentration is bolded)		Application Rate Associated With Lowest Effect Concentration (per acre-ft)	
			Invertebrates	fish	invertebrates	fish
GreenClean Pro						
Hydrogen peroxide[1]	9 -90 lb	0.9-9.1	2.3-17.7	16.4-37.4	23 lb	162 lb[2]
GreenClean Liquid						
Hydrogen peroxide	1.2-30 gal	1.08-27	2.3-17.7	16.4-37.4	1.89 gal	13.47 gal
Peracetic acid	1.2-30 gal	0.08-2	0.135-1.1	0.35-3.3	2 gal	5.25 gal

[1]While hydrogen peroxide is not listed as an active ingredient for this product, the oxidative effect of this product is due to hydrogen peroxide once it decomposes in water.
[2] This rate of application falls outside of recommended label rates.

Table 2 also summarizes effect levels. When conducting a risk assessment , a theoretical "no adverse effect level" is often estimated from a "low adverse effect level" by dividing the no adverse effect level by a factor of 10. In ecological risk assessment, an effect level known as a Maximum Acceptable Toxicant Concentration (MATC) is defined to fall somewhere in the range defined by the No Adverse Effect Concentration (NOAEC) and low adverse effect concentration (LOAEC). These approaches are used to extrapolate information on effect level when data are limited. In the case of the peroxides addressed in this evaluation, there are limited data on effect levels for limited endpoints, (i.e., immobility for *Daphnia* and lethality for fish). There are uncertainties involved with extrapolating effect concentrations below experimental effect levels. Thus, while extrapolation of a NOAEC would be a conservative measure and acceptable under risk assessment protocols, we note that if "no effect levels" are used as a basis for making recommendations on allowable GreenClean application rates, the allowable application rates would be ineffective and essentially useless. We recognize that there are risks and benefits associated with using any pesticide product. We are therefore addressing our conclusions and recommendations for these products on measures that will minimize effects on the populations of concern.

A comparison of the reported EC50 and LC50 values was done to information on persistence of hydrogen peroxide in water. These data were presented in a water column degradation study (OPP, 2004) of a granular product by another manufacturer similar in composition to GreenClean Pro. It is assumed for the purposes of this comparison that the stabilizer contained in the test product is equally effective as that contained in GreenClean. The degradation study follows the dissolution rate of the hydrogen peroxide-based product applied to the water column at four application rates ranging from 2.5-20 lbs per acre-foot. The measured concentrations at five time increments following application were found to decrease in a time and concentration-dependent manner. The reported concentrations at the two highest application rates tested are reported in Figure 3. These data indicate that hydrogen peroxide residue levels dissipate rapidly and are mostly undetectable at 24 hours. The highest application rate evaluated in this study was 20 lbs per acre-ft. The GreenClean Pro label allows application rates up to 90 lbs per acre-foot, four and a half times higher than the stated allowable application rate for the product evaluated. In addition, the GreenClean Liquid application rates result in application concentrations that are even higher than the GreenClean Pro application concentrations. Given the much higher initial application concentration and interpolating between the 3-hr and 8-hr measured concentrations, it is possible that residual hydrogen peroxide concentrations 1 hour and 4 hours after application will have adverse effects on *Daphnia*. These data are therefore used as a basis for recommending more stringent usage guidelines when using GreenClean products to protect sensitive invertebrates.

Figure 3. Time Decay of Hydrogen Peroxide Concentrations Applied to Water. Vertical Lines Represent Range of Reported Values. Fitted Lines are Exponential Decay Curves.



CONCLUSIONS

Based upon the above discussion, the information presented in Table 2 and the supporting information in the H_2O_2 tox/fate profile, ORS draws the following conclusions:

If GreenClean Pro application rates are less than 23 lb/acre-foot, or GreenClean Liquid rates are less than 2 gallons per surface acre-foot (based on the calculations in the Appendix and Table 2), then organisms would not be exposed to concentrations shown to be toxic and it would be expected that the majority of these organisms would not experience effects. These rates correspond to situations with low-density algae, which may be treated with lower application rates as specified on the product labels.

ORS recommends that additional restrictions be placed on treatment with GreenClean products at higher application rates for several ecological groups, including juvenile amphibians, invertebrate zooplankton (e.g., such as *Daphnia*), and benthic invertebrates. Juvenile amphibians and fish may inhabit shallow waterbody margins. Larger fish species may be found in deeper waters. Benthic invertebrates may be found throughout the lake but especially in the shallow areas. *Daphnia* and other invertebrate zooplankton may be present in any part of the waterbody as well. To protect zooplankton such as *Daphnia*, ORS recommends that **with applications at or above about 23 lb/acre-foot for GreenClean Pro and at or above about 2 gallons per surface acre-foot for GreenClean Liquid, no greater than 1/2 of the waterbody area be treated in any particular year.** The 1/2 area cutoff has not been derived quantitatively but instead recognizes that the remaining 1/2 of the waterbody area should be able to provide stock for repopulation of potentially impacted populations in the treated area. This is particularly important with stagnant waters, which do not mix as quickly with surrounding waters. While the potential risk to fish is unclear based upon conflicting toxicity evidence, the use restrictions for zooplankton would also be protective for fish.

Juvenile amphibians and possibly young fish are particularly at risk when shorelines are treated at high application rates, especially during breeding seasons (typically in the spring) when waterbody margins are populated with tadpoles and young fry. The GreenClean Pro label warns that this product should not be used during amphibian breeding season in shallow waterbody margins. Although the concern with contact burns from crystallized product is not an issue with GreenClean Liquid, aquatic concentrations of oxidants after application, both hydrogen peroxide and peracetic acid, may be high. ORS therefore also extends the above recommendations to the GreenClean Liquid applications, to address concerns related to amphibians.

RECOMMENDATIONS

ORS recommends that these products be applied according to label instructions with the following restrictions:

With application rates equal to or greater than 23 lb per surface acre-foot for GreenClean Pro or greater than or equal to 2 gallons per surface acre-foot for GreenClean Liquid, the following measures should be taken to limit impacts to zooplankton and fish populations:

- Apply product to affected surface areas only during times when fish and other aquatic biota are at depth and away from the surface. While the GreenClean product labels advise that application should generally be made early in the day, under calm, sunny conditions when the water temperature is at least 60^{o} F, care should be taken to avoid application during prime fish feeding times, including very early in the morning. In general, mid-day application is best; avoid applying at dawn or at dusk and anytime flying insects are visibly hovering over the water's surface and/or other biota are present.

- Apply product starting from the shallows and proceeding towards the deeper waters to allow mobile biota the opportunity to move away from the treatment area.

- Only treat one-half of waterbodies at any particular time to avoid completely damaging the ecology of the waterbody due to depletion of biota and subsequent anoxia from decaying organic matter. This recommendation is especially targeted at the concern for a reduced ability of benthic biota populations to recolonize.

In addition, for all GreenClean Pro applications and for GreenClean Liquid applications greater than 5 gallons per surface acre-foot, do not apply to shallow waterbody margins in the spring and summer to protect amphibians and any susceptible juvenile fish during the breeding season.

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APPENDIX

APPENDIX. CALCULATION OF WATER CONCENTRATION RESULTING FROM MAXIMUM APPLICATION RATE FOR A POND:

GREENCLEAN PRO
(Hydrogen Peroxide)
Application rate: 90 lbs of GreenClean Pro (of which 27.6% is H_2O_2) per acre-foot of water

- **Conversions**: 1 pound = 453.59 g
  1 gallon = 3.785 liters
  1 acre-foot = 325,850.58 gallons

- **Calculate # grams in 90 pounds of product:**
  90 lb x 453.59 g/lb = 40,823.1 g GreenClean Pro

- **Calculate # grams H_2O_2 assuming is 27.6% H_2O_2:**
  0.276 x 40,823.3 g = 11, 267.24 g

- **Convert from grams to milligrams:**
  11,267.24 g = 11,267,240 mg H_2O_2

- **Calculate # liters in an acre-foot:**
  1 acre-foot = 325,851 gallons x 3.785 liters/gallon = 1,233,346.035 liters

- **Calculate H_2O_2 concentration resulting from a 90 lb per acre-foot application rate:**
  11,267,240 mg/1,233,346.035 liters = **9.1 mg/L H_2O_2**

- **Calculate H_2O_2 concentration resulting from a 9 lb per acre-foot application rate:**
  9.1 mg/L H_2O_2 / 10 = **0.9 mg/L H_2O_2**

GREENCLEAN LIQUID
(Hydrogen Peroxide)
Application rate: 1.2 gallons of GreenClean Liquid per acre-foot of water

- **Conversions**: 1 gallon = 3.785 liters
  1 acre-foot = 325,850.58 gallons

- **Density of GreenClean product (from MSDS) (assume is roughly equivalent to 27% GreenClean solution)**: 1090 g/L H_2O_2

- **Calculate # grams of product in 1.2 gallons:**
  1.2 gallons x 3.785 liters/gallon x 1090 g/L = 4950.78 g

- **Calculate # grams H_2O_2 in 4950.78 g product by weight (assuming 27% weight):**:
  4950.78 x 0.27 = 1336.710 g (1,336,710 mg) H_2O_2

- **Calculate # liters in an acre-foot:**
  1 acre-foot = 325,851 gallons x 3.785 liters/gallon = 1,233,346.035 liters

- **Calculate H_2O_2 concentration resulting from a 1.2 gallon per acre-foot application rate:**
  1,336,710 mg/1,233,346.035 liters = 1.0838 = **1.08 mg/L**

- **Calculate H_2O_2 concentration resulting from a 2.4 gallon per acre-foot application rate:**
  1.0838 milligrams/liter x 2 = 2.167 mg/L = **2.17 mg/L**

(Peracetic Acid)
Application rate: 1.2 gallons of GreenClean Liquid per acre-foot of water

- **Conversions**: 1 gallon = 3.785 liters
  1 acre-foot = 325,850.58 gallons

- **Density of GreenClean <u>product</u> (from MSDS) (assume is roughly equivalent to 2% GreenClean solution)**: 1090 g/L H_2O_2

- **Calculate # grams of product in 1.2 gallons:**
  1.2 gallons x 3.785 liters/gallon x 1090 g/L = 4950.78 g

- **Calculate # grams H_2O_2 in 4950.78 g product by weight (assuming 2% weight):**:
  4950.78 x 0.02 = 99.0156 g (99,015.6 mg) H_2O_2

- **Calculate # liters in an acre-foot:**
  1 acre-foot = 325,851 gallons x 3.785 liters/gallon = 1,233,346.035 liters

- **Calculate H_2O_2 concentration resulting from a 1.2 gallon per acre-foot application rate:**
  99,015.6 mg/1,233,346.035 liters = 1.0838 = 0.0802 **= 0.08 mg/L**

- **Calculate H_2O_2 concentration resulting from a 2.4 gallon per acre-foot application rate:**
  0.0802 milligrams/liter x 2 = 0.1604 mg/L = **0.16 mg/L**