



Pilot Test Report

Milking R Inc. Dairy Wastewater Treatment

December 2013

Protected by U.S Patents

7,699,994; 7,699,988; 7,785,470; 7,943,087

Patents Pending

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CERTIFICATION PAGE

The information presented in this report documents the pilot test results completed by Ecosphere Technologies, Inc. on the treatment of wastewater at the Milking R Dairy in Okeechobee, FL. I certify that all work and experimental procedures were completed under my direct supervision and this report provides reasonable assurances that laboratory standard operating collection procedures were followed and the analytical results presented in this report have not been altered or changed and are true to the best of my knowledge.

Prepared/Reviewed By:

Brendon Blüm, P.E.
FL License # 73006
Project Engineer
Ecosphere Technologies, Inc.

Date: _____

1. PROJECT DESCRIPTION AND OBJECTIVE

1.1 Company History

Ecosphere Technologies, Inc. is a water engineering, technology licensing and innovative manufacturing company that develops non-chemical water treatment solutions for industrial markets. The Company is a leader in emerging advanced oxidation processes and has an extensive portfolio of intellectual property that includes five United States ("U.S.") patents for the Ecosphere Ozonix® process.

Water is a vital component in our customers' production processes and scarce water resources and environmental regulations can limit their ability to bring clean energy to market. Our goal is to help clean energy producers gain more control over their water resources, quality and completion costs by providing effective mobile water recycling solutions. Since 2008, Ecosphere Technologies, Inc. has commercialized its patented technology and water know-how to the oil and natural gas industry allowing energy companies to treat and recycle water used during oil and gas well drilling and completion operations.

Ecosphere Technologies Inc. has been a water industry innovator since 1998, when company founders began developing eco-friendly technologies to solve major water remediation challenges on land and at sea. Our commitment to clean water includes ongoing investment in research and development of advanced water treatment processes.

Recent events in history made our development of water filtration technology for mobile delivery systems extremely relevant. The tragedies of 9/11, the Banda Aceh Tsunami and Hurricane Katrina, showed the magnitude of suffering when clean water supplies are compromised. During those years, the federal government enacted legislation calling for the nation's 8552 municipalities to have emergency backup water systems in place. The EPA tested and verified our water filtration and purification technology to treat waters contaminated by biological pathogens and other toxic substances. We also developed a mobile emergency water filtration system for first responder organizations and deployed the first unit to Waveland, Mississippi to provide safe drinking water for thousands of people in Katrina's aftermath.

Ecosphere Technologies' most recent 'eco-technology' breakthrough is Ozonix®, a revolutionary advanced oxidation process that is currently being used by energy exploration companies to reduce costs, increase treatment efficiencies and eliminate liquid chemicals from wastewater treatment operations around the United States. We have established eco-alliances with leaders in business, industry, government and environmental organizations. Together, we are developing technologies and services to ensure clean water availability in emergency disaster situations and to enable more environmentally responsible water management practices for industry.

1.2 Dairy Site Description

The Site is located on the northeast side of Highway 98 North in Okeechobee, FL (**Figure A**). The site location is a large dairy farm with 3 separate wastewater lagoons that receive washing wastewater from the dairy barns. A fourth lagoon, the largest, is an approximately 100 acre rain collection wetland.

Figure A – Site Area Map

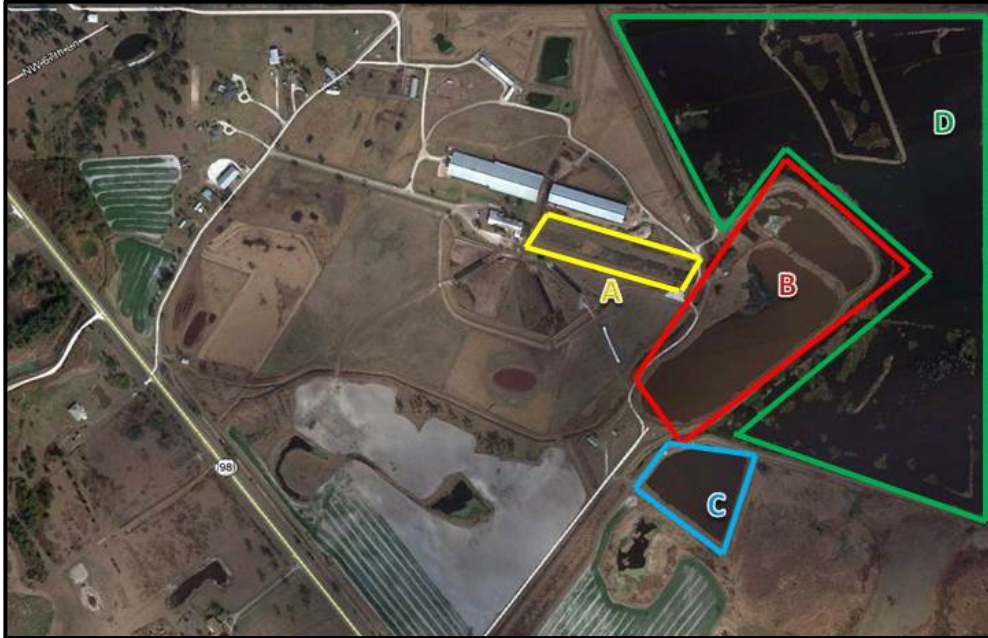


Site Address = Milking R Dairy
6300 Hwy 98 N
Okeechobee, FL 34972

Site Owner = Sutton Rucks, 863-634-7113

1.3 Site Background Information

Location and description of water management ponds



The Milking R site is an active dairy operation with more than 1,200 cows and utilizes 3 working water management ponds (Pond A, Pond B, Pond C) and one 100 acre lagoon for rain water containment. The process starts in Pond A where washed down cow manure and debris (feed, bedding material, etc.) is mixed with water and is received from all 3 cow barns and living areas. This pond is periodically cleaned of sludge and is spread as crop fertilizer. Pond B is a secondary settling pond that is supplied over a weir pipe from Pond A. This water is held until it is pumped to Pond C which supplies the feed and housing barn with a constant supply of wash cycle water to repeat the process. (The milking barn always uses fresh well water for wash down scenarios for sanitary reasons.) The lagoon Labeled D was constructed with the assistance of the South West Florida Water Management District (SFWMD) for rain water retention and crop irrigation. The farm manages all site water run off using these 4 areas.

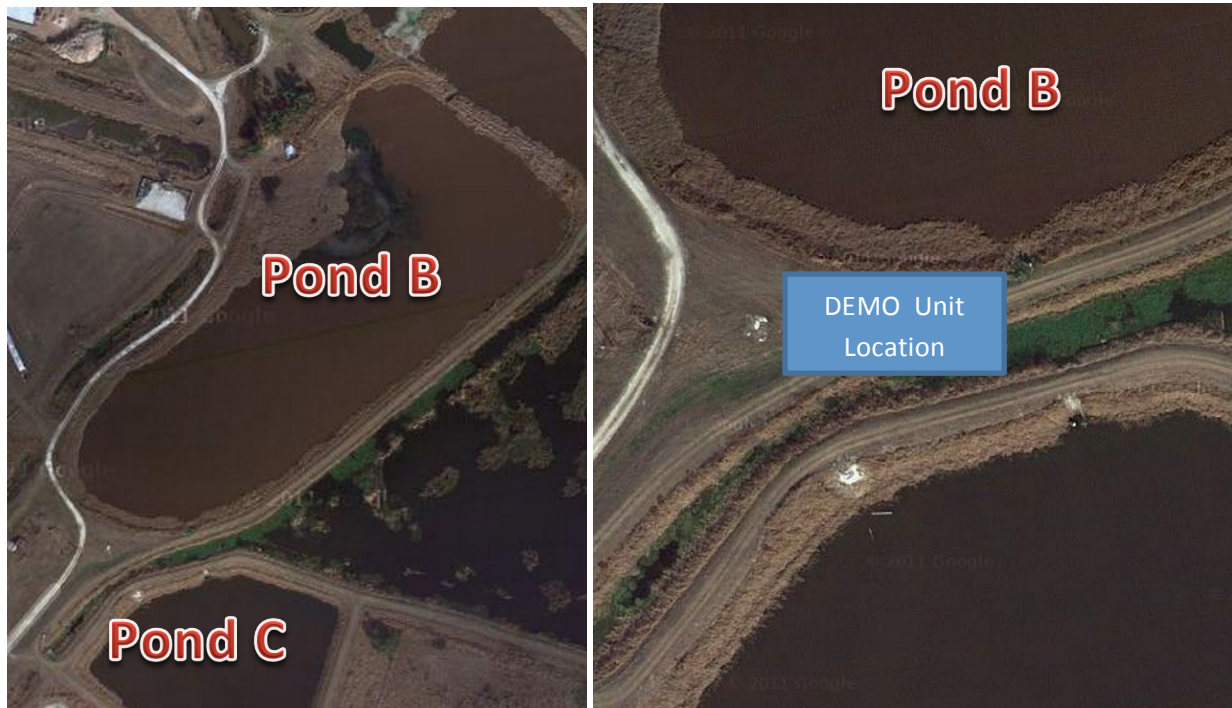
Pond Label A	Initial sludge flow from all barns	Worst Case – Dredged and sold as fertilizer
Pond Label B	Secondary settling pond	Supplied from pond A via weir system
Pond Label C	Tertiary settling pond	Source for feed barn cleansing operations
Lagoon Label D	Manmade rain water holding lagoon / ground water mainly	Source for crop watering, and holding rain water. Some pumping from Pond Label C

Close up of barns and possible setup arrangement



Label 1,2,3,4	Wash down tanks from Pond C	Source of water is Pond C
Label 5	Primary sludge pond A	Receives wash water from barns

Close up of setup by pond B



Pond B	Source of wash down water	Great source of influent, close to pond and away from barn activity.
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1.4 Objective

The focus of this pilot test is to determine the effectiveness of the Ozonix® treatment process on lowering the levels of TSS, BOD, Fecal Coliforms, Total Coliforms, nitrogen species, and also phosphorus in the second settling wastewater pond (Pond B). The Ozonix® system will be integrated into the site as to not disturb the facilities daily activities. Influent and Effluent water samples will be collected and analyzed to determine treatment efficiencies.

2. WASTEWATER TREATMENT TECHNOLOGY

The following section will explain the Ozonix® technology as well as explain the Ozonix® Demonstration unit.

2.1 Overview of the Ecosphere Ozonix® Technology

Ozonix® is a patented chemical free water treatment technology that is categorized as an advanced oxidation process (AOP) that creates highly reactive hydroxyl radicals to disinfect water. Ozonix® was designed to maximize the creation of hydroxyl radicals by combining hydrodynamic cavitation, ozone disinfection, acoustic cavitation and electrochemical oxidation. A five (5) to twenty (20) increase in disinfection potential can be achieved by using Ozonix® compared to other treatment technologies.

2.1.1 Hydrodynamic Cavitation

The first step in the patented Ozonix® process is hydrodynamic cavitation. Hydrodynamic Cavitation is a physiochemical process (physical process that acts as a catalyst for a chemical reaction) that creates the highly reactive hydroxyl radicals (oxidation potential: 2.80V). The cavitating bubble produces localized hot spots, which causes sonochemical reactions that create hydroxyl radicals. Disinfection due to hydrodynamic cavitation happens in a matter of nanoseconds. The Ozonix® process maximizes the efficiency of hydrodynamic cavitation through the use of patent pending static mixers. The static mixers use a proprietary hole design that maximize the amount of cavitating bubbles that are being created and resulting in an increasing amount of hydroxyl radicals.

2.1.2 Ozone Injection

After hydrodynamic cavitation, ozone is injected into the system. Ozone has an oxidation potential of 2.07V allowing it to oxidize a wide range of pollutants. Ozone kills bacteria in the water through a process called lysis; this is a process where ozone will penetrate the cell wall and oxidize all the essential components such as DNA. Ozonix® injects ozone into the system using a venturi; the use of a venturi combined with the other oxidation processes maximizes the mass transfer efficiencies of ozone thus allowing less to be used than typical ozone disinfection processes. It is important to note that while ozone kills the bacteria it also eliminates the food source that makes it nearly impossible for bacteria to regrow.

2.1.3 Acoustic Cavitation

After the ozone is injected, the next step in the Ozonix® process is the Ozonix® reactor that consists of simultaneous treatment of acoustic cavitation and electrochemical oxidation. Acoustic cavitation uses ultrasonic waves to induce cavitation in the water. Similar to hydrodynamic cavitation, acoustic cavitation produces localized hot spots causing sonochemical

reactions to occur thus producing hydroxyl radicals. The Ozonix® process uses multiple wavelengths to maximize the intensity of cavitation.

2.1.4 Electrochemical Oxidation

The additional process in the Ozonix® reactor is electrochemical oxidation. Electrochemical oxidation uses electricity to create hydroxyl radicals. Ozonix® uses specialty coated electrode rods to achieve electrochemical oxidation; the proprietary coating was chosen because of its longevity and its ability to maximize production of hydroxyl radicals. This area of the Ozonix® process is responsible for the removal of nitrogen species and other metals.

3 Ozonix® Demonstration Unit Specifications

The Ozonix® Demonstration Unit is a 150 GPM mobile water treatment pilot system that is fully automated by a PLC allowing easy integration into any water treatment facility. The demonstration unit was design based on the successful Ozonix® EF80 unit that is being used by numerous energy exploration and production companies in the oil and gas industry. Currently Ecosphere Technologies has manufactured and deployed over 40 Advanced Oxidation Ozonix® units in over 800 wells sites and has treated over 3 billion gallons of water. Table B below shows the design parameters of the Ecosphere Ozonix® Demonstration unit. In addition to the parameters shown below, the Ozonix® Demonstration unit has a diesel generator to run power to the system in an off-grid application, but also has the ability to run shore power (1000 A 208V) and bypass the generator.

Table B: Demo Unit Design Parameters

Design Parameters	Ozonix® Demo Unit
Flow Rate	150 GPM
Residence Time (reactor)	34 sec
Residence Time (system)	5.5 min
Current of each rod	100 Amps
Number of Reactors	2

4. PILOT TEST AND WATER SAMPLING PROCEDURES

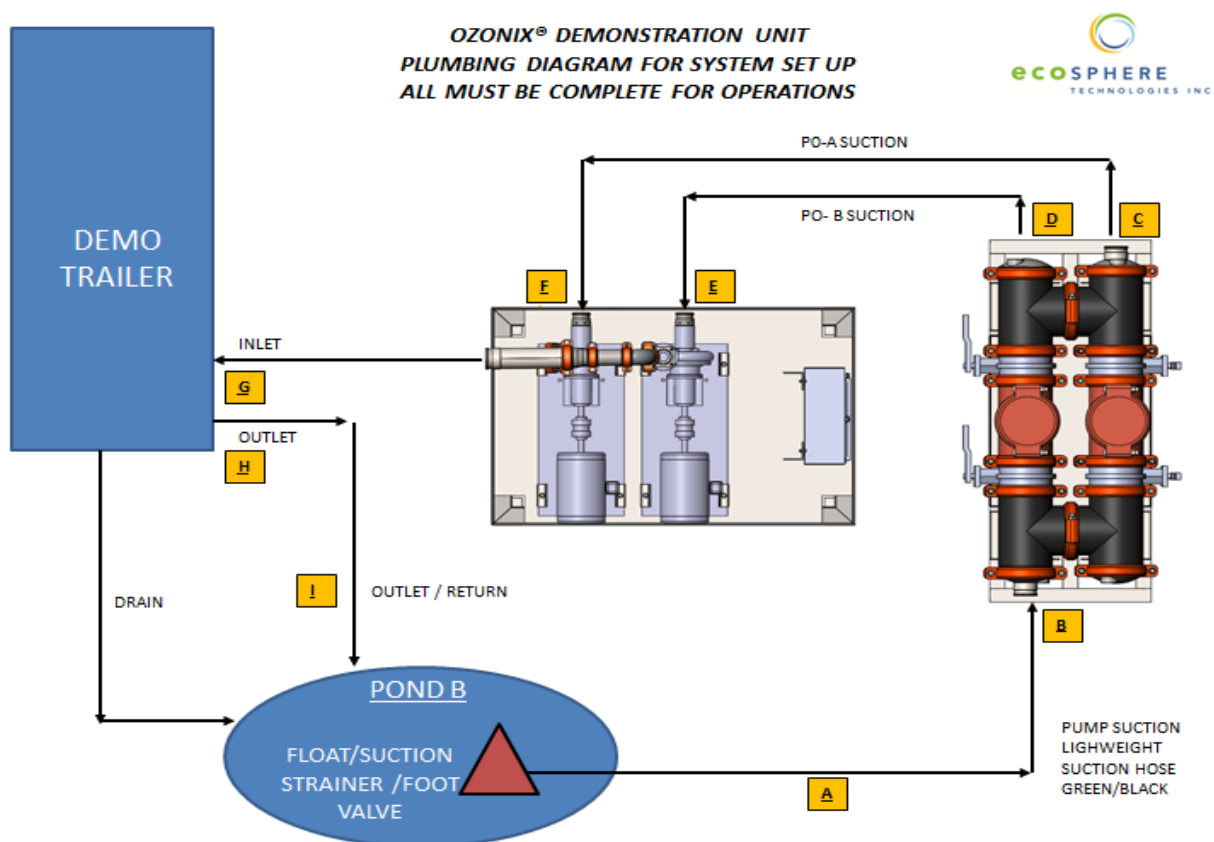
4.1 Baseline Water Sampling

On November 16, 2013 prior to the Pilot Test, Ecosphere Technologies, Inc. (Ecosphere) collected 3 surface water samples from Pond A, Pond B, and Pond C to provide baseline water quality data from each pond.

Samples (Pond A, Pond B, and Pond C) were collected from approximately 0 to 1 ft below the water's surface. The samples were properly collected, labeled, placed on ice and sent to Florida Spectrum Environmental Services, Inc. (Spectrum) located in Ft Meade, FL for laboratory analysis of Carbonaceous Biological Oxygen Demand (CBOD) (Method SM5210B), Fecal Coliforms (9222D), Total Coliforms (9223B), Total Suspended Solids (SM2540D), Nitrate (300.0), Nitrite (300.0), Ammonia (350.1), Total Kjeldahl Nitrogen (351.2), Total Nitrogen (300+351), Total Phosphorus (365.1), Sulfate (300.0), Bromide (300.0), Ortho-Phosphate (300.0), Alkalinity (SM2320B), Total Dissolved Solids (TDS) (SM2540C), Boron (200.7), Calcium (200.7), Iron (200.7), Magnesium (200.7), Potassium (200.7), Sodium (200.7), Strontium (200.7) Chloride (300.0) and Fluoride (300.0). A copy of the laboratory analytical data reports is included in **Appendix A** and data is shown on **Table 1**.

4.2 Equipment Set-up

On November 18, 2013, Ecosphere mobilized to the site to receive and integrate all pilot test equipment to allow treatment in Pond B as detailed in the following process diagram:



Pilot Test equipment consisted of the following:

- Ozonix® Demonstration Trailer
- 45 kw Diesel Generator
- Supply Pump Skid
- Filter Strainer (3/8")
- Approx. 200 ft of 4" suction/discharge hose

4.3 Test Procedure

On November 19, 2013 The Ozonix® Demo Unit was staged with the ability to treat influent water from Pond B through the system in either "single pass" treatment mode or with the ability to recirculate approximately 1,500 gallons of influent water for an indefinite amount of time. Influent water was pulled from a floating strainer in Pond B through a supply pump skid capable of feeding the Demo unit an approximate 130 gpm water feed. Water was then treated through the Ozonix® Demo Unit and the effluent water was discharged back into Pond B at a sufficient distance away from the influent strainer as not to interfere with the influent water stream characteristics.

Ecosphere chose to treat influent water in three separate test environments:

1. The first test of influent water was "single pass" treatment with system treatment parameters set at 25% of maximum treatment capability. (As characterized by Sample #1 and Sample #2 in Section 4.4)
2. The second test of influent water was "single pass" treatment with system treatment parameters set at 50% of maximum treatment capability. (As characterized by Sample #3 and Sample #4 in Section 4.4)
3. The third test of influent water was a batch recirculation of approximately 1,500 gallons of influent at 100% system treatment capability. This batch was treated for approximately 120 minutes. (As characterized by Sample #5 through Sample#9 in Section 4.4)

During the treatment the following parameters were recorded and logged in the systems Programmable Logic Controller (PLC): Sensor data for corresponding sample collection times is included in **Table 1**.

Influent and Effluent Sensors:

- Temperature (F)
- ORP (mV)
- Conductivity (µS/cm)
- pH
- Flow Rate (gpm)

4.3.1 Oxidation Reduction Potential (ORP)

Oxidation Reduction Potential (ORP) is a measurement of the activity, or strength, of a solution's ability to oxidize or reduce species in a given solution. ORP is measured in millivolts (mV) where a higher positive number indicates a solution's ability to oxidize (attract electrons) and a negative number indicates the ability to reduce (give electrons). For example, a chlorine solution will exhibit a positive ORP value (oxidizing agent) and sodium sulfite will lose electrons and show a negative ORP value (reducing agent). ORP is a common measurement in water treatment and disinfection. For example, in microbial terms high ORP water (oxidizing) will pull electrons away from a cell's membrane leading to destruction of the integrity of the cell wall and death of the cell. Typical ORP values for municipal drinking water are 250-300 mV range. Highly disinfected water will show ORP values above 650 mV. Anaerobic wastewater is in the -100 to -200 mV range.

4.3.2 Conductivity

Conductivity is the measurement of a solution's ability to conduct electricity. Conductivity is measured in $\mu\text{S}/\text{cm}$. Deionized water will have a conductivity value of 0.05 $\mu\text{S}/\text{cm}$ whereas seawater will be approximately 54,000 $\mu\text{S}/\text{cm}$.

4.4 Sampling Procedure

Influent Sample Collection

Samples (Sample #1 and Sample #3) were collected at the influent sample port located on the influent piping of the Demo Unit prior to contact with any treatment operations. Influent samples were collected to compare with baseline sampling event of Pond B completed prior to the Pilot Test to provide three sets of influent water quality analytical data points.

Effluent Sample Collection

Samples (Sample #2, Sample #4, Sample #5, Sample #6, Sample #7, Sample #8, and Sample #9) were collected at the effluent sample port located on the system discharge piping prior to discharging back into Pond B. Sample #2 and Sample #4 represent "Single Pass" treatment data. Samples #5 through Sample #9 represent "Recirculation" batch treatment and samples were collected on approximate 15 and 30 minute timed intervals or when a noticeable water color change was observed.

Samples (Sample #1 through Sample #9) were properly collected, labeled, placed on ice and sent to Florida Spectrum Environmental Services, Inc. (Spectrum) located in Ft Meade, FL for laboratory analysis of Carbonaceous Biological Oxygen Demand (CBOD) (Method SM5210B), Fecal Coliforms (9222D), Total Coliforms (9223B), Total Suspended Solids (SM2540D), Nitrate (300.0), Nitrite (300.0), Ammonia (350.1), Total Kjeldahl Nitrogen (351.2), Total Nitrogen (300+351), Total Phosphorus (365.1), Sulfate (300.0), Bromide (300.0), Ortho-Phosphate (300.0), Alkalinity (SM2320B), Total Dissolved Solids (TDS) (SM2540C), Boron (200.7), Calcium (200.7), Iron (200.7), Magnesium (200.7), Potassium (200.7), Sodium (200.7), Strontium (200.7) Chloride (300.0) and Fluoride (300.0). A copy of the laboratory analytical data reports is included in **Appendix A** and data is shown on **Table 1**.

5 RESULTS

5.1 Pilot Test System Data

The following chart shows the real-time data measured directly from the systems PLC as each sample was taken:

Table A. System Data Parameters

Sample ID	Location	pH	ORP(mV)	Conductivity (µS/cm)	Temp (F)
Sample #1	Influent	8.4	-3.7	3333	80.9
Sample #2	Effluent	8.1	58.1	3288	74.9
Sample #3	Influent	8.4	-30.4	3320	81.2
Sample #4	Effluent	8.2	55.3	3223	75
Sample #5	Effluent	8.2	40.2	3100	75.1
Sample #6	Effluent	8.0	44.4	3029	81.9
Sample #7	Effluent	7.9	62.5	3113	87.5
Sample #8	Effluent	7.8	55.3	3243	92.5
Sample #9	Effluent	7.8	36.3	3113	105.5

The average influent water quality (Sample #1, and Sample #3) had a pH of 8.4, conductivity of 3327 µS/cm, ORP -17.1 mV, and Temperature of 81 F.

5.2 “Single Pass” Treatment Results

Test #1

“Single Pass” treatment data (Sample #2) represents a decrease in pH of 0.3 units, a 61.8 mV increase in ORP, a 1.4% reduction of conductivity, and a 7.4% decrease in temperature.

Test #2

“Single Pass” treatment data (Sample #4) represents a decrease in pH of 0.2 units, an 85.7 mV increase in ORP, a 2.9% reduction of conductivity, and a 7.6% decrease in temperature.

Test #3

“Recirculation” treatment data (Sample #5 through Sample #9) represents an overall decrease in pH of 0.4 units, a maintained ORP level increase and a temperature rise as time increased.

5.3 Water Treatment Results

Laboratory analytical results from baseline sampling collection completed on November 6, 2013 and the pilot test completed on November 19, 2013 is summarized on **Table 1** and laboratory analytical data reports are provided in **Appendix A**.

Based on the results in **Table 1**, the best treatment efficiencies were observed during the 'Recirculation' test (Samples #5 through Samples #9) and a summary of key analyzed constituents for these samples is discussed below:

Color

Effluent water samples showed a noticeable color decrease to the naked eye throughout the pilot testing. A direct relationship between treatment time and decrease in color was observed along with a reduction in turbidity.

Image A – Sample Color Difference



Influent Raw (rightmost), Effluent water (Right to Left) over ~120 minutes of treatment

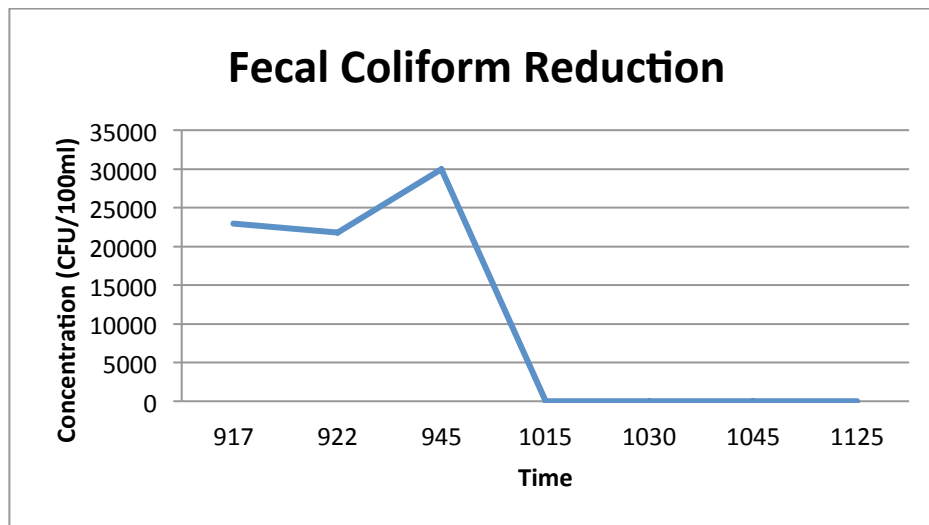
Fecal Coliform

Influent

Influent samples, Pond B, Sample# 1 and Sample #3, indicated concentrations of 42,000, 20,000 and 22,900 CFU/100 ml, respectively.

Effluent

The greatest reduction to below laboratory detection levels was reported in Sample #6, Sample #7, Sample #8, and Sample #9 at a value of 1.0 U. (The “U” denotes the laboratory reported value was below the Method Detection Limit.)



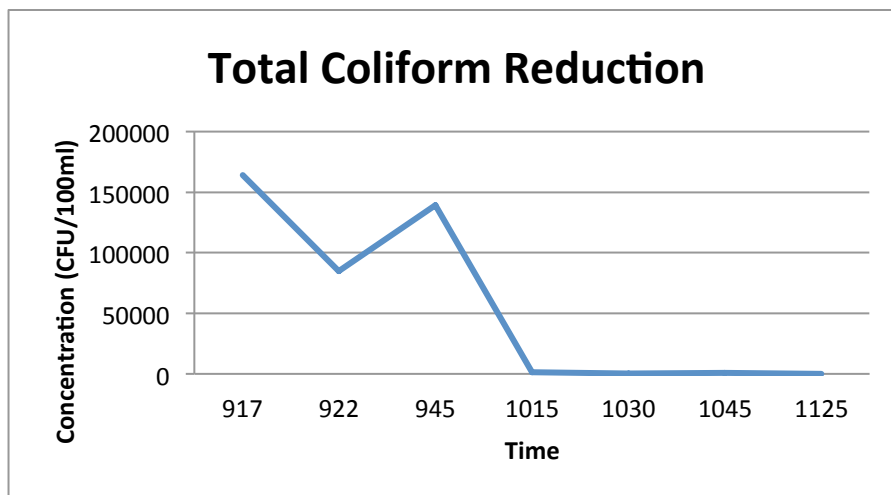
Total Coliform

Influent

Influent samples, Sample# 1 and Sample #3, indicated concentrations of 158,000 Q and 164,000 Q CFU/100 ml, respectively. (The “Q” denotes the sample was held beyond a 6-hr hold time. The local laboratory branch in Okeechobee was not equipped to run Total Coliform testing and the sample was shipped to the Ft. Lauderdale location to complete testing. However, it is important to note the Total Coliform results and reduction amounts are very similar to those seen in the Fecal Coliform and CBOD sample sets. Additionally, studies have shown that Coliform sampling by common methods beyond the hold time yield similar and comparable results to samples collected within the standard hold times provided the sample was collected and cooled below 10°C (Pope et al.)¹. All samples collected by Ecosphere were properly collected and immediately placed in a cool environment until reaching the laboratory for testing. Based on the above, Ecosphere believes that the Total Coliform data is suitable for the purpose of this report and resulting discussions.)

Effluent

The greatest reduction to below laboratory detection levels was reported in Sample #9 at a value of 100 QU. An immediate 48% reduction in Total Coliform was also observed in “single pass” treatment Sample #4 accompanied by a drastic and steady decline throughout sample collection.



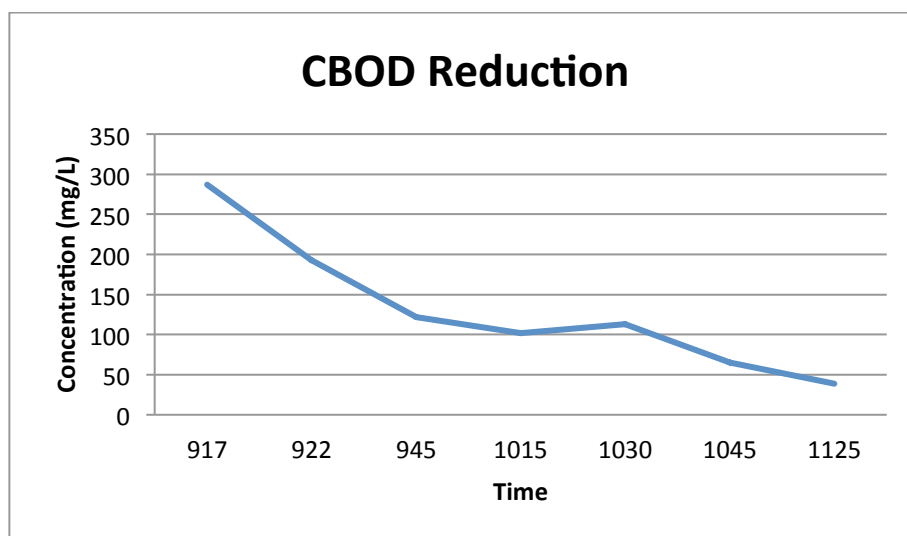
Carbonaceous Biological Oxygen Demand (CBOD)

Influent

Influent samples, Pond B, Sample# 1 and Sample #3, indicated concentrations of 471, 285, and 287 mg/L, respectively.

Effluent

The greatest reduction was observed in Sample #9 at a concentration of 38.4 mg/L. A steady linear decline in CBOD concentration was observed throughout sample collection.



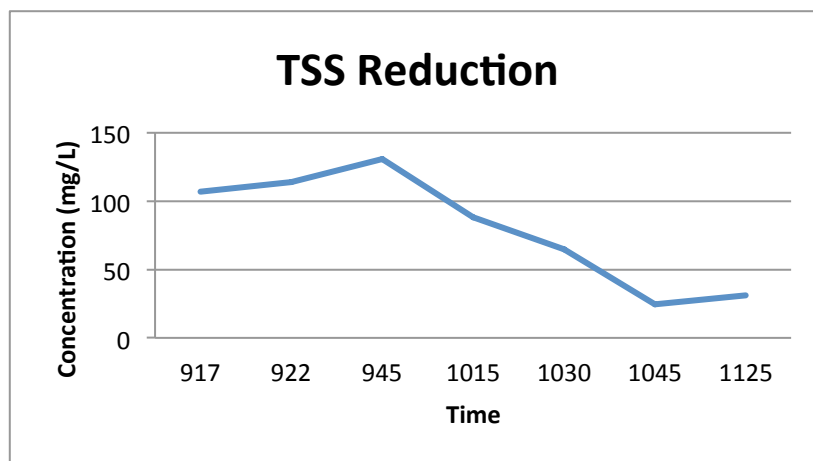
Total Suspended Solids (TSS)

Influent

Influent samples, Pond B, Sample# 1 and Sample #3, indicated concentrations of 116, 83, and 107 mg/L, respectively.

Effluent

The greatest reduction was observed in Sample #9 at a concentration of 31.0 mg/L. A small spike in TSS concentration was immediately observed during treatment followed by a steady linear decline in TSS concentration.



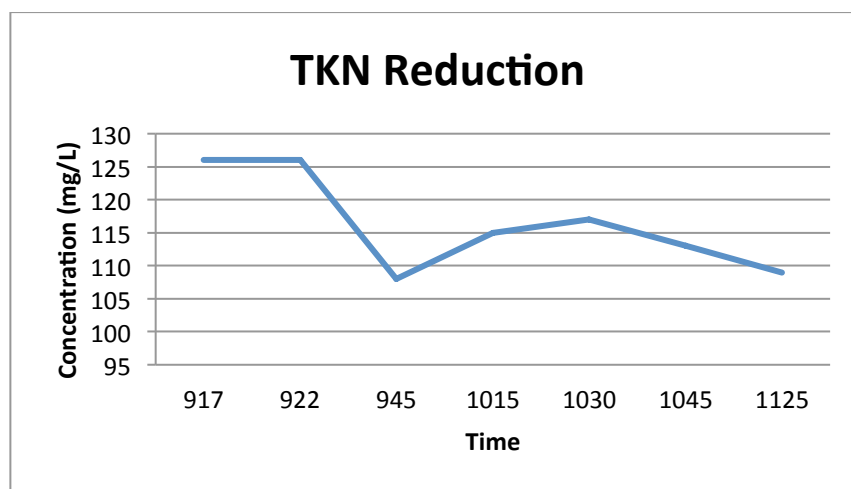
Total Kjeldahl Nitrogen (TKN)

Influent

Influent samples, Pond B, Sample# 1 and Sample #3, indicated concentrations of 123, 128, and 126 mg/L, respectively.

Effluent

The greatest reduction was observed in Sample #9 at a concentration of 109 mg/L. A small, but steady, decline in TKN concentration was observed throughout sample collection.



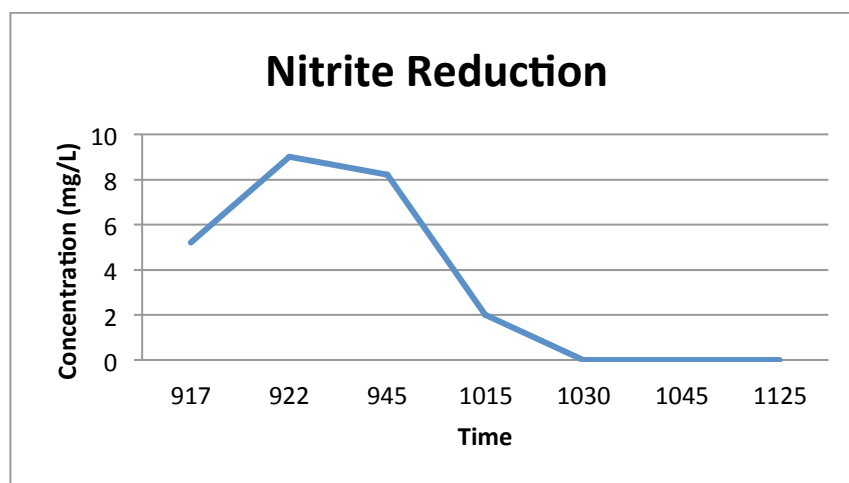
Nitrite

Influent

Influent samples, Pond B, Sample# 1 and Sample #3, indicated concentrations of 0.420 U, 3.20, and 5.20 mg/L, respectively.

Effluent

The greatest reduction was observed to below laboratory detection levels in Sample #7, Sample #8, and #9 at a concentration of 0.420 U mg/L.



Nitrate

Nitrate concentrations were not observed above laboratory detection limits.

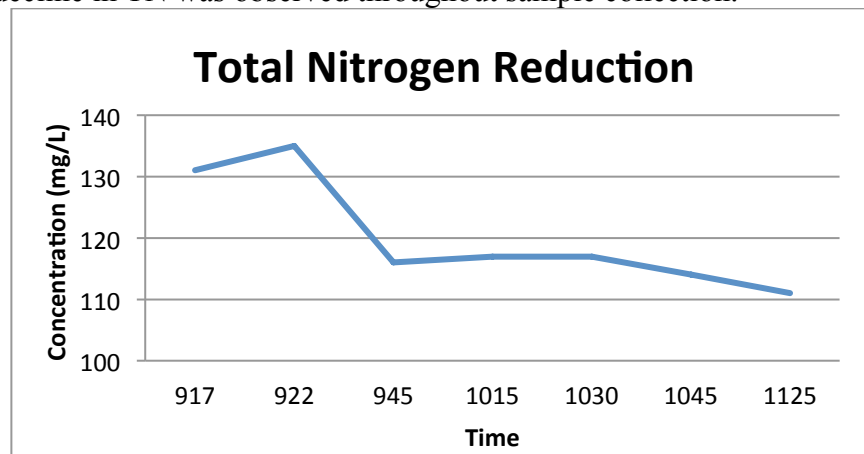
Total Nitrogen

Influent

Influent samples, Pond B, Sample# 1 and Sample #3, indicated concentrations of 123, 131, and 131 mg/L, respectively.

Effluent

The greatest reduction was observed in Sample #9 at a concentration of 111 mg/L. An overall steady linear decline in TN was observed throughout sample collection.



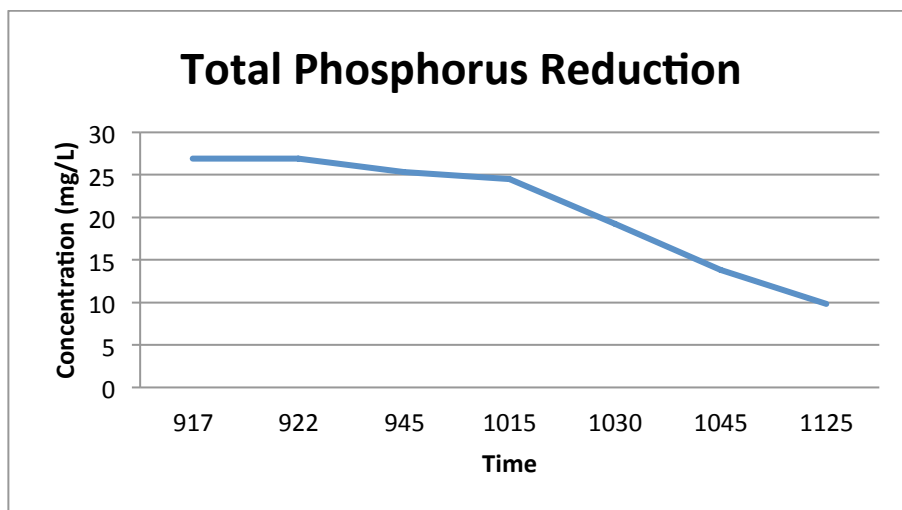
Total Phosphorus

Influent

Influent samples, Pond B, Sample# 1 and Sample #3, indicated concentrations of 25.6, 27.2, and 26.9 mg/L, respectively.

Effluent

The greatest reduction was observed in Sample #9 at a concentration of 9.82 mg/L. A steady linear decline in Total Phosphorus concentration was observed throughout sample collection.



The following table summarizes the greatest reduction percentage for key contaminants of concern:

Table B – Greatest Percentage Reduction of Contaminants of Concern

<u>Fecal Coliform</u>	<u>Total Coliform</u>	<u>CBOD</u>	<u>TSS</u>	<u>TKN</u>	<u>Nitrite</u>	<u>Total Nitrogen</u>	<u>Total Phosphorus</u>
99.99%	99.94%	86.6%	71.0%	14.8%	91.9%	15.3%	63.9%

6 Discussion and Recommendation

Ecosphere Technologies, Inc. (Ecosphere) completed a comprehensive testing and analysis on wastewater that is generated at the Milking R Dairy facility in Okeechobee, FL. Ecosphere treated wastewater that is generated from periodic daily wash down procedures of the cow barns located on site. The wash down wastewater is settled in a series of wastewater Ponds (Pond A, Pond B, and Pond C) before the water is reused from Pond C in daily cow barn wash down procedures. Ecosphere treated water from Pond B through the patented Ozonix® technology includes hydrodynamic cavitation, acoustic cavitation, ozone, and electrochemical oxidation. Completed tests were documented and samples were sent to a certified laboratory for analytical data analysis.

The main contaminants of concern for the dairy industry are Coliform bacteria, Total Suspended Solids (TSS), Total Nitrogen, Nitrate, Ammonia, and Total Phosphorus. These water quality parameters are important measurements that directly impact the health of the cows, the site specific nutrient management plans regulating Nitrogen and Phosphorus uptake levels in crops, and also the groundwater and nearby surface water runoff areas.

Ecosphere completed testing on wastewater classified as the secondary settling pond (Pond B) at the Milking R Dairy in Okeechobee, FL. The best results were achieved when influent wastewater was continuously re-circulated in the Ozonix® Demo Unit. An obvious color change was observed within the first 15 minutes from a raw dark brown to a light green color that progressed to a clear white color after 120 minutes of treatment. Analytical data results supporting the color change indicate the reduction of many contaminants of concern increased steadily and linearly over time. An over 99.9% reduction in Fecal and Total coliform, 86.6% reduction in CBOD, 71.0% reduction in TSS, 14.8% reduction in TKN, 15.3% reduction in Total Nitrogen, 91.9% reduction in Nitrite, and a 63.9% reduction in Total Phosphorus was observed in effluent Sample #9 which represents treatment recirculation in the Ozonix® Demo Unit for approximately 120 minutes.

Test results indicate a positive reduction in key contaminants that are increasingly becoming a water management problem for the dairy industry worldwide. Based on the reductions of these key contaminants, Ecosphere recommends a full scale system installation at a dairy facility utilizing the Ozonix® treatment process as a cost effective, environmentally friendly solution to improve the cleanliness of dairy facilities and the health of the animals.

7 References

- 1.) Pope, et al. **Assessment of the Effects of Holding Time and Temperature on *Escherichia coli* Densities in Surface Water Samples.** Appl Environ Microbiol. 2003 October; 69(10): 6201–6207.

TABLE

APPENDIX A

Laboratory Analytical Data Reports