August 18, 2017

Ohio Lake Erie Commission
347 North Dunbridge Road
Bowling Green, Ohio 43402

Dear Ohio Lake Erie Commission:

Enclosed you will find the final report for the Lake Erie Protection Fund (LEPF) grant that supported the “Evaluation of runoff from manure treated agricultural plots” study. The LEPF money supported an existing study funded by Ohio Water Development Authority (OWDA) that was delayed due to unforeseen obstacles. The unexpected delays in the project made extra funding necessary to complete the original project goals. The LEPF funding allowed for the necessary additional water sampling and lab-scale testing required for successful completion of the project. These assessments were critical in the planning of the pilot test and in the evaluation of the runoff during the pilot test.

Thanks to the generous funding from the LEPF, we were able to start a pilot test in May 2017 to determine the effectiveness of a low cost dewatering treatment process for dairy Concentrated Animal Feeding Operations (CAFO) manure that will reduce nutrient runoff from land application and serve as a fertilizer for crops. The pilot test will continue through the end of 2017 and will involve runoff sampling and nutrient analysis. Our hypothesis is that the treated manure will contribute less nutrients to runoff than raw, untreated liquid manure. Initial results from the pilot test do suggest that the treated manure does not release nutrients as quickly as raw manure. The results will continue to be analyzed and will be reported on at the conclusion of the pilot test.

Sincerely,

W. Robert Midden, PhD
Assoc. Prof. of Chemistry
Assoc. Vice Provost
Technical Report

Activities and Timeline:
Overall, the research team was able to accomplish the proposed activities in the timeline given in the proposal. A few activities had to be modified slightly due to findings after the research was proposed but nothing significant. The goal of this grant was to help the research team collect necessary background data to plan and execute a field pilot test. The team was able to do that, and the pilot test is still running at the conclusion of this grant. The grant funding was actually received earlier than was expected, so several activities that were proposed in certain quarters ended up being done in a later quarter. It did not affect whether or not the activities were actually accomplished, just in which quarter they were completed.

Quarter 1 August - October 2016:
1. Collect runoff samples from test plots to establish baseline nutrient conditions in runoff
   • 16 automated samplers operated at the sample collection building at the Northwest Agricultural Research Station (NWARS), programmed to collect 600 mL water samples from surface and subsurface runoff from 8 test plots during rain events.
   • Baseline water samples were collected from rain events on August 12 - 16, 2016, September 17 - 18 and October 20-21, 2016.
   • The collected water samples were analyzed for dissolved ortho-phosphate, ammonia and nitrate and total phosphorus and nitrogen.
     o Ortho-phosphate levels – all but three samples were below 0.5 mg P/L
     o Ammonia levels – all but two samples were below 1 mg N/L
     o Nitrate+Nitrite –plot averages ranged from 11 – 22 mg N/L

2. Conduct lab tests of polymers and coagulants on manure to find treatment that adequately dewater’s manure and performs well in rain/soil simulations
   • Batches of raw, liquid dairy manure that were used for lab-scale testing were analyzed for dissolved nutrients and the following averages were found:

<table>
<thead>
<tr>
<th></th>
<th>Phosphate</th>
<th>Ammonia</th>
<th>Nitrate</th>
<th>Total Solids</th>
</tr>
</thead>
<tbody>
<tr>
<td>Batch 1</td>
<td>662.08 mgP/L</td>
<td>1095.06 mgN/L</td>
<td>79.96 mgN/L</td>
<td>17.60 mg/L</td>
</tr>
<tr>
<td>Batch 2</td>
<td>522.74 mgP/L</td>
<td>1360.96 mgN/L</td>
<td>163.9 mgN/L</td>
<td>20.24 mg/L</td>
</tr>
</tbody>
</table>

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• Manure treatments using AH 1010P and Zetag 8816 allowed for the best solid/liquid separation and have decreased nutrient levels in the liquid portion of manure from batches 1 & 2 to the following averages: orthophosphate 2.0 mg P/L, ammonia 575 mg N/L and nitrate+nitrite 1.1 mg N/L.

• In order to test the way the raw manure and treated manure will perform in soil after field application, lab-scale rain simulation tests are conducted. These simulations use 120 mg of sieved soil to ensure similar particle make-up. Either raw or treated manure is added to the 120 mg of soil. The added amounts of raw or treated manure are determined based on the total phosphorus content of the manure. Once the manure is added to the soil, rains are simulated by added 100 mL of purified water. The water is allowed to run through the soil and then is collected for nutrient analysis. The soil is allowed to dry out some and then subsequent rains are added. At least ten cycles of rain are added to determine how the raw and treated manure perform in extended environmental conditions. The dissolved phosphate release and potential runoff from treated and raw manure in soil showed that treated manure did not release dissolved phosphate as quickly as raw liquid manure. The treated manure, however, did release phosphate at a slower rate that would make it available for longer periods of time for fertilizer for crops.

• The graph below shows the average amount of ortho-phosphate in runoff collected from lab-scale rain simulations. Each point on the graph was a day that 100 mL of water was added to the simulation. In this particular test, twelve, 100 mL rains were added to the soil of each simulation over a period of 21 days. The runoff was collected for analysis before each rain addition. Two simulations were set up as controls with no manure added to determine baseline ortho-phosphate concentration in the soil. Raw, liquid manure was applied to two simulations, and manure treated in the lab with AH 1010P and Zetag 8816 was applied to six simulations. The average results are shown in the graph.

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Quarter 2: November 2016 – January 2017

1. Work with the Village of Ottawa Wastewater Treatment Plant to ensure they can reproduce manure treatments at larger volumes

- A 200 gallon test batch prepared at Village of Ottawa Wastewater Treatment Plant (OWWTP) using AH 1010P and Zetag 8816 had very good solid/liquid separation and filtered well with sand bed filtration. Samples from liquid filtrate were analyzed for dissolved nutrients. The following averages were found:

<table>
<thead>
<tr>
<th>Sample Average</th>
<th>Phosphate (mgP/L)</th>
<th>Ammonia (mgN/L)</th>
<th>Nitrate+Nitrite (mgN/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>6.7</td>
<td>763</td>
<td>1.4</td>
</tr>
</tbody>
</table>

- The results below were from a rain simulation that used treated manure from the 200 gallon test batch from the OWWTP. Each point on the graph was a day that 100 mL of water was added to the simulation. In this particular test, fourteen, 100 mL rains were added to the soil of each simulation over a period of 42 days. The runoff was collected for analysis before each rain addition. One simulation was set up as a control with no manure added to determine baseline ortho-phosphate concentration in the soil. Raw, liquid manure was applied to two simulations, manure treated and filtered at the OWWTP was applied to three simulations, and manure treated at the OWWTP and filtered in BGSU’s lab was applied to three more simulations. These results again showed that the treated manure released nutrients at a slower rate than raw manure.

![Ortho-Phosphate Concentration Graph](Image)

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2. Continue evaluating baseline conditions of test plots and making necessary corrections to allow plots to function comparably

- Runoff flow was analyzed during rain events on October 20-21, 2016 and November 28, 2016. In both of these events, as well as events analyzed in previously, plot 5 showed less runoff (particularly surface) volume than the other plots. Controlled tests were run to identify what might account for the lower volume. An issue with the data collection technology was identified with plot 5’s surface collection and corrected. That plot was then identified to have similar flow to adjacent plots. Plot 2 was noted to have inconsistent flow results, particularly with surface flow. Dirt was removed from the plot to expose tiling and possible breaks, but none were found. It was determined that plot 2 has a different flow profile than other plots and cannot be paired with another plot during the pilot test. Manure will not be applied to plot 2. It will just be used to monitor background nutrient levels in the soil.

- In November 2016, the plots were tilled to re-establish grade for runoff and break up macropores that had formed over time.

- Baseline water samples were collected from events on 1/2/17 and 1/12/17.

- The collected water samples were analyzed for dissolved ortho-phosphate, ammonia and nitrate and total phosphorus and total nitrogen.
  - Ortho-phosphate levels – all but one sample were below 0.5 mg P/L. The highest ortho-phosphate levels were found in surface runoff samples. The subsurface levels were below 0.02 mg P/L.
  - Ammonia levels – all samples were below 1 mg N/L with many below detection limit.
  - Nitrate+Nitrite –plot averages ranged from 6 – 20 mg N/L for subsurface samples and 1 – 7 mg N/L for surface samples.

Quarter 3: February – April 2017

1. Continue evaluating baseline conditions of test plots and making necessary corrections to allow plots to function comparably

- Runoff flow and dissolved phosphate load were analyzed for rain events for the last year to determine if the plots produced consistent volumes of runoff and carried similar phosphate loads. Each plot was compared to the mean runoff for the event; plots that similar mean comparisons were identified as pairs for the pilot test with one plot received raw and one plot receiving treated manure. The identified pairs were 3 (treated) with 4 (raw), 5 (treated) with 6 (raw), and 7 (treated) with 8 (raw). The plots all showed variation in the different events, so the most consistent and similar plots were chosen for treatments. See Attachment 1 for flow results.

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• Plot 1 did not have a similar surface flow to any of the other plots and often had very high surface volume, so it could not be paired for treatment. Plot 1, like plot 2, will not receive manure and will just be monitored for background nutrient levels in the soil.

Quarter 4: May – July 2017

1. Village of Ottawa Wastewater Treatment Plant treats 1000 gallon batches of manure to be applied to test plots at NWARS

- During the period of 5-9-17 to 5-17-17, 4000 gallons of liquid manure were treated with AH 1010P and Zetag 8816 and allowed to filter and dry on sand beds at the Ottawa Wastewater Treatment Plant. This manure was treated for the purpose of the pilot scale test.
- Batches of raw, liquid dairy manure that were used for the pilot test were analyzed for ortho-phosphate and total phosphorus. The following averages were found:
  - Ortho-phosphate - 161.7 mg P/L or 1.4 lb P/1000 gallons of liquid manure
  - Total phosphorus – 320.6 mg P/L or 2.7 lb P/1000 gallons of liquid manure
- The manure treatments using AH 1010P and Zetag 8816 decreased the ortho-phosphate and total phosphorus in the liquid filtrate after the manure treatment used for the pilot test to the following averages:
  - Orthophosphate - 1.8 mg P/L
  - Total phosphorus – 5.4 mg P/L
- The solid portion of the manure treated for the pilot test was analyzed for total phosphorus. The following average was found:
  - Total phosphorus – 2.8 lb P/ton of dry solid treated manure cake

2. Treat two test plots with raw manure, two test plots with one treatment protocol, and two test plots with a different treatment protocol.

- This activity was modified based on the results of lab-scale testing. The lab testing showed that the treatment protocol of AH 1010P and Zetag 8816 worked far better than other protocols, so only that protocol was used for the pilot test.
- On 5-18-17 manure was applied to the test plots at the Northwest Agricultural Research Station (NWARS) with amounts calculated to apply the same amount of total P to all treated plots.
  - 800 gallons of liquid manure were applied to the surface of plots 4, 6, and 8
  - 1400 pounds of treated manure were applied to the surface of plots 3, 5, and 7
o Plots 1 & 2 did not receive manure treatment in order to serve as control plots.

3. Plant all eight test plots with corn
   • On 5-26-17 all plots were tilled in the same method, and on 6-1-17 all plots were planted with corn.

4. Collect runoff samples from test plots after manure application and plant growth
   • Runoff samples after manure application were collected during this quarter from events on 5-20-17, 5-27-17, 6-13-17, 6-23-17, 7-7-17, and 7-10-17.
     o The collected runoff samples were analyzed for dissolved phosphate, ammonia and nitrate and total phosphorus and nitrogen. While the samples were analyzed for all of the mentioned nutrients, the focus for the treatment effectiveness is dissolved phosphate since that is the target nutrient for reducing HABs.
     o The preliminary dissolved phosphate load results show that the raw manure treated plots are contributing significantly greater amounts of dissolved phosphate to the runoff water than the polymer treated manure.

Proposed Outcomes Discussion:
Outcome 1. If we collect sufficient runoff samples from plots after modifications are made, we will be able to develop a flow profile for each plot during a rain event. This will allow us to be able to quantify what nutrients are added to the runoff from our manure applications. Successful profiling of the plots will also allow us to compare the untreated, raw manure, and treated manure plots with each other.

The sample collection and flow analyses done in the first quarter of the grant identified issues with the flow from two of the plots. An issue with technology was identified with one of the plots (plot 5), and it was corrected; therefore, that plot was found to function similar to other plots. In rain events in Quarter 3, little to no surface runoff was being measured from plot 2. The issue with the plot could not be identified so that plot was designated for no manure treatment for the pilot test. See Attachment 1 for flow results.

The nutrient results from sampling events in the first and second quarter were used to determine the load of dissolved phosphate coming off the plots. See Attachment 1 for for dissolved phosphate loads. In addition to considering runoff volume from plots, dissolved phosphate load was also considered. Plots with similar baseline dissolved phosphate loads and runoff volumes were assigned as comparison pairs for the pilot test, where one plot in a pair would receive raw manure and one would receive treated manure. See Attachment 2 for plot treatments. Since the baseline dissolved phosphate loads have been determined, runoff during the pilot test can be evaluated to see how the manure is contributing to the dissolved phosphate.

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Outcome 2. If we complete our lab testing of various polymers and coagulants for dewatering manure, we will have a treatment protocol that produces a clear liquid portion from the manure and a solid cake that can be used as fertilizer. The solid portion will release nutrients at a slower rate that is more useful to plants than raw manure and will not run off as quickly during rain events.

The treatment protocol using AH 1010P and Zetag 8816 is very effective at dewatering manure. The research team has found other treatments that also dewater manure; however, they require more treatment materials resulting in a higher cost for treatment. See attachment 3 for solid/liquid separation after treatment and solid cakes produced after filtration.

As shown in the Technical Report, the rain simulations conducted to test raw manure and treated manure in soil show that the raw manure releases higher amounts of dissolved phosphate initially in runoff. This indicates that the nutrients are staying with the treated manure longer. The same results are also being noted in the pilot test. Prior to any manure application, surface runoff from the plots receiving raw manure had an average ortho-phosphate concentration of 0.38 mgP/L, and the plots receiving treated manure had 0.31 mgP/L. After respective treatment with raw or treated manure, the plots receiving raw manure had an average ortho-phosphate concentration of 0.59 mgP/L, and the plots receiving treated manure had 0.27 mgP/L. See Attachment 4 for pilot test preliminary results. Further data collection and analysis is still needed to be confident in these results, and that is funded by an OWDA grant. The pilot test will continue through the end of 2017. Funding is being sought for a second pilot test to help validate any findings from the current pilot test.

The results from this grant and the larger study it supplemented offer a very promising option for waste treatment at CAFOs. The materials used for treatment are commonly used at municipal wastewater treatment plants and are safe for environmental application. The mixing protocol is simple and can be done in tankers on-site at CAFOs. Dewatering the waste allows for transportation of a product that is more nutrient dense and most cost effective. The product can therefore be moved farther from the CAFO decreasing the likelihood of over application of nutrients.

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