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CRADA Final Report: CRADA Number NFE-19-07851 with Electro-Active Technologies Inc.



Alex Lewis
Miguel Rodriguez
Costas Tsouris

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Innovation Crossroads

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Alex Lewis
Miguel Rodriguez
Costas Tsouris

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Prepared by
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831-6283
managed by
UT-BATTELLE, LLC
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1. Abstract

Cooperative Research and Development Agreement (CRADA) NFE-19-07851 between Oak Ridge National Laboratory (ORNL) and Electro-Active Technologies Inc. focused on developing a modular stack system to produce renewable hydrogen from food waste and renewable electricity. Developing technologies that can produce renewable, affordable hydrogen is key to enabling wider adoption of fuel cell technologies. Electro-Active has developed a microbial electrolysis process that leverages microbes growing on an anode to convert waste into electrons and protons, which are recombined and reacted with the help of an additional applied voltage to generate pure hydrogen at a cathode. The work done under the CRADA focused on developing a deeper understanding of the microbial community biocatalyst and how feedstock and process conditions affect structure and function in a prototype system to increase and sustain performance. Analysis indicates that developing a robust microbial community for conversion of food waste feedstocks at high rates within a microbial electrolysis system is possible, as well as maintaining this optimal microbial biocatalyst in larger systems. However, further work is needed to maintain performance in the system as it is scaled up to enable commercial deployment of the technology.

2. Statement of Objectives

Following are the three objectives of the CRADA and their relevant tasks:

1. Determine differences in community structure and metabolism arising from use of food waste

Task one involves determining the differences in community structure and metabolism that arise in shifting from biomass waste feedstock (switchgrass pyrolysate) to food waste (kitchen scraps from The Tomato Head restaurant).

- 1.1 Characterization of microbial community omics data from biomass converting reactor
- 1.2 Characterization of microbial community omics data from food waste converting reactor
- 1.3 Comparative analysis of microbial community dynamics and performance

2. Characterize impact of process and reactor design changes on microbial community performance with food waste

The results from Task 1 will guide an effort to maintain highly functional and stable microbial communities for conversion of food waste into hydrogen via microbial electrolysis.

- 2.1 Correlate metabolic, expression, and electrochemical data to community structure
- 2.2 Develop process and design changes to stabilize performance
- 2.3 Analyze impact of implemented changes on microbial community and performance

3. Investigate growth of optimized microbial community in larger systems

After establishing an optimized protocol, during the third task we will investigate growth of the new microbial community in larger systems and the role of quorum sensing in establishing the biofilm.

- 3.1 Identify active quorum sensing genes and target molecules
- 3.2 Compare growth, structure, and performance with different formulations
- 3.3 Commence pilots to generate data in the field

3. Benefits to the Funding DOE Office's Mission

Developing new pathways for renewable, zero-emission fuels that are cost competitive with fossil fuel alternatives is challenging and requires technological innovation and collaboration. The microbial electrolysis technology being pursued is a cross-cutting technology combining biology and electrochemistry, as well as unique reactor stack design. Working with teams in the Biological Sciences Division helped leverage analytical tools to understand and improve the process. The cost of leading analytical equipment can be prohibitive for startups. Access to this equipment and help from expert staff were invaluable to Electro-Active, especially in characterizing the chemical profile of food waste feedstocks as well as the microbial community structure and metabolic activity.

4. Technical Discussion of Work Performed by All Parties

Task 1 involved comparing the microbial community biocatalyst within the microbial electrolysis cells adapted to food waste compared to previous work done by the co-founders using a biomass waste stream in a similar system. Several differences were observed in community structure for the two feedstocks. This part of the work included different proportions of fermenting microbes such as *Bacteroidia*, which were present at higher proportions in the food waste reactors. Meanwhile, *Clostridia* had a lower observed proportion in the food waste reactors compared to the biomass reactors, as was the overall presence of carbohydrate active enzymes in the community, which is likely due to the presence of simpler compounds (Figure 1). This in large part can be attributed to the significant differences in chemical composition of each feedstock.

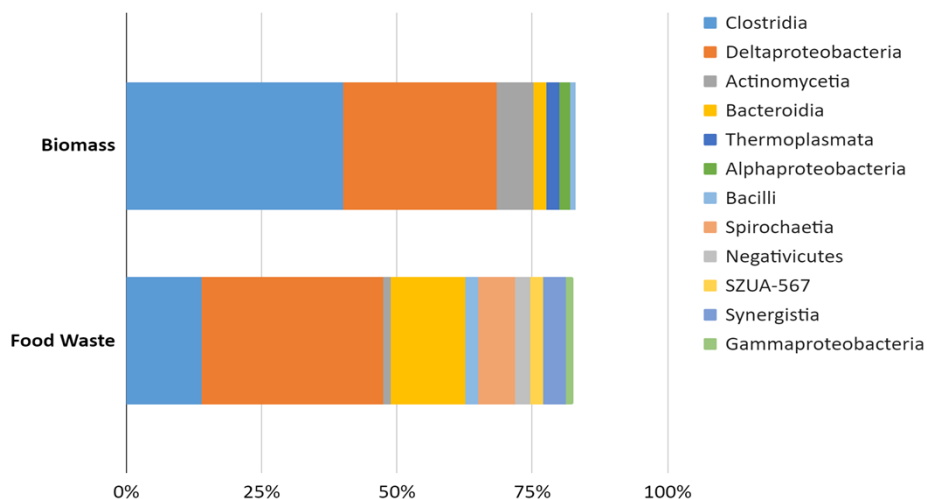


Figure 1: Comparison of microbial communities with biomass vs food waste.

High performance liquid chromatography was carried out at ORNL to investigate individual compounds present in the food waste. The food waste stream was found to contain much simpler sugar-derived compounds and acids, such as glucose, xylose, and tagatose among others compared to large anhydrosugars, furans, and phenolic compounds that were created during the biomass deconstruction. Lastly, a difference was also observed related to electron-generating bacteria, with food waste reactors containing a 2nd, more prevalent species that was not observed to appreciable levels in the biomass fed reactors.

Looking at electrochemical performance, higher microbial conversion of organic matter, as well as overall higher electron generation rates, was observed with food waste compared to biomass waste. Again, this can be attributed to the lower complexity and more easily convertible compounds present in food waste compared to biomass waste. This result provides evidence of a stronger commercial pathway through food waste. Table 1 shows a comparison of performance parameters with biomass vs food waste.

Table 1: Performance comparison of MEC with biomass waste vs food waste

Feedstock	COD removal	Coulombic efficiency	Current density (A/m²)	H₂ productivity (L/L-d)
Biomass	37.6%	81.4%	9.1	7.6
Food waste	87.3%	70.0%	11.9	26.5

With characterizations in Task 1 providing a baseline for performance and community structure with food waste, changes in the microbial community structure due to process conditions were investigated in Task 2. Firstly, control of substrate delivery was investigated, including supplementing the food waste with pure substrates during initial growth to control and enhance the microbial community biofilm development. Figure 2 highlights the difference in community structure at 10% food waste and 100% food waste

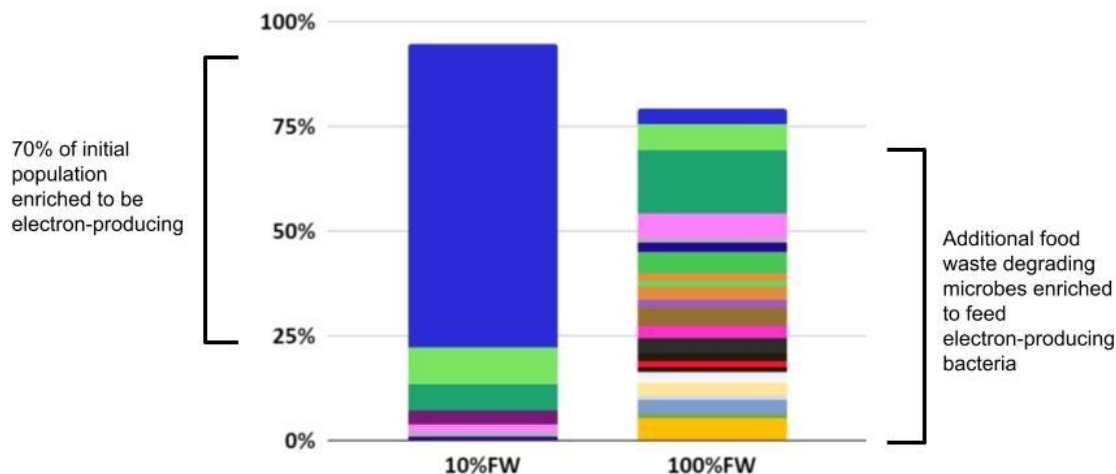


Figure 2: Comparison of microbial community with 10% food waste vs 100% food waste.

Supplementing the main waste stream with pure compounds can help build up the electron generating community first, as this group needs to be in direct contact with the electrode surface. Different types of fermenting microbes are needed to break down the diversity of compounds present in food waste, but don't need to be in direct contact with the electrode, so this group can be enriched after the electron-generating bacteria, as the latter fraction feeds off the products produced by other members of the community.

Performance was analyzed to see if target metrics observed at 10% food waste could be maintained in moving to 100% food waste (Table 2). While coulombic efficiency, current density, and H₂ productivity did dip slightly, they stayed above target thresholds and COD removal was improved with more controlled organic loading.

Table 2: Performance comparison of MEC with 10% food waste vs 100% food waste

Feedstock	COD removal	Coulombic efficiency	Current density (A/m²)	H₂ productivity (L/L-d)
10% FW	60.5%	88.1%	13.6	30.3
100% FW	87.3%	70.0%	11.9	26.5

Task 3 investigated whether the microbial community structure could be maintained as the system is scaled up to develop pilot, and eventually commercial systems. It is critical for commercial applications to be able to sustain performance at larger scale, which is substantially affected by the ability to develop similar microbial communities that have been demonstrated and validated to be capable of reaching desired performance metrics. Duplicate single cell reactors of 80 ml and 360 ml were analyzed for performance and microbial community structure. Data showed that similar community structures were obtained in each of the systems (Figure 3).

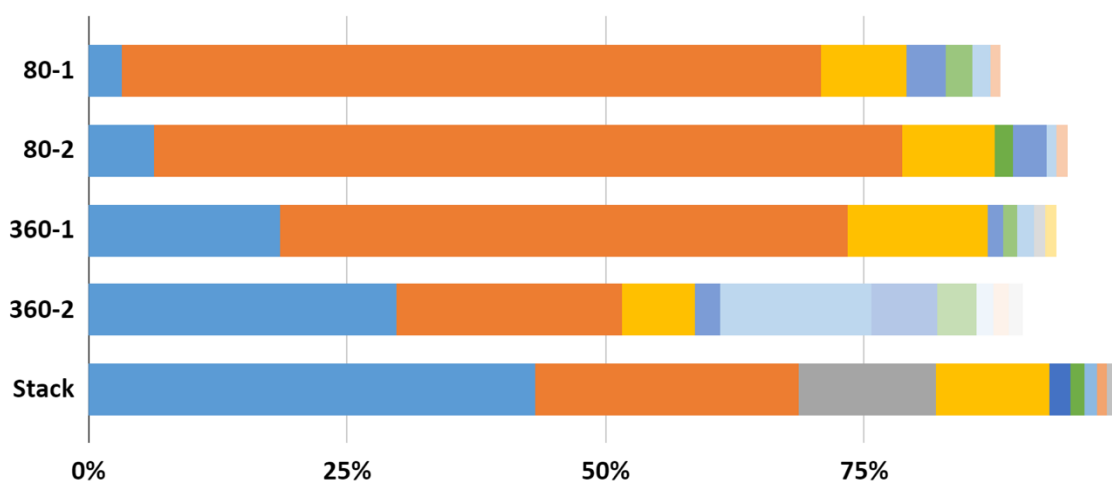


Figure 3: Microbial community comparison between 80- and 360-ml reactors.

However, one of the 360 mL duplicates did deviate from the proportions seen in the other 3 reactors, but overall, the same microbial groups were present. This result validated that similar microbial communities could be developed in larger scale systems. Due to additional time required for previous tasks, investigation of quorum sensing genes and molecules, and development of formulations for testing could not be carried out. Looking at performance comparison, while the microbial community structure was similar, performance was not (Table 3). Efficiency values for COD removal and CE were similar, indicating that the microbial community could efficiently take up COD and produce electrons. However, productivity rates were low, indicating there were other limitations in the system impacting the rates, which need further investigation.

Table 3: Performance comparison of individual MEC reactors at 80 ml and 360 ml scales.

Size	COD removal	Coulombic efficiency	Current density (A/m ²)	H ₂ productivity (L/L-d)
80ml	89.4%	92.1%	22.2	30.9
360ml	71.2%	84.4%	6.1	9.5
21.3L stack	35.9%	102.7%	2.6	4.5

In addition to scaling up the size of individual reactor cells, stacking cells together to make larger systems is needed for commercialization. A stack design, developed previously by Electro-Active Technologies, was analyzed for performance metrics and microbial community composition compared to previous single cell reactors. For the microbial community, Figure 3 shows that the stack system in general contained a very similar microbial community compared to the single cells, specifically with the electron-generating bacteria, further demonstrating the microbial community composition can be preserved in scaled systems. Overall, performance was found to suffer in larger stack systems, with low COD removal, and low current and hydrogen output compared to individual cells. Additional research is needed to uncover the main limitations in larger scale systems for increasing productivity rates.

5. Subject Inventions (As defined in the CRADA)

None.

6. Commercialization Possibilities

Organic waste is widespread and a major economical as well as environmental challenge for a growing global population. It is estimated there will be 10 billion people by 2050, while 70% of that population will live in urban areas, making waste management a significant challenge. Additionally, with the growing threat of climate change, affordable zero-emission fuels are needed to transition our economies away from fossil fuels. The microbial electrolysis system under development can help address both areas, providing a means to convert waste into a needed zero-emission fuel in hydrogen. The results obtained indicate that obtaining a microbial community

capable of high-rate organic waste conversion into electrons for food waste is possible, as well as the subsequent production of hydrogen at commercially relevant rates. However, scaling up the system still requires additional research, and further process optimization and designs need to be developed to retain the performance observed in smaller systems. A pilot study is underway in South Korea, where data will be gathered on the performance of a larger stack system to further inform and optimize the scale up process to develop a commercial system.

7. Plans for Future Collaboration

Further research into the microbial community biocatalyst could yield important information on how to stabilize the activity of this community in larger systems. Furthermore, collaboration on reactor design and assembly, including integration of advanced manufacturing techniques could yield more advanced electrodes, better and more reproducible results, and a scalable design. Grant opportunities will be investigated for opportunities to fund this type of collaborative work with groups within ORNL.

8. Conclusions

Work carried out under this CRADA yielded a deeper understanding of the key microbial players for conversion of food waste feedstocks into electrons. Additionally, insights were gained into the stability of the microbial community in larger systems and the work also allowed for analysis of the impact of different process conditions, including feedstock supplementation to optimize the development of the microbial biofilm. Overall, while similar microbial communities were achieved in larger systems, the performance observed was not similar for the larger systems and did not meet targets, indicating additional work on reactor design and process conditions in the larger systems is needed to improve performance at larger scales.