Arvia Organics Destruction: An Innovative Treatment Approach for Dioxins, Furans and Other Problematic (Orphan) Organic Wastes – 15380

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ABSTRACT

The safe and efficient disposal of radionuclide-contaminated organic wastes is a key component in the lifecycle of a nuclear power plant. Technologies are available for the processing of some of these wastes although there are a number of waste streams for which the disposition path is not clear or does not exist at all. For example, waste streams such as dioxin, furan and PCB-laden oils pose an issue for the US Dept. of Energy complex. These wastes present a challenge as they cannot be readily treated using existing treatment technologies because of legislative and material constraints such as gaseous phase radioactivity discharge limits, waste elemental content, by-product generation and waste resistance to thermal decomposition.

Alternate methods of organics destruction can circumvent some of these issues by using different mechanisms of organics oxidation. One such innovative method, known as the Arvia organics destruction process, combines adsorption with electrochemical combustion and offers such an alternative. This technology, based on a patented material, Nyex™ that is able to act as both an adsorbent and as an electrode, allows the electrochemical combustion of adsorbed organics to occur on the material's surface. The result is the destruction of organics whilst the bulk of inorganic and radionuclide components remain in the aqueous phase. These components can subsequently be treated using existing waste processing technologies.

This paper outlines recent work conducted on demonstrating the Arvia process against the destruction of dioxin, furan and PCB-laden wastes that demonstrates that the technology successfully removes these target species to below discharge standards in the aqueous phase. Based on these successful trials a test unit has been designed to demonstrate the process on actual radioactive wastes at the DOE Oak Ridge facility. The successful treatment of F027-coded waste has also been demonstrated and will be reported.

The above work showcases the development, scaling and application of an innovative technology, first presented to the industry at the Waste Management Symposia in 2012, as another option for the treatment of challenging wastes with expensive, complex or no current disposal routes. The Arvia process adds to the portfolio of available treatment technologies and will help facilitate the decommissioning and waste disposal responsibilities of the nuclear industry.

INTRODUCTION

Innovative Technology for Problem Liquid Organic Waste Destruction

Operation and decommissioning of nuclear facilities results in the generation of a range of radioactive liquid organic wastes including oils, chelating agents, solvents, corrosion inhibitors and cleaning agents. Many of these wastes are difficult to treat using existing technologies (e.g., incineration) as a result of their radioactive content and/or the presence of certain chemical components that preclude these processes from being successfully or economically applied. New, innovative technologies are required for the treatment of these ‘orphan’ or ‘problematic’ wastes.

Arvia has developed a process of adsorption coupled with electrochemical oxidation that has enabled treatment of this category of organic wastes. This process is based on a novel, highly conducting, adsorbent material called Nyex. Treatment is achieved by dissolving or emulsifying the organic waste in water, adsorption of the organic component onto the adsorbent (Nyex) and the regeneration of the adsorbent for reuse by electrochemically oxidizing the adsorbed organic.

The benefits of oxidation of organics as a destructive technology have long been recognised and a range of techniques have been investigated. These include chemical [1], photocatalytic [2], electrochemical [3], supercritical water [4], biological [5] and plasma. None of these alternative oxidation technologies have yet proven technically or economically viable at scale.

Arvia’s process has been demonstrated both on simulant and on real difficult-to-treat liquid organic wastes. A demonstration plant has treated 10 L of LLW and ILW oil contaminated with alpha radioactivity at the Magnox Ltd nuclear decommissioning site, at Trawsfynydd, in the UK [6]. This joint Arvia/Magnox project [7] showed that the oil could be successfully destroyed using an average regeneration energy of 42.5 kWh/l.

The potential of the Arvia process to treat problem waste streams in the United States is being investigated by a consortium of NuVision Engineering, Arvia Technology and Perma-Fix Environmental Services Inc. Of specific interest are dioxin, furan and PCB-laden organics which exist on the DOE estate. These F027-coded liquid organic wastes do not currently have a disposal pathway as regulations on their F027 content and their radioactive content preclude existing technologies from being used in their disposal. This paper addresses the work taking place on developing a treatment path for these, and other radioactivity-containing orphan waste streams, using the Arvia process.

Introduction to Arvia's Process

Arvia's core technology is able to remove organics from solution and electrochemically oxidise them on the surface of its novel adsorbent/ electrode material, Nyex [8]. Arvia has two configurations of its technology: a catholyte system which is based around traditional electrochemical cell technology and a reverse current system in which the polarity of the cell is switched intermittently and the two sides of the electrochemical cell play the role of both the anode and the cathode (Figure 1). This work focuses on the use of a reversing current configuration of the Arvia technology outlined in Figure 1 for the treatment of a fixed inventory of wastes in a batch treatment operation.

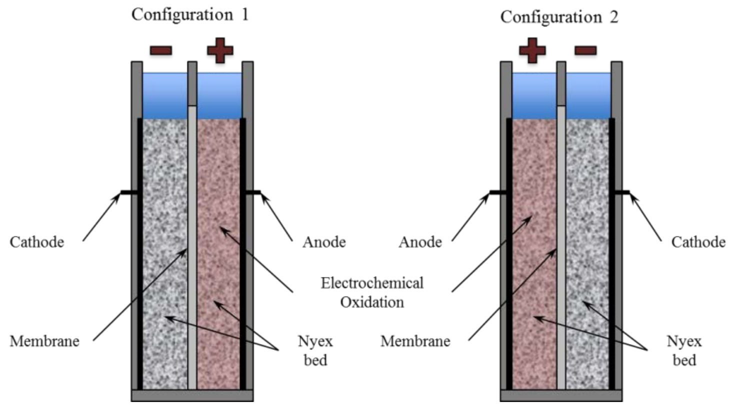


Figure 1: Reverse current version of Arvia's organics destruction cell technology showing the two configurations of operation.

Typical operation for a batch system includes the following concurrent treatment processes:

1. *Adsorption* **–** Achieved by mixing the Nyex and effluent through the injection of effluent into the reactor through a Nyex bed. The effluent can be fed from the top or bottom of the bed depending on system requirements. Effective mass transfer and the non-porous nature of the Nyex results in quick adsorption from the effluent.

2. *Electrochemical Destruction* **–** A direct electric current is passed through the bed while the effluent is passing through the reactor. This current destroys the organic pollutant through anodic oxidation of the organic matter to water, carbon dioxide and small amounts of hydrogen and carbon monoxide. This serves to regenerate the adsorbent, now free to remove further organic from the system.

These processes are shown in the diagram in Figure 2 which is the basic model for the different configurations of cells demonstrated in this work. In this case a bottom feed configuration was used as opposed to the top feed shown in the figure.

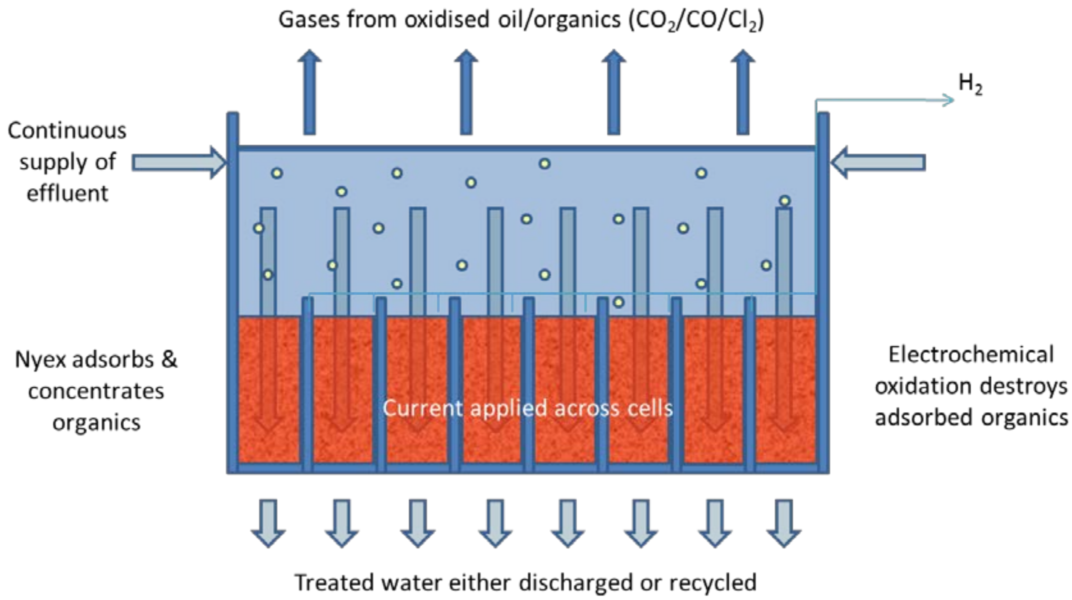


Figure 2: Arvia technology's continuous treatment process shown here in top-feed configuration. Effluent can also be injected from the base of the cell.

When applying Arvia's technology to a waste inventory, different system design and operation parameters have to be taken into consideration. The readiness with which a specific organic is adsorbed affects the energy requirement of completely oxidising the organic on the Nyex surface [9]. This has an impact on the rate of organics destruction per treatment cell and the overall energy cost of treatment per unit organic. This variation in performance has led to Arvia using a feasibility study followed by a pilot plant approach when considering new applications.

To determine the size of a plant appropriate for a specific treatment application the rate at which organics are destroyed needs to be balanced by the number of cells required to deliver this destruction. A larger plant has implications both in terms of capital cost and secondary waste production from the process. The balance between these two parameters – in other terms, the balance between rate of treatment and volume reduction achieved – drive differences in plant sizes given waste characteristics and end user requirements.

Nyex

Two different grades of Nyex have been used in this study; Nyex 1108 and Nyex 2104. Although similar in properties (surface area order 1 m^2 per gram, zero porosity, density order 2.5 g per cm^3), the morphology of the Nyex grades are different. Nyex 1108 is a flake and Nyex 2104 is a granule. Although similar in terms of general adsorption properties (see Figure 3) the granule morphology affords Arvia a better option for higher throughput systems from a pressure drop perspective where the flake Nyex morphology is believed to offer more selective removal for specific applications [9]. Arvia can select the Nyex grade best suited to a specific application and customer requirement.

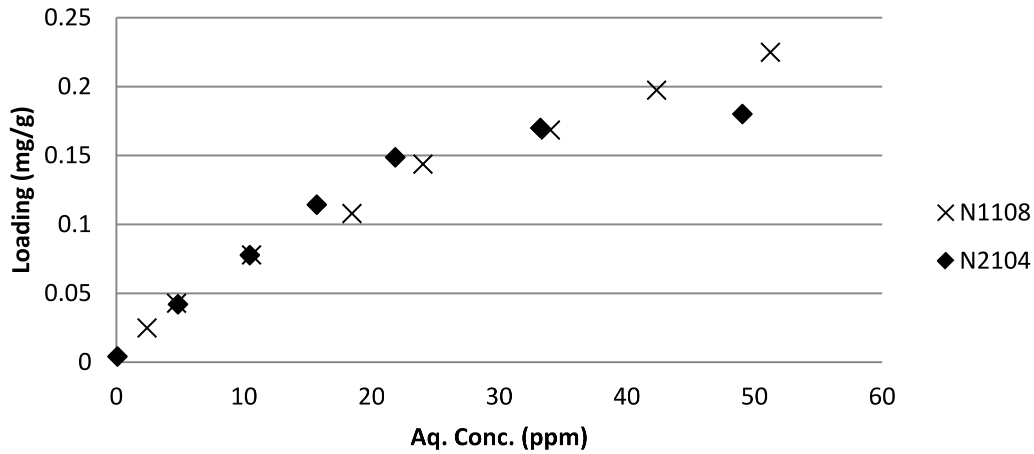


Figure 3: Adsorption isotherm of phloroglucinol for both grades of Nyex; Nyex 1108 (flake) and Nyex 2104 (granule)

EXPERIMENTAL

To determine technology efficacy for the treatment of dioxin, furan and PCB laden wastes a number of experiments have been conducted. The central studies conducted key to this evaluation are outlined here and comprise the demonstration of effective adsorption of the target components, lab scale treatment of simulant F027-coded wastes and the performance of the scaled pilot unit designed and built for waste treatment in the USA.

Adsorption Studies

Adsorption studies were conducted to demonstrate the removal of dioxin, furan and PCB species from the aqueous phase by the adsorbent, Nyex, using both grades of Arvia's Nyex material. The dioxin, furan and PCB assays tested were provided and certified by a third party laboratory, Marchwood Scientific Services. The dioxin, furan and PCB content was specified by the supplier as 7, 10 and 12 μg/l respectively in total, 1 μg/l per congener. Two different assays with different supporting organic matrices were provided; an assay containing 1-nonanol at 1000 ppm and an assay containing 1000 ppm 1,3 butanediol. The purpose of the two assays was to demonstrate any differences in adsorptive uptake of the target species in different organic classes, namely hydrophobic and hydrophilic supporting organic matrices. Experiments were conducted in laboratory glassware and were repeated. Analysis was conducted by the third party labs that provided the assays, Marchwood Scientific Services.

Simulant Wastes in Arvia's ODC1B20 Lab Scale Cell

The ODC1B20 cell is Arvia's standard lab scale cell used for treatability trials and basic testing. It has an electrode area of 400 cm^2 and holds 1 kg of Nyex of either grade. The system is typically used to treat 3 L batches of effluent to determine operational performance. It operates the reverse current version of the Arvia process in which the Nyex adsorbent behaves as an adsorbent, an electrode material in the anode and as a solid electrolyte in the cathode. A schematic of the reactor portion of the ODC1B20 cell in forward and reverse current configuration is shown in Figure 1. Standard experimental conditions are operation at a current density of 10 mA/cm^2 with each configuration applied for 30 minutes. Organics analysis was conducted by gas chromatography with a flame ionisation detector (GC-FID) [9], total organic carbon (TOC) [10] and chemical oxygen demand (COD) [11]; analysis of dioxin, furan and PCB congeners was conducted by a third party laboratory, Marchwood Scientific Services.

Model Wastes in Arvia's 4 Cell Demonstration Unit

The ODC4B20 demonstration unit is the unit designed and developed specifically for the destruction of F027-coded wastes from the DOE estate. It has a total of 1600 cm^2 of electrode area and holds up to 8 kg of Nyex of either grade. Two model wastes, representative of the wastes from the DOE estate to be treated as part of this project, were tested in trials lasting 120 hours. Waste 1 is a waste containing TCP and methanol. The unit was periodically spiked with a TCP – methanol mix and treatment was measured by GC-FID, TOC and COD. Dioxin, furan and PCB content was also measured in the aqueous phase. Waste 2 is an organic mix with the F027 dioxin and furan waste code of unspecified organic content. The waste was simulated with a mix of a solvent (1,3 butanediol was used) and oil (1,nonanol was used) spiked with dioxin, furan and PCB congeners. This trial is currently ongoing and will be reported in a future revision of this paper. Again organics analysis was conducted with GC-FID, TOC and COD. Dioxin, furan and PCB content was measured in the aqueous phase.

RESULTS AND DISCUSSION

Adsorption

Adsorption of the target species – dioxins, furans and PCBs – is central to the effective treatment of F027-coded waste streams with the Arvia process. Adsorption of these species has to be effective in the presence of different supporting organic matrices. Adsorption in the Arvia process is in part driven by the hydrophobicity of the organics adsorbed [9]. As a result it is important to demonstrate adsorption of the target species in supporting organic matrix of both hydrophobic and hydrophilic organics. Two model compounds, 1,3 – butanediol ($LogK_{ow}$ -0.29) and 1- nonanol ($LogK_{ow}$ 3.77) were used to model both a hydrophilic and hydrophobic supporting organic matrix so that the adsorption could be demonstrated in both cases.

Adsorption isotherms were produced in the aqueous concentration range of 0-1000 ppm for 1,3 butanediol and 1-nonanol. The isotherm for Nyex 1108 is shown in Figure 4. It is clear from this data that the more hydrophobic 1-nonanol is much more readily adsorbed than the 1,3-butanediol. The effect was consistent for both Nyex grades. This effect is significant and is a difference of over 2 orders of magnitude in adsorption, on a per Nyex mass basis, above aqueous phase concentrations of 100 ppm. The adsorption experiments were repeated with the matrices spiked with dioxin, furan and PCB congeners. The adsorption results of these species by both grades of Nyex are shown in Figures Figure *5* and Figure *6*.

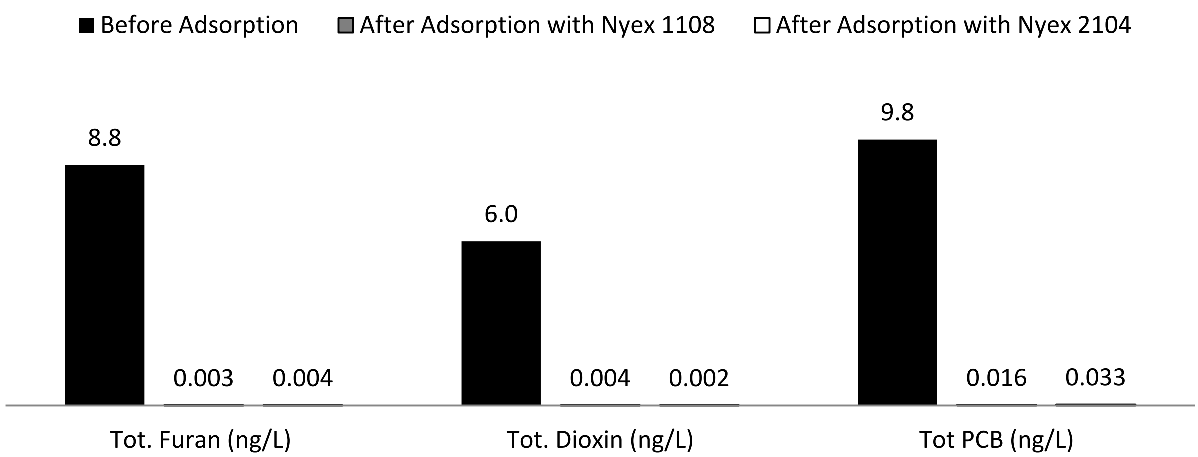


Figure 4: Adsorption isotherms of 1,4 butanediol and 1 nonanol on Nyex N1108 matrix by 2 different grades of Nyex

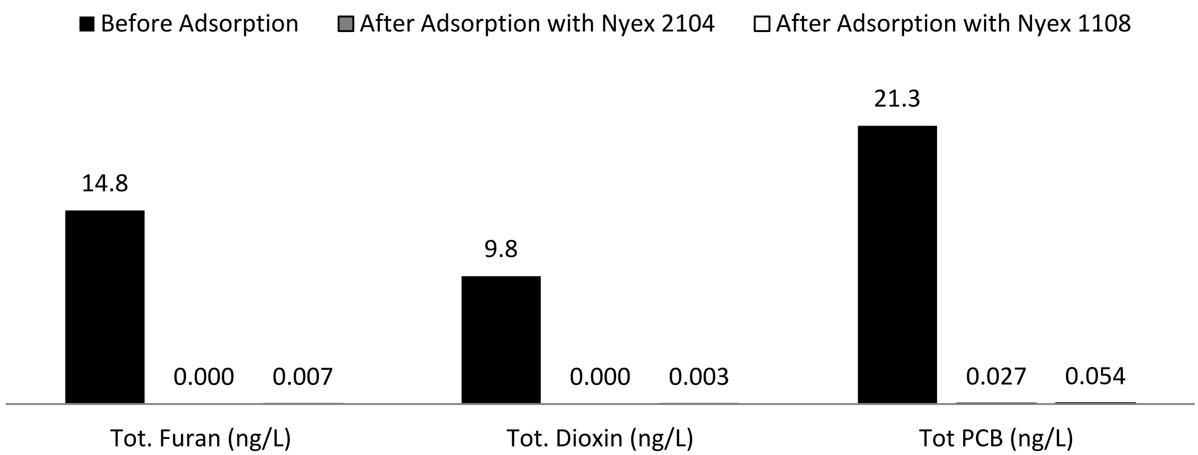


Figure 6: Adsorption of dioxin, furan and PCB congeners in a 1000 ppm 1 nonanol organic matrix by 2 different grades of Nyex

Figures Figure *5* and Figure *6* demonstrate that Nyex is a very effective adsorbent of dioxin, furan and PCB species removing over 99.7% of the species from solution after a single adsorption cycle. Importantly there does not seem to be any significant variation in performance at the 1000 ppm organic concentration level for the hydrophilic (1,3-butanediol) or hydrophobic (1-nonanol) supporting organic in the liquid matrix. This implies that this effective adsorption performance can be expected independent of organic supporting matrices.

The results do suggest a slightly better adsorptive performance of the flake morphology Nyex (N1108) than the granular Nyex (N2104) however the benefit is slight so as to be negligible when error is considered. Importantly this result suggests that the selection of Nyex type does not need to be driven by the adsorption of dioxin, furan or PCB species.

Simulant Wastes in Arvia's ODC1B20 Lab Scale Cell

To determine expected system performance, simulant wastes were treated in Arvia's lab scale demonstration unit, the ODC1B20. The system was tested with both hydrophobic and hydrophilic waste simulants and was used with both grades of Nyex. A selection of the trials conducted with 1,3 butanediol are shown in Figure **7**. As is clear from the figure there is little in

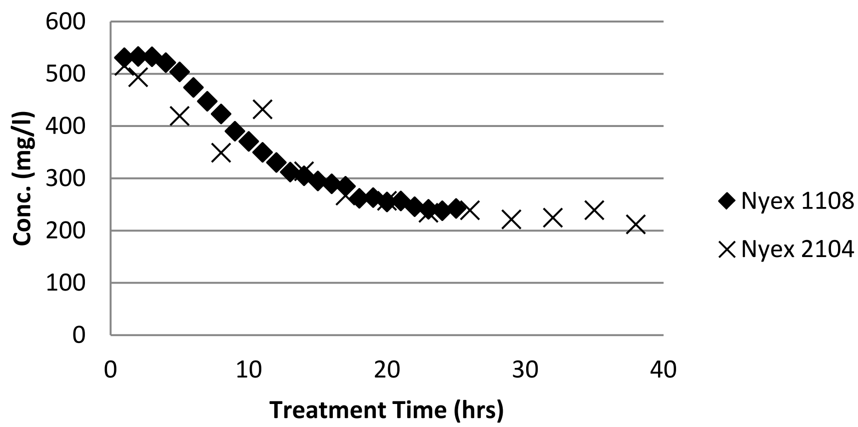


Figure 8: Treatment of 1,3 - butanediol with flake (N1108) and granular (N2104) Nyex in the ODC1B20

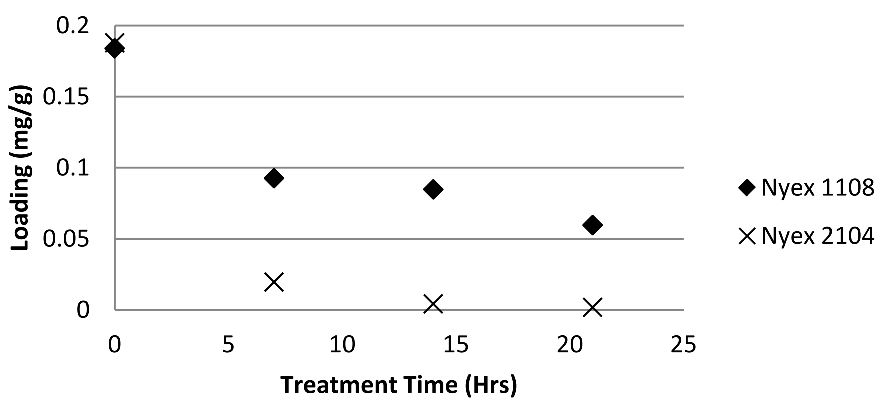


Figure 7: Treatment of 1-nonanol with flake (N1108) and granular (N2104) Nyex in the ODC1B20

terms of difference in treatment performance between the two grades of Nyex for the treatment of solvent type organics. Figure 8 shows a similar comparison for oily type compounds demonstrating slightly superior treatment with granular N2104 Nyex. As can be seen from the results above both grades of Nyex were able to perform the required adsorption and electrochemical destruction processes in Arvia's lab scale unit. Despite there not being significant differences from an organics treatment perspective, there was a difference in terms of operational effectiveness. The granular Nyex was much more suited to the flow through operation required for this application as the pressure drop is more favourable. Experimentally, operation with Nyex 2104 was also found to be more robust. For this reason it was decided that Nyex 2104 would be the material used in the pilot plant deployed in the US to treat the dioxin, furan and PCB laden wastes.

Trials were conducted with dioxin, furan and PCB laden wastes in the ODC1B20. These trials were conducted with Nyex 1108. Trials with Nyex 2104 on dioxin and furan spiked waste were done in the pilot scale unit covered in Section 3.2. The experiments were conducted with both a solvent simulant (1,3-butanediol) and an oil simulant (1-nonanol). Dioxin and furan species were the species of interest in these studies.

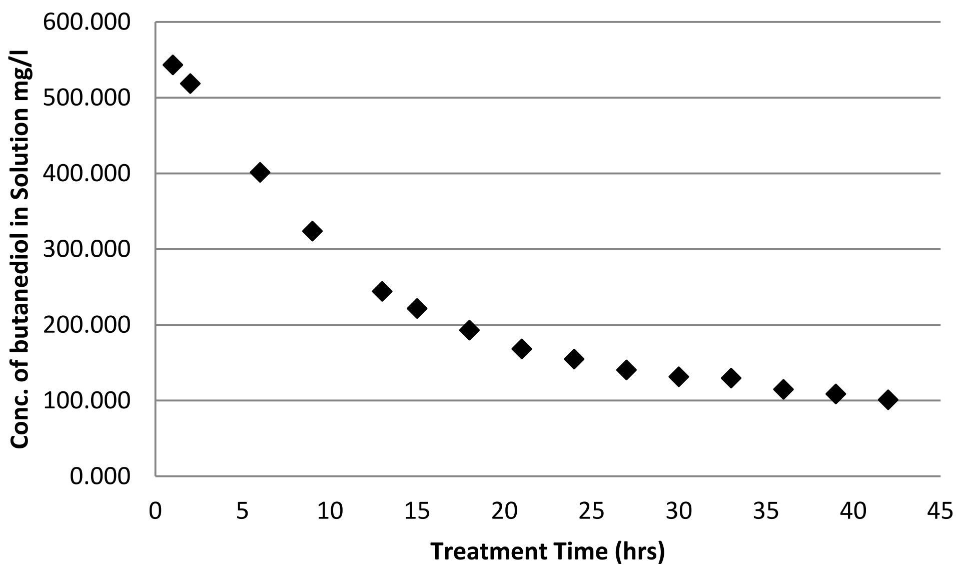
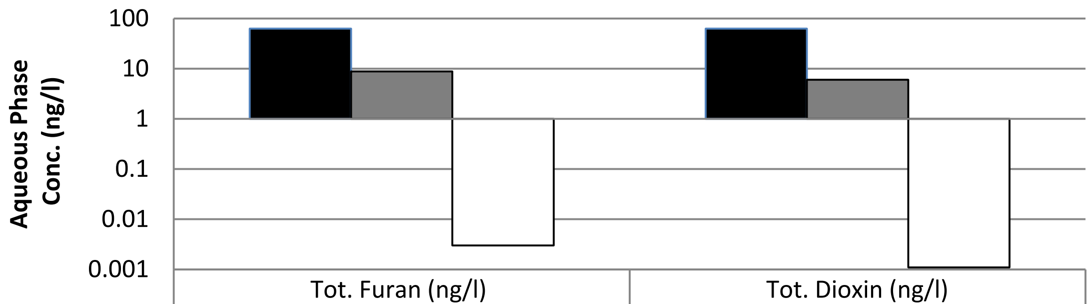


Figure 9: Treatment of 1,3 butanediol spiked with dioxin, furan and PCB in the ODC1B20

Figure 9 shows the treatment of 1,3 butanediol showing the typical trend for solvent treatment with the Arvia process – decreasing treatment efficiency with decreasing effluent organic concentration [12]. Figure 10 shows the dioxin and furan content at the beginning and at the end of the experiment, together with the RCRA[1] discharge limit for these specific species. Despite the starting dioxin and furan content being below the RCRA limit, a result of the lack of availability of these species at higher concentrations, Figure 10 clearly demonstrates a significant reduction in both dioxin and furan species in this organic matrix to levels well below RCRA discharge limits.



	Tot. Furan (ng/l)	Tot. Dioxin (ng/l)
RCRA Limit	63	63
Exp. Start	8.8	6.0
Exp. End	0.0030	0.0011

Figure 10: Dioxin and furan content at the end of effluent treatment – 1,3-butanediol as the supporting organic matrix

Figure 11 and Figure 12 are the analogue of the 1,3 – butanediol experiments with 1-nonanol. In this instance the organic is treated more quickly but the effect on the dioxin and furan species present in the organic matrix is very similar – they are removed to significantly below the RCRA discharge limits. These promising results suggest that, independent of the type of organic in the supporting matrix, the Arvia process is able to remove dioxin and furan species from the aqueous phase to below the discharge limit as required by the RCRA. Solid phase analysis was also conducted to demonstrate treatment of dioxin and furan species and therefore demonstrate both removal and destruction of these species.

To demonstrate dioxin and furan species destruction on Nyex another approach was used as described below.

[1] *Values taken from McCoy's RCRA Reference Book, 2007 McCoy and Associates, Inc. ISBN: 0930469445*

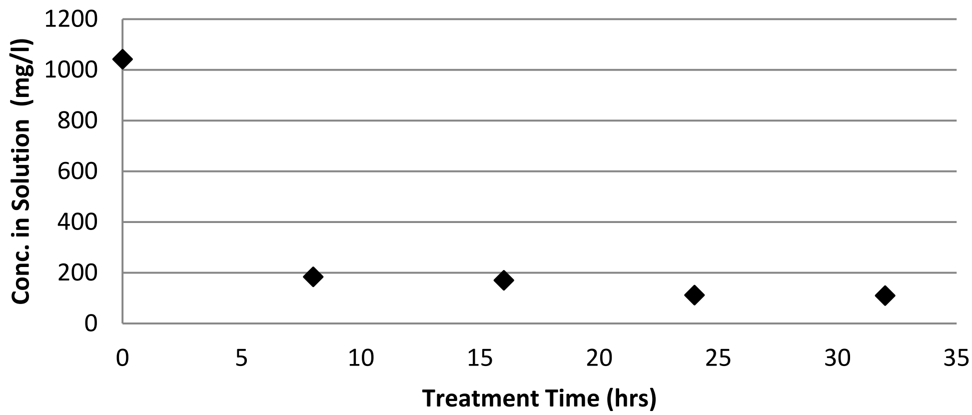
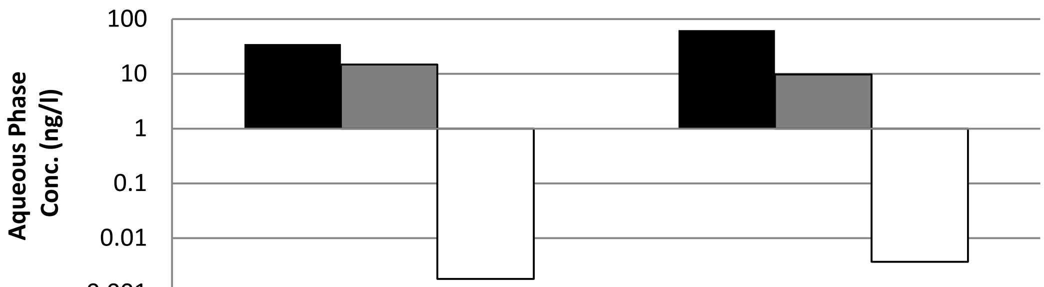


Figure 11: Treatment of 1-nonanol spiked with dioxin, furan and PCB in the ODC1B20



	Tot. Furan (ng/l)	Tot. Dioxin (ng/l)
■ RCRA Limit	35	63
■ Exp. Start	14.8	9.8
□ Exp. End	0.0018	0.0037

Figure 12: Dioxin and furan content at the end of effluent treatment – 1,3-butanediol as the supporting organic matrix

F027- coded wastes in the most part carry this code without dioxin or furan species having been detected in them. The F027 label originates from chlorinated phenol content – a dioxin/furan species precursor which can, under the right conditions, form dioxin and furan species when oxidized. As the Arvia process oxidises the chlorinated phenols on the Nyex surface it is likely that dioxin and furan species would be formed as intermediates during the breakdown process. It was therefore supposed that if a larger amount of dioxin/furan precursor (trichlorophenol) was added to the Nyex and treated, the amount of dioxin/furan species generated would allow Arvia to demonstrate the destruction of these species.

It was found that the destruction of trichlorophenol (TCP) led to a net increase of the furan content measured on the Nyex surface with little measured change in the dioxin content of the Nyex surface. Of this increase in furan on the Nyex surface, the dominant species produced as a breakdown product after 3 hours of electrochemical treatment was 2,3,7,8-trichlorodibenzofuran, shown in Figure 13. It was then possible to trace both this individual species, as well as the net

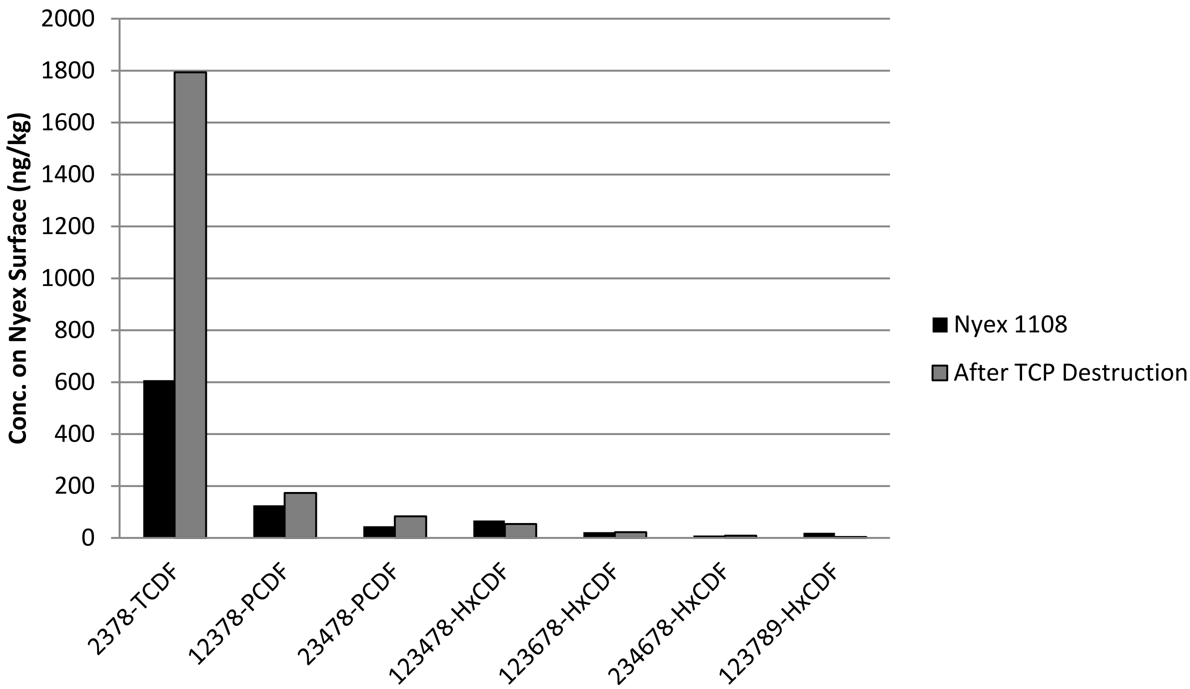


Figure 13: Change in furan content on the surface of Nyex after treatment of TCP in the ODC1B20 for 3 hours

furan concentration, with treatment time. This experiment was conducted with a supporting solvent in solution to mimic treatment under normal conditions. The TCP in solution was shown to disappear within 5 hours of treatment. The results from furan analysis for this experiment are shown in Figure 14 showing native furan content of Nyex 1108 followed by a large increase after 3 hours of treatment of the TCP organic matrix. Importantly both the 2,3,7,8 – TCDF (trichlorodibenzofuran) and the total furan content are shown to decrease with time after this point implying that the Arvia process does indeed destroy these species on the surface of its adsorbent, Nyex. In other words, TCP oxidation on the Nyex surface results in the generation of the intermediary furan, 2,3,7,8 – TCDF, which is subsequently destroyed by the oxidation process. This finding is central to the project as it demonstrates that the technology is capable of treating F027-coded wastes by electrochemical oxidation, not simply effective adsorption.

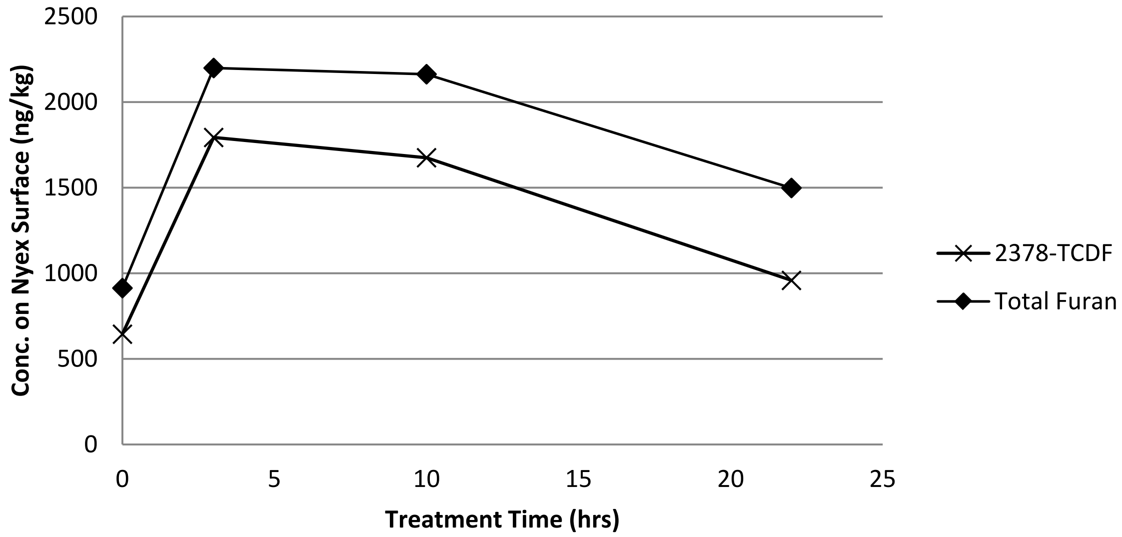


Figure 14: Furan content of Nyex during treatment of a TCP containing simulant waste

Model Wastes in Arvia's 4 Cell Demonstration Unit

The commissioning phase of the pilot ODC4B20 unit allowed some parameter optimisation to take place. Initially the system was periodically spiked with 1,3-butanediol as the simulant waste and the concentration of remaining waste was measured by COD. Each treatment cycle lasted 24 hours. Two different concentrations were used, 1250 ppm COD and 2500 ppm COD. The results are shown in Figure 15. The figure shows that a steady state was reached between the dosing at the 1250 ppm level and the resulting concentration after 24 hours of treatment. When the dose was doubled the results suggest that more organics destruction occurred per 24 hour period, but the steady state achieved appears to have been lost with a net buildup of COD apparent. This would suggest that the system was being dosed with more organic than it could destroy per 24 hour period.

Further, as it appeared that greater organics destruction was achieved at the higher organic loadings the relationship between organics destruction and concentration was also investigated.

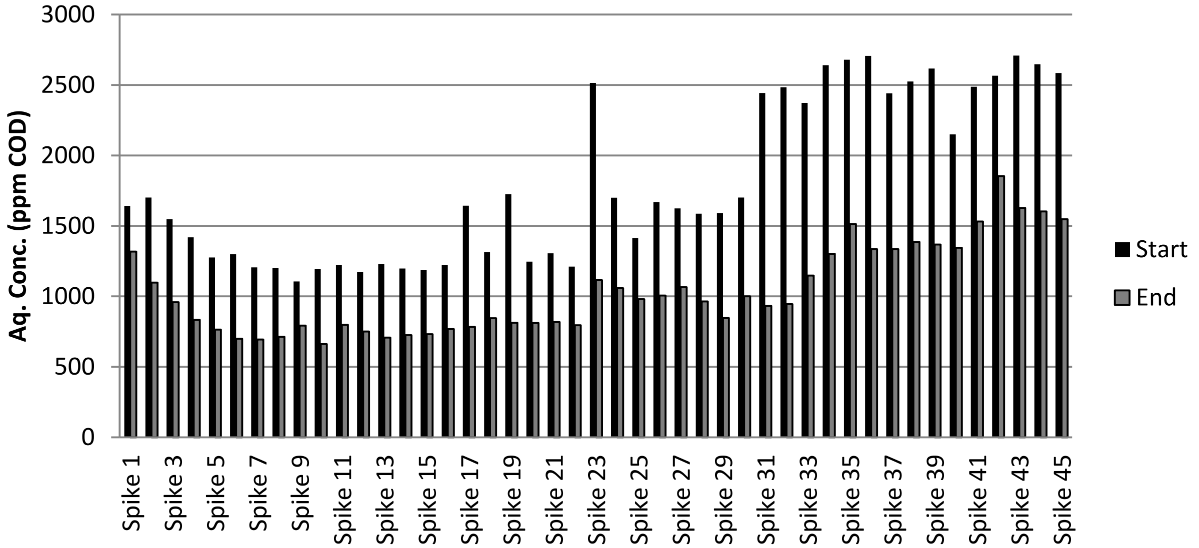


Figure 15: Treatment of 1,3-buitanediol with the ODC4B20 unit at two different levels of dosing

The experiment was repeated starting with a higher organic dose and the results are shown in Figure 16. This experiment confirmed that the addition of an excess of organic per cycle resulted in the net buildup of COD in the system and that the higher doses led to a more favourable organics destruction per cycle than the lower organic doses. This was also tested by following a single organic dose with time; Figure 17.

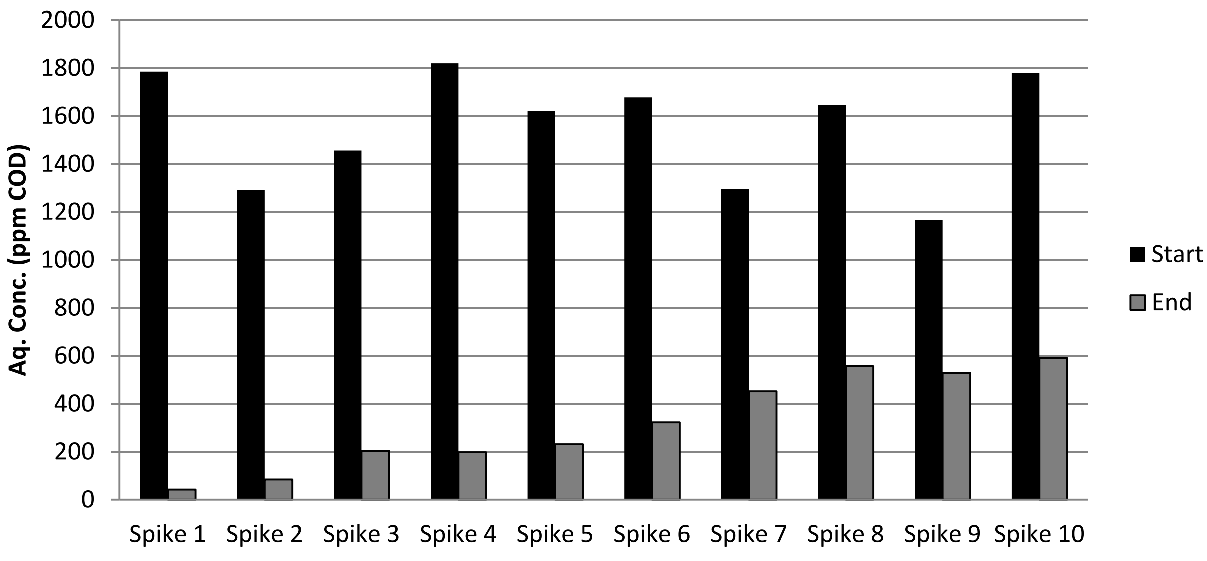


Figure 16: Treatment of 1,3-butanediol - higher organic dose

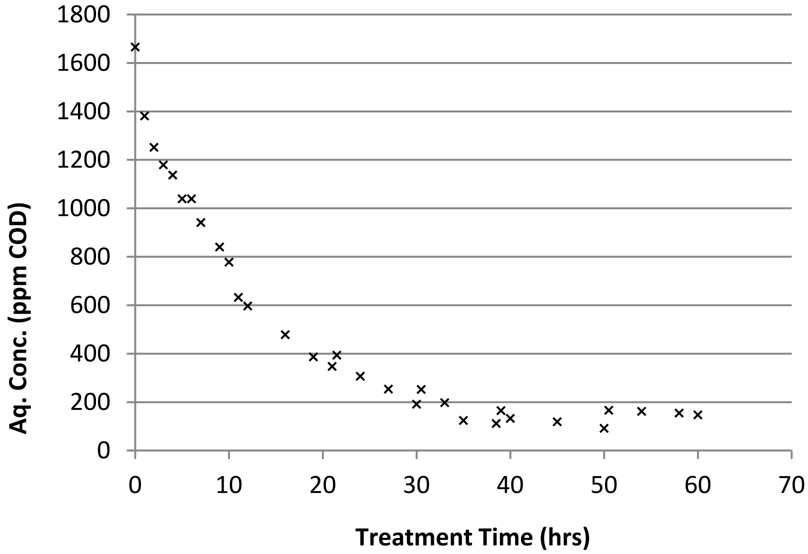


Figure 17: Treatment of a single dose of 1,3-butanediol

Figure 17 demonstrates that the rate of organics destruction is proportional to the concentration in solution. As a result of this and the other observations above, when deciding on what organic dosing philosophy to adopt when using the treatment system it is important to manage dose size to ensure that both optimum treatment rate and minimum COD buildup are achieved.

A trial comprising the treatment of TCP in methanol, a matrix representative of one of the problem wastes on the DOE estate, was conducted in Arvia's pilot ODC4B20 unit. The system was periodically spiked with simulant waste and the concentration of remaining waste was measured by COD. Each treatment cycle lasted 24 hours. The amount spiked was increased throughout the trial to determine if the system had reached its maximum organics destruction capacity. The TCP content of the samples will be measured but this has not yet been completed. Dioxin and furan analysis is also outstanding at this time and both will be reported in the conference presentation. The COD results from the trial are presented in Figure 18. Again, organic dosing was varied to determine the point at which COD began building up in the effluent implying that the maximum organics destruction capacity of the system had been achieved. The results show that the bulk of COD added at the beginning of each cycle is destroyed by the system for all waste doses added. This implies that the amount dosed did not surpass the maximum organics destruction capability of the system for this waste type.

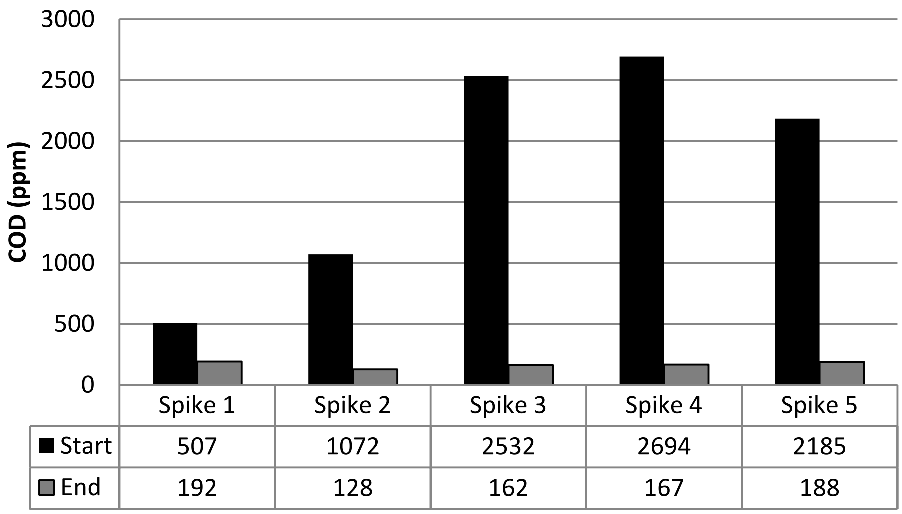


Figure 18: COD of simulant methanol - TCP waste during treatment in the demonstration plant

The above experiment was repeated with a different waste matrix representative of another waste type from the DOE estate. The waste comprised a combination of 1,3-butanediol and 1-nonanol spiked with a total of 6 µg/l of dioxin and furan species. This experiment aimed to follow both the decay of the organic component in the waste over several spikes, and the fate of the dioxin and furan species in this waste matrix. As the previous experiment had shown that dosing waste once every 24 hours did not give an optimum performance, waste was dosed every 12 hours in this experiment. The results are summarised in Figure 19. Again the results demonstrate that the system can perform for this different category of organic waste; this time more effectively as a result of the dosing frequency.

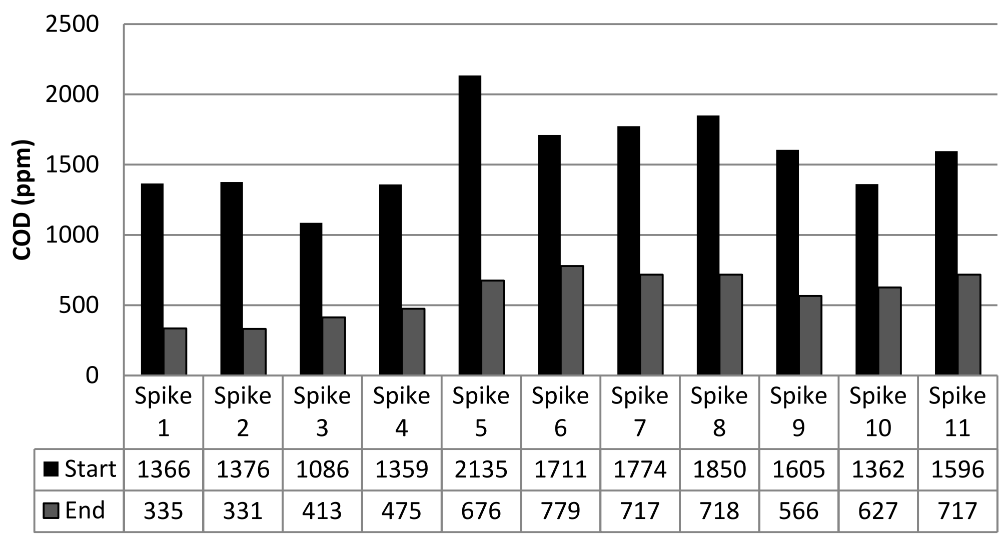


Figure 19: COD of simulant methanol - TCP waste during treatment in the demonstration plant

Comparisons were conducted between the treatment of the two categories of waste. Overall, the average energy consumption per kg of COD was higher for the TCP & methanol waste dosed every 24 hours, 233 kWhrs/kg COD and lower, 130 kWhrs/kg COD, for the 1,3-butanediol & 1-nonanol waste dosed at 12 hour intervals. It is notable that for both wastes much better energy consumptions were observed during treatment than the average energy consumption quoted above, as good as 42 kWhrs/kg COD. These results suggest that further optimization work can be conducted to ensure that the system runs more energy efficiently. The results also correlate with earlier work reported at this conference [12] which demonstrated more effective treatment of hydrophobic compounds than hydrophilic compounds.

CONCLUSIONS AND NEXT STEPS

The presented data demonstrates how Arvia's organics destruction technology has begun to be successfully applied to different organic waste stream types containing F027 coded organic contaminants. This work marks a significant step forward in addressing a waste disposal issue for the US DOE. Key findings were as follows:

- The process has been demonstrated to be effective against both solvent and oily type wastes representative of the orphan F027 coded waste streams identified in the project.
- Dioxin and furan species were shown to be adsorbed and destroyed by the process allowing for the safe disposal of F027 coded wastes.
- Adsorption of these species was effective both in solvent and oily organic matrices and resulted in below RCRA discharge limits.
- Trichlorophenol could be destroyed without the formation of hazardous dioxin or furan species being released into the aqueous phase; any species generated remained adsorbed and were destroyed on the Nyex.

It is clear from the data presented that the principle of safe F027-coded waste destruction has been showcased in the demonstration work carried out as part of this project. The implication is that the F027 coded waste stream currently representing a disposal challenge for the DOE has a candidate technology capable of safe and effective disposal. The technology is to be tested on active wastes in the USA early 2015.

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