

On-site Destruction of Radioactive Oily Wastes Using Adsorption Coupled with Electrochemical Regeneration – 12221

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ABSTRACT

Arvia[®], working with Magnox Ltd, has developed the technology of adsorption coupled with electrochemical regeneration for the degradation of orphan radioactive oil wastes. The process results in the complete destruction of the organic phase where the radioactivity is transferred to liquid and solid secondary wastes that can then be processed using existing authorised on-site waste-treatment facilities.. Following on from successful laboratory and pilot scale trials, a full scale, site based demonstrator unit was commissioned at the Magnox Trawsfynydd decommissioning site to destroy 10 l of LLW and ILW radioactive oils. Over 99% of the emulsified oil was removed and destroyed with the majority of activity (80 – 90%) being transferred to the aqueous phase. Secondary wastes were disposed of via existing routes with the majority being disposed of via the sites active effluent treatment plant. The regeneration energy required to destroy a litre of oil was 42.5 kWh/l oil. This on-site treatment approach eliminates the risks and cost associated with transporting the active waste oils off site for incineration or other treatment.

INTRODUCTION

A significant problem at nuclear reactor sites is the treatment of radioactive contaminated organic wastes. Whilst a number of process routes are available for the treatment of these wastes, those with high activity levels cause significant issues. An example of difficult organic waste are LLW and ILW oils contaminated with alpha radioactivity at a Magnox Ltd nuclear decommissioning site [1]. The current Magnox baseline disposal route for waste oil is incineration, however, this route is not available for significantly contaminated oil and hence these wastes have been identified as orphan wastes or wastes requiring additional treatment (WRAT).

Arvia Technology Ltd and Magnox Ltd have been working together over the past three years to demonstrate that the Arvia[®] Process for water treatment [2-4] can be applied to the destruction of radioactive organic wastes, specifically oils. This process comprises four stages:

Emulsification – the oil is emulsified in water (using an organic emulsifying agent and a high shear mixer) to give a stable emulsion.

Adsorption - is achieved by mixing the Nyex[®] and effluent through fluidising the adsorbent particles, where vigorous mixing and the non-porous nature of the Nyex[®] results in quick adsorption.

Sedimentation - results when the fluidising air is switched off and the dense Nyex® particles settle rapidly under the influence of gravity to form a bed.

Electrochemical Destruction - Two electrodes are placed either side of the bed of particles and a direct electric current is passed through the bed which destroys the pollutant through anodic oxidation of the organic matter to water, carbon dioxide and small amounts of hydrogen, carbon monoxide and chlorine. This serves to regenerate the adsorbent. The regenerated adsorbent is then ready for immediate reuse and the whole cycle is repeated.

The current progress of this initiative to develop a process for the destruction of radioactive organic wastes is reported, specifically describing the large scale demonstration trial results from Trawsfynydd.

EXPERIMENTAL METHOD

Magnox commissioned a 3 year development programme with Arvia to evaluate the potential of the process to destroy these legacy and orphan waste oils. This programme was split into three stages:

Stage 1 – To prepare emulsified solutions containing oils and to prove the destruction of non-active oils in the laboratory (Scale - 1 l) [5]

Stage 2 – To optimise the process at laboratory scale using non-active oils to develop design data and to prove the process at larger scale (Scale - 60 l) [6]

Stage 3 – To construct an on-site (Trawsfynydd nuclear decommissioning site) demonstration plant treating LLW and ILW active oils (Scale - 200 l). The aims of this were to assess the partitioning of the active isotopes, to demonstrate that all secondary wastes could be successfully disposed of and to prove that the active oil could be destroyed [7].

Materials

Throughout the test period the following oils were used

Stage 1 – Emulsification trials used the following fresh oils: Syntilo R (Castrol), Hysol G (Castrol), Perfecto T46 (Castrol), Nucleol 520 (Castrol), Tellus 46 (Shell) and Tellus 32 (Shell). The oil identified for adsorption and regeneration trial was Tellus 46.

Stage 2 – Tellus 46 for laboratory optimisation and pilot scale trials.

Stage 3 – Tellus 46 for on and off site commissioning trials. On-site active oil trials were undertaken using a high alpha LLW (LLWO/11/09) and ILW oil (ILW JN54) wastes.

Additional materials were the Nyex® (Arvia's proprietary adsorbent material), CLAX 200S (an organic emulsifying agent supplied by Diversey Inc), hydrochloric acid, sodium hydroxide and sodium chloride.

Equipment

A number of Arvia adsorption and electrochemical regeneration systems were used in this work. Stage 1 made use of Arvia's mini-sequential batch reactor (mini-SBR Figure 1a), which was also used in the stage 2 optimisation trials. Stage 2 used the large sequential batch reactor

(SBR Figure 1b) to prove pilot scale operation of the unit and Stage 3 used an automated version of the mini-SBR (the M-ORC) to determine an activity balance across the three phases. On-site treatment was achieved using Arvia's "Titan" demonstration unit (Figure 1c). A summary of the unit designs is given in Table 1.

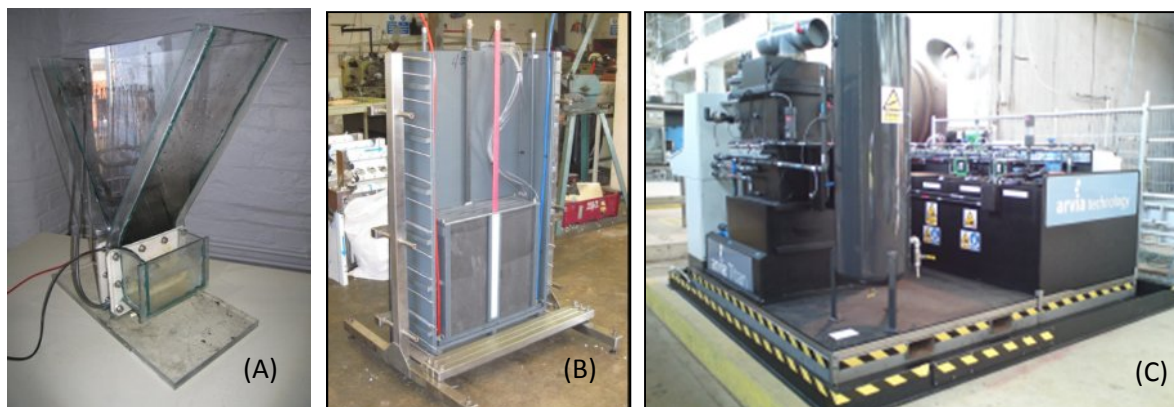


Fig 1. Arvia Test Units (A) Mini-SBR; (B) SBR; (C) Titan

Table I. Arvia Test Unit Parameters

Unit	Electrode		Material of construction		Nyex® Mass (kg)	Volume (l)		Current Density mA/cm ²
	Number	Size (mm)	Anode	Cathode		Oil/water emulsion	Catholyte 0.3% NaCl	
Mini-SBR	1	50 * 100	Carbon	Perf SS (316)	0.1	1	0.1	Upto 20
M-ORC						2.2	1.8	20
SBR	6 ^(a)	500 * 500	Carbon (bi-polar)	Carbon (bi-polar)	25	60	20	20
Titan	18 ^(b)	500 * 500	Carbon (bi-polar)	Carbon (bi-polar)	60	218	180	10

^(a)Cells operated in series

^(b)Cells operated as three parallel banks of 6 cells in series for safety (operating as banks of six cells kept the voltage across the unit low enough that no safety interlocks were required)

Four studies were performed on the M-ORC as outlined in Table II:

Table II . Oil Added During the M-ORC stages

Study Number	Oil Added (ml)	Oil Identity
MO1	5	ILW JN54
MO2	10	LLWO/11/09
MO3	10	ILW JN54
MO4	5	ILW JN54

The undiluted gaseous outlet from the M-ORC was passed through a furnace at 600 °C containing a copper catalyst, to oxidise any tritiated species to tritiated water, and then through

two *Dreschner* bottles containing water to trap the tritiated water. These samples could then be scintillation counted for tritium and thereby the tritium in the off-gas quantified.

Table III. Oil Added During the Titan stages

Study Number	Oil Added (ml)	Oil Identity
T101	1355	LLWO/11/09
T102	1326	LLWO/11/09
T103	1323	LLWO/11/09
T104	1249	JN54
T105	1249	JN54
T106	1252	JN54

Samples were taken of the supernatant effluent, the catholyte and the Nyex[®] and sent to GAU Radioanalytical Laboratories for radiochemical analysis. Organic loading in the liquid phase was measured by COD (Chemical Oxygen Demand) to give an indication of the aqueous oil loading using Hach COD vials and a DR90 Colorimeter.

RESULTS AND DISCUSSION

For clarity this section has been split into two parts. The first part describes the destruction of the oil and the processing parameters. The second concentrates on the fate of the radioactive species.

Oil Destruction - Titan Inactive Oil Studies

Prior to starting on-site active oil destruction, the Titan unit was commissioned both off- and on-site using inactive Tellus 46 oil. Adsorption of the oils results in the partitioning of the oil between the aqueous phase and the Nyex[®]. To demonstrate the removal of oil from the aqueous phase, the Chemical Oxygen Demand (COD) was monitored on a regular basis. The COD of the oil water solution in the treatment tank (assuming no oil was removed) was calculated to be 90,400 mg/l. Off-site and on-site trials operated for 46 and 43 hours respectively, with resultant CODs of the supernatant solution after treatment being 750 and 280 mg/l respectively. This gives oil removal rates in excess of 99%. Earlier research work [2,3] had shown that regeneration of the Nyex[®] could result in the multi-layered adsorption of oil, causing the agglomeration of adsorbent particles and ultimately in the “clumping” and “balling” of particles to the detriment of the process, particularly causing an increase in cell potential. It had been shown [3] that SEM pictures can be used to show the presence of oil on the adsorbent through the formation of droplets when water vapour condenses on the dried adsorbent particles. Hence images after 21 hours (Fig. 2.) operation show both the tendency to clump and the presence of droplets, whereas those after 46 hours operation (Fig. 3.) demonstrate that there is no retention of organic material (oil) on the surface of the regenerated Nyex adsorbent.

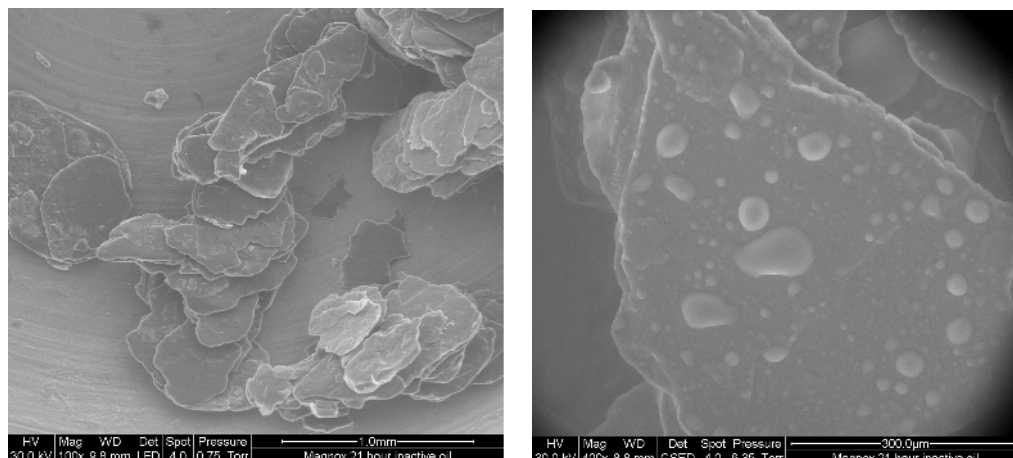


Fig. 2. The picture on the left after 21 hours operation shows that the Nyex® have a tendency to clump together and on the right it shows the formation of droplets with water vapour.

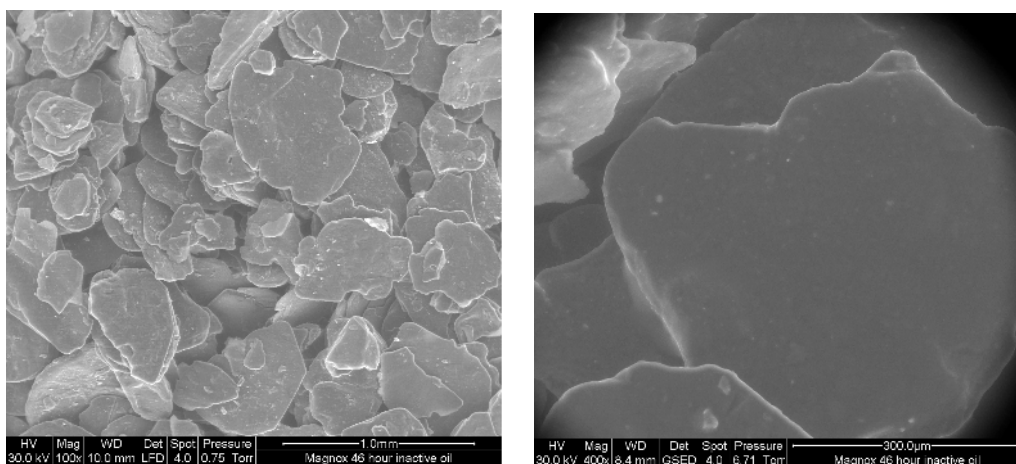


Fig. 3. The picture on the left after 46 hours operation shows that the Nyex® are single flakes and on the right it shows no droplet formation as with water vapour.

Oil Destruction - On-Site Active Trials - For the M-ORC trials it can be calculated that the supernatant COD, assuming that no oil is destroyed, is 41,000 mg/l. All the supernatant effluent samples (after dilution at 50:1) for studies MO3 and MO4 had a COD of 0 mg/l (± 6 with a Limit of Detection 20 mg/l). This demonstrates that the Nyex® is picking up the added oil quickly; this is consistent with the earlier inactive oil trials, which showed oil removal efficiencies in excess of 99%.

Similarly the the supernatant effluent from the Titan plant active trial was tested for COD at all stages, i.e. TI01 – TI06 and the results are shown in Figure 1. Peaks in the graph are due to the addition of aliquots of oil/water emulsion. The predicted COD at the end of the trial assuming no oil was removed from the supernatant emulsion was calculated as 70,000 mg/l. The supernatant COD values (after dilution at 50:1) at the end of the trial were 0 mg/l showing that the oil had been successfully removed from the oil/water emulsion, consistent with the M-ORC trial.

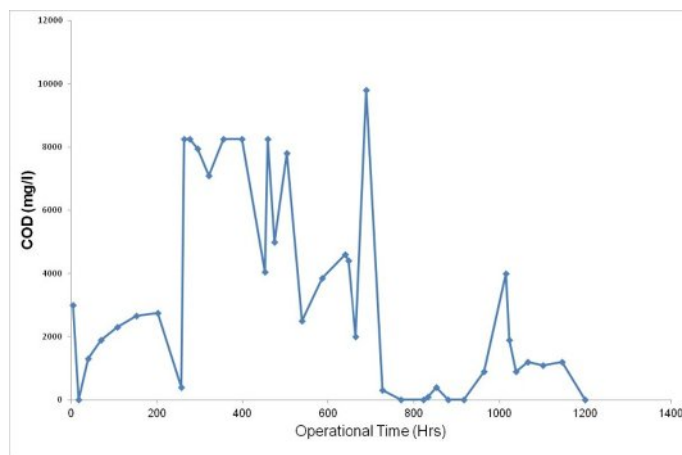


Fig. 4. COD of supernatant during trials T101 – T106

Comparison of different oils

The performance of the Arvia® Process in treating inactive oil and two different active oils using the same treatment unit with the same operating conditions (current density 10 mA/cm² regenerating for 20 hours per litre oil destroyed) demonstrates that the process is robust enough to cope with variations in the incoming feed. Table 1 below shows how key parameters compare between the different oils during on and off-site trials.

Table IV. Comparison of Key Parameters treating different oils

Type of oil	Operational parameters (V/stack)	Regeneration Power (kW)	Energy kWh/l	Oil destruction rate ml/hr
Tellus 46 (neat inactive) Off-site	20.7	1.55	47.9	34.9
Tellus 46 (neat inactive) On-site	22.3	1.73	47.5	32.6
LLWO 11/09	16	1.2	42.5	28.2
ILWO (JN54)	16	1.2	42.5	28.2

Energy Requirements

In the processing of the LLW oil waste (trial phase TI01-03) the Titan was operated at a fixed current of 25A, where the average cell voltage was recorded as 16 V, giving an energy consumption for the 4.5 litres of oil treated as 42.5 kWh/l. The voltage variation across each bank of six cells recorded during the regeneration is shown in Figure 5. For the processing of the ILW oil (TI04 – TI06), the Titan unit was operated in exactly the same manner as that for the LLW oil trial (TI01 – TI03) and again the average voltage per stack of six cells was 16 V. The reduction of voltage over time is due to the increase in liquid conductivity and reduction in liquid pH due to the addition of hydrochloric acid. The increased conductivity reduces the voltage at the cathode and the reduction in pH reduces the voltage across the membrane.

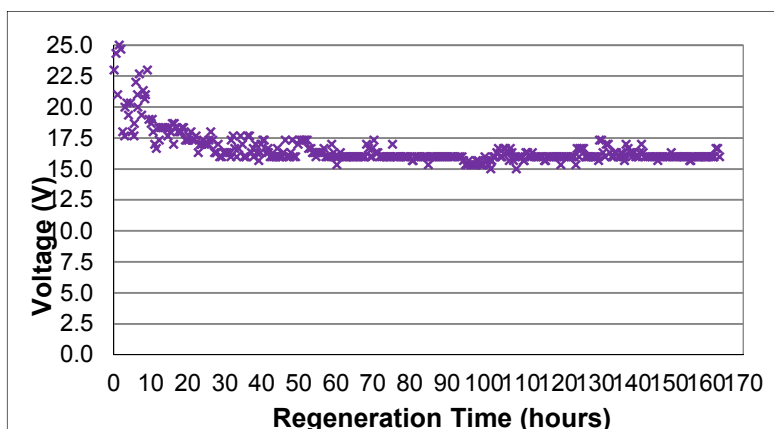


Fig.5: Average voltages per cell stack of 6 cells for TI01 – TI03

Scale up

A key factor in the application of this technology will be scalability of the process. The scale up factor from the mini-SBR to the Titan unit is 900. As can be seen from the data (Table V), destruction of the oil in the various units gives similar results, with the key variable being the current density. Reducing the current density will reduce the energy needed to destroy the oil, but will increase the time or the number of cells required.

Table V. Effect of Scale up on Key parameters

Unit	Electrode size (mm)	No Cells	Current Density (mA/cm ²)	Energy kWh/l	Ave. Cell Voltage (V)	Scale up factor
Mini-SBR	50 * 70	1	20	60	6V	1
M-ORC	50 * 70	1	20	57	3.8	1
SBR	500 * 500	6	20	56.25	4.5V	425
Titan (inactive oil)	500 * 500	18	10	47.7	3.6	1285
Titan (active oil)	500 * 500	18	10	42.5	2.7	1285

No scale-up issues are envisaged as larger capacity plants will use larger numbers of the same size electrodes. For the purposes of this on-site trial, the Titan unit was operated manually. This has provided confidence that unattended continuous operation on a 24/7 basis is possible.

Distribution of Radioactivity Between Arvia Process Secondary Wastes

Baseline analysis of the LLW and ILW oil employed in the trial was undertaken by GAU Radioanalytical Laboratories [8]. Significant heterogeneity was associated with the LLW oil sample, demonstrated by the disconnect between the observed Americium-241 (Am-241)

activity concentration derived from gamma spectrometry of the “As received” sample and from subsequent analysis of a small sub-sample by alpha spectrometry ($\text{Am-241 } (\gamma)/\text{Am-241 } (\alpha) = 1.5$). Consequently it was decided to normalise the test results derived from the analysis of the sub-sample digest to the “As-received” Am-241 activity. The normalised results are reported Table VI.

In contrast to the LLW oil the analytical results demonstrate that the ILW oil is reasonably homogeneous in that there is excellent tie-up in the Am-241 (γ) / Am-241 (α) ratio (0.9). Furthermore, there is very good comparison between the activity concentrations reported for a number of the key species and those reported from previous characterisation exercises. The exception is caesium-137 (Cs-137) and as will be seen this has caused some problems in the assessment of this nuclides distribution between the various secondary process wastes arising from the Arvia process.

Table VI. Baseline Activities for the active oils

Radionuclide	LLW Oil (LLWO-M77) Specific Activity (Bq/g)	ILW Oil (JN54) Specific Activity (Bq/g)
Gamma Emitters (by Gamma Spectrometry):		
Cobalt-60	0.96±0.05	2.1±0.1
Caesium-137	140±7	2.9±0.2
Europium-154	4.7±0.3	87±5
Europium-155	0.88±0.16	24±3
Americium-241	43±3	1130±70
Beta Emitters (by Beta Spectrometry):		
Tritium	8±1	8±1
Carbon-14	0.2±0.1	1.1±0.2
Chlorine-36	<0.2	<0.2
Iron-55	2.2±0.3 ^a	3.3±0.6
Nickel-63	1.4±0.2 ^a	17±2
Strontium-90	71±8 ^a	1200±120
Alpha Emitters (by Alpha Spectrometry):		
Plutonium – 239/240	21±2 ^a	99±9
Plutonium – 238	9±1 ^a	300±20
Americium-241	43±3 ^a	1030±50

^a These values are normalised to the bulk Am-241 measurement derived by gamma spectrometry to accommodate sample heterogeneity

Throughout both the LLW and ILW oil processing trials, samples of the secondary waste phases were taken for full analysis against the baseline oil fingerprints determined by GAU. The results of the analysis of the secondary wastes arising from the processing of the ILW oil are the more complete with correspondingly low uncertainties because of the higher level of radioactivity and the greater homogeneity seen for the ILW waste oil. Hence this data was taken as the definitive data-set for interpretation. Table VII summarises the activity distribution between the supernate and Nyex[®] after treatment, reported as the observed phase distribution relative to the calculated level of radioactivity arising from the processed oil (from the baseline fingerprint), Assessment A, and also in terms of the directly observed activity balance, Assessment B.

It can be seen that in general the correlation between the two assessments is excellent for the activation products and Sr-90, which suggests that the entire radioactivity is accounted for. For these radionuclides 80 - 90% of the total is associated with the supernate phase and 10 – 20% associated with the solid Nyex phase. Data related to Cl-36, Fe-55 and Ni-66 are not reported since their observed waste activity concentrations were, typically at or below the analytical limits of detection.

Table VII. Radioactivity Partitioning Data

Nuclide	ILW Trial ID	% Distribution Relative to Fingerprint inferred System Activity (A)		% Observed Activity Distribution (B)	
		Supernate Phase	Nyex Solid Phase	Supernate Phase	Nyex Solid Phase
H-3	Ti04	1213	370	77	23
	Ti05	476	277	63	37
	Ti06	204	82	71	29
Co-60	Ti04	98	0	100	0
	Ti05	91	10	90	10
	Ti06	87	19	82	18
Eu-154	Ti04	124	13	90	10
	Ti05	113	14	89	11
	Ti06	103	17	86	14
Eu-155	Ti04	64	8	89	11
	Ti05	69	7	91	9
	Ti06	66	9	88	12
Sr-90	Ti04	103	23	82	18
	Ti05	104	14	88	12
	Ti06	97	17	85	15
Cs137	Ti04	361	215	69	37
	Ti05	209	215	56	44
	Ti06	215	166	57	43
Am-241 (γ -Spec)	Ti04	71	11	87	13
	Ti05	76	9	89	11
	Ti06	78	12	87	13
Am-241 (α -Spec)	Ti04	78	12	87	13
	Ti05	80	10	89	11
	Ti06	84	13	87	13
Pu- 239/240	Ti04	54	9	86	14
	Ti05	57	2	96	4
	Ti06	37	10	79	21
Pu-238	Ti04	54	9	85	15
	Ti05	57	2	97	3
	Ti06	40	11	78	22

There are however, disconnects between the two assessments for H-3, Cs-137 and also the Plutonium alpha isotopes (Pu-238, and Pu-239/240).

With regards to H-3, this is a highly mobile species and it is not unexpected that Assessment A should give rise to > 200% of the fingerprint implied added H-3 in each phase. Clearly the baseline fingerprint for this radionuclide is suspect, and so the observed activity balance is a more reliable report of the distribution of the H-3 activity between the supernate and Nyex phases. In this case 70% of the H-3 activity is associated with the supernate phase and 30%

with the solid Nyex phase. This would suggest that the Nyex phase tritium may be associated with trace organically bound tritiated species. It should be noted that the tritium is a minor component of the oil inventory, where the actual measured Nyex activity is 0.4 – 0.9 Bq per gram ($\pm 20\%$). These results are in line with the results from the M-ORC study.

The situation with regard to the actinides requires special attention. The Am-241 (assessed by both gamma and alpha spectrometry) distribution implied by both assessments is very consistent indicating 90% in the supernate phase and 10% in the Nyex phase, suggesting that all the Am-241 is accounted for. However subsequent gamma spectrometry of electrode plates retrieved from the end of the ILW trial clearly demonstrated that Am-241 and Cs-137 contamination. When this is taken into account, then the distribution implied from the final gamma spectrometry Am-241 measurements (for trial phase Ti06) is as reported in Table VIII.

Table VIII. Am-241 Secondary Waste Distribution as a Result of the Processing of ILW Oil

Am-241 Secondary Waste Distribution Assessment	A ^a	B ^b
Supernate Phase (Electrolyte + catholyte)	78	70
Nyex Solid Phase	12	10
Electrode	22	20

^a Assessment A – Observed % radionuclide activity distribution relative to the calculated added radioactivity inferred from the GAU fingerprint for the processed ILW oil.

^b Assessment B – Observed % radionuclide distribution

The situation with Cs-137 is similar. When the electrode bed Cs-137 burden is taken into account Assessment A, (Table IX) clearly demonstrates the inherent heterogeneity of the ILW oil for this radionuclide and so on the basis of the data-sets for the other radionuclides, Assessment B is considered to be give a more reliable picture of the Cs-137 distribution, Hence 42% is associated with the supernate with the remainder distributed between the Nyex adsorbent and the carbon electrode bed.

Table IX. Caesium-137 Secondary Waste Distribution as a Result of the Processing of ILW Oil (Final Measurement; Sample T106)

Cs-137 Secondary Waste Distribution Assessment	A ^a	B ^b
Supernate Phase (Electrolyte + catholyte)	215	42
Nyex Solid Phase	166	33
Electrode	127	25

^a Assessment A – Observed % radionuclide activity distribution relative to the calculated added radioactivity inferred from the GAU fingerprint for the processed ILW oil.

^b Assessment B – Observed % radionuclide distribution

This may explain why for the Plutonium isotopes there is a large disparity between the different assessments of the activity distribution reported in Table VII. It is unlikely that the Plutonium would be any less homogeneously distributed than the Am-241 in this oil, so the observed disparity could be due to a higher fraction of the Plutonium associated with the carbon electrode plates. If Assessment A is correct then from the available data, it would appear that from the final measurement 40-50% of the Plutonium is unaccounted for, the implication being that this is associated with the electrode.

As a consequence one of the Titan plant electrode plates has been secured for further study of its radioactive burden and its distribution within the body of the electrode.

Assessment of Gaseous Discharges

The main purpose of the M-ORC trial was to provide an assessment of the secondary radioactive wastes, particularly with reference to the gaseous phase. Since the process produces hydrogen and other gases, the possibility that radioactive species, particularly tritium, could end up in the gaseous phase was considered. Samples were taken for analysis throughout the trial for on-site monitoring and a further sample at the end of study number MO4 was sent for analysis by GAU. These results showed that only negligible amounts of tritium were detected, requiring no further treatment before direct discharge to atmosphere (Fig. 6). The sample taken for analysis by GAU gave an activity value of 0.007 kBq/m^3 , very much in line with the site samples.

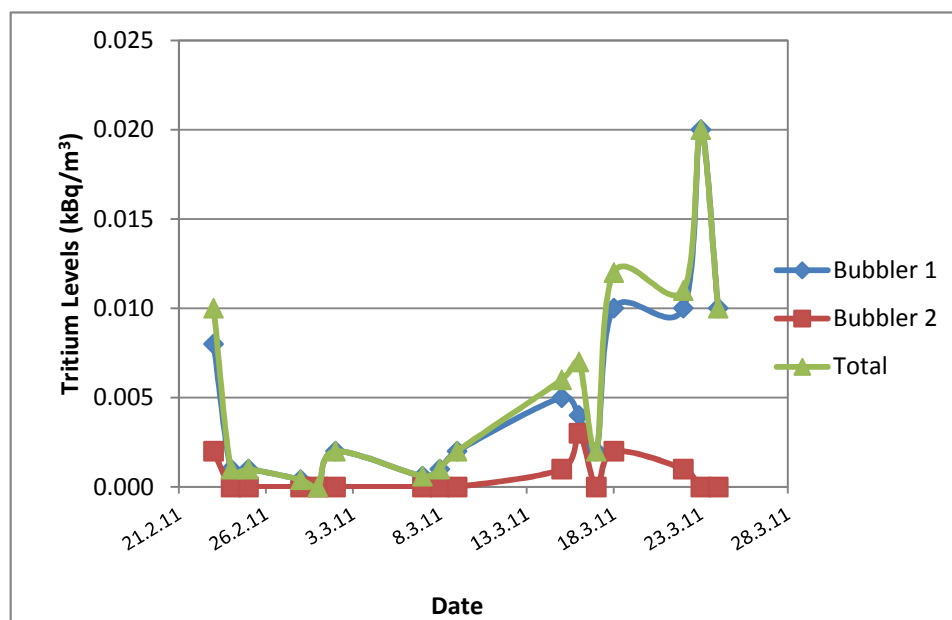


Fig. 6. Levels of Tritium in the M-ORC exhaust gas bubbles

Disposal of Main Secondary Process Waste

To confirm that the all wastes can be routinely disposed of through existing Magnox site disposal routes, after LLW oil trial TI03, the supernate (combined catholyte and electrolyte) and Nyex[®] were removed and processed as follows:

- The recovered supernate and associated cell washings were neutralised using 47 % w/v sodium hydroxide to a pH of between 6 and 8, the discharge consent from the active effluent treatment plant (AETP) being pH 6 – 9 and successfully discharged to the site active effluent treatment plant (AETP) for disposal via the site active drain..
- The spent Nyex[®] graphite flake adsorbent was collected in a 205 litre drum as a wet cake. When dry, this will equate to less than 50 litres of LLW Nyex[®] to be transported to the UK

Low Level Waste Repository for final disposal. An identical procedure was followed subsequent to the final stage (TI06) without any issues arising from the higher levels of radioactivity.

CONCLUSIONS

- The Arvia[®] process of adsorption coupled with electrochemical regeneration has successfully demonstrated the removal and destruction of LLW and ILW radioactive oils on a nuclear site. Over 99.9% of the emulsified oil was removed, with the majority of the radioactive species transferred to the aqueous, supernate, phase (typically 80 – 90 %). The exception to this is Cs-137 which appears to be more evenly distributed, with 43% associated with the liquid phase and 33 % with the Nyex, the remainder associated with the electrode bed. The situation with Plutonium may be similar, but this requires confirmation, hence further work is underway to understand the full nature of the electrode bed radioactive burden and its distribution within the body of the electrodes.
- Tritium gaseous discharges were negligible; hence no off-gas treatment before direct discharge to atmosphere is necessary. All secondary wastes were suitable for disposal using existing disposal routes, with the majority of the activity being successfully discharged as active water via the site active drains.
- Oil destruction was achieved at a rate of 28.2 ml/hr using a regeneration energy of 42.5 kWh/l oil. The treatment of different active and non-active oils was achieved using the same operating parameters, providing strong evidence that the process is robust and will treat a wide range of oils, organic wastes and additives.
- Currently the design of a plant capable of processing 1000ml/hr is being established in discussion with Magnox Ltd. The plant will run automatically with little operator attention and so process between 5-8 m³ of ILW oil per annum.

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