ENVIRONMENT '75

'WASTE WATER TREATMENT IN THE NUCLEAR INDUSTRY'

by

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1. INTRODUCTION

The nuclear fuel cycle comprises all the activities connected with the fuel in the production of electricity from fissile materials, and includes mining and milling of uranium ore, conversion to uranium hexafluoride, enrichment of uranium-235, fabrication of fuel, operation of nuclear power reactors, reprocessing of spent fuel and treatment, storage and/or disposal of radio-active wastes.

The waste water streams which occur in the nuclear industry are very similar to those found in any chemical process industry, except that they are contaminated with radioactivity. Even for high concentrations of radioactivity, treatment is concerned with the efficient removal of low concentrations of radioactive chemical contaminants from waste waters ranging from those which are otherwise clean, e.g. cooling waters, to those with high concentrations of dissolved solids. The problems in treating these wastes with their highly diverse chemical and radiochemical content and the high degree of monitoring and control necessary for safe handling, allied to very restrictive legislation on releases, call for more elaborate waste management techniques than have been common in other industries. This experience will be valuable to industries which are becoming subject to more stringent pollution control legislation and to the requirement for on-site treatment of wastes.

This paper reviews only those techniques used to treat wastes containing low levels of radioactivity with the object of re-using or releasing the water. Liquids containing high levels of radioactivity are a major concern in the nuclear industry but the techniques proposed for their treatment[1] are more specialised and of less general interest, and are not discussed further.

2. CLASSIFICATION OF WASTES

The radionuclides in waste water streams vary throughout the fuel cycle. Natural uranium is very slightly radioactive and has associated with it a chain of radioactive daughter products, including radon gas and radium. Thus, until the fuel is irradiated in a reactor, uranium and its daughters are the only source of radiological hazard. In the reactor very large quantities of radioactive nuclides are formed by three processes: fission, neutron absorption in the fuel to form transuranium elements, and neutron activation of materials in the coolant.

Fission products are the major sources of radioactivity, there being about 200 different nuclides, some of which are highly radiotoxic. The transuranium elements, plutonium, americium and curium, have high alpha radioactivity, very long half-lives and extreme radiotoxicity which means that they present a long-term management problem as wastes, but they can be recycled in fuel. The transuranium elements and the bulk of the fission products are retained within the fuel elements until the reprocessing stage. The activation products and a small amount of fission products are present in the reactor coolant and hence in reactor waste streams.

Waste streams are first classified qualitatively, on their chemical nature, e.g. acid, alkaline, organic-containing, toxic etc, and then quantitatively, on their radioactivity. The concentration of radioactivity in liquid wastes from the nuclear fuel cycle varies from thousands of curies per litre to less than microcuries per litre. A common procedure is to classify these wastes as 'high', 'intermediate or medium' and 'low' level wastes. The actual levels of radioactivity covered by these terms vary greatly depending on the site and on local or national customs. In an attempt to provide a unified categorisation, the IAEA[2] has defined a more complete system of five classifications of radioactivity. As a guide, however, a typical classification used in the industry[3] is:

low level	-	microcuries per litre;
medium level	-	millicuries per litre;
high level	-	curies per litre.

The use of concentration alone is not sufficient as a basis for classification. The levels of release from any site are also defined by classifying radionuclides in terms of their radiological properties, i.e. their radiotoxicity, half-life and radiation-emission, as well as their behaviour in biological systems and their interaction with the environment, particularly possible concentration mechanisms.

The basis of most national legislation for radiation protection standards is the recommendations of the International Commission on Radiological Protection[4]. A widely accepted procedure for the determination of allowable releases to the environment is first to establish the composition of the waste and propose a procedure for release, and then to find and quantify over a lengthy period the routes (both chemical and biological) by which radioactivity may return to man. In practice, one or two of these routes are normally found to be significant and the release limits set are such that exposure by these routes is limited within the ICRP recommendations.

This method of establishing release limits is termed the 'critical path approach'[5,6,7]. Widespread monitoring of the major critical pathways is then carried out during subsequent operation to confirm the original predictions.

3. CHOICE OF WASTE TREATMENT PROCESSES

There are three concepts common to the management of all wastes in the nuclear industry:

- Concentrate and confine.
- Dilute and disperse (under conditions governed by legislation).
- Delay and decay.

The first two concepts lead to the terms, storage and disposal. The first implies that the material is retained in such a way that it keeps its identity and can be retrieved. The second implies that the material is irretrievable and loses its identity, e.g. in the case of liquid wastes with very low levels of radioactivity, they may be dispersed into natural waters[8,9] or into the ground[10]. The delay and decay concept for dealing with radioactive wastes arises because each radionuclide has an unchangeable half-life and decays to a stable isotope if stored for sufficient time. In practice, radioactive wastes are usually managed by a combination of these three possible procedures. The object of treatment methods for low- and medium- level waste water streams is usually to separate and concentrate the radioactivity for storage, passing the purified water to further processing and/or eventual release or recycle.

In the nuclear industry of various countries[11-16], the three basic unit processes used over the last 20 years for the treatment of low- and medium- level wastes have remained chemical methods, ion exchange and evaporation. The overall requirements for treatment are usually met by a combination of these methods allied to suitable filtrations[11,12]. The choice of processes depends on the degree of purification required to meet licensing restrictions for discharge or specifications for recycle. It should also be remembered that treatment is often required for reasons other than the removal of radioactivity; there may, for example, be restrictions relating to chemical toxicity, solids content, acidity or B.O.D. Table 1 gives a simplified summary of the major waste water streams produced in the nuclear industry and the processes used in their treatment.

Although the basic processes have remained unchanged over the years, there have been considerable advances both in modifying and refining these processes and in the development and evaluation of new techiques. Many processes have been examined including foam separation, electrodialysis, reverse osmosis, steam-stripping and freeze-thaw processing. In general, so far, these processes have not found any major application in nuclear waste water treatment but, with the current trend towards reducing any release of radioactivity and, where possible, re-using process water, they are quite likely to find a place in future waste management schemes.

The major processes of chemical treatment, ion exchange, evaporation as well as some processes under development are described briefly in the subsequent sections of this paper.

4. CHEMICAL TREATMENT

Chemical treatment[3,17] consists essentially of coagulation-flocculation and sedimentation processes. In its simplest form, it can consist of a lime neutralisation treatment but often neutralisation is a prerequisite to a flocculation-coagulation step which provides improved decontamination.

In general, chemical treatment is the least expensive processing method and is used for treating and clarifying both small and large volumes of water which contain high total and suspended solids. Chemical treatment can be used to give a gross removal of radioactive material from low- and medium-level waste waters. The decontamination factors* obtained are rarely greater than 10 (90 per cent removal). This type of process concentrates radioactivity into an insoluble sludge and a disadvantage is that the volume of sludge is relatively large, necessitating further treatment before passing to solid waste storage.

4.1 Lime - Soda Process

Early studies[7,17] on the removal of radioactive contamination by standard water-treatment procedures showed that closer control of the processes resulted in improved removals by both cold or hot lime-soda softening processes. Subsequently the method has formed part of the waste water treatment system at a number of establishments[7,18].

4.2 Coagulation-flocculation with Iron and Aluminium Salts

Another standard method of water treatment which is very widely used in the nuclear industry is to add alum or ferric salts and raise the pH by the addition of lime, soda-ash, or caustic soda to precipitate their hydroxides. The treatment involves the formation, coagulation and precipitation of chemical flocs that take up any finely divided, suspended, and colloidal matter. Soluble ions can also be removed by precipitation of hydroxides and basic carbonates during the neutralisation step and by adsorption/ion exchange processes on the floc or by entrainment in the loose floc structure.

Generally, soft waters coagulate best with alum over a narrow range of pH values between 5.8 and 6.4, and harder waters at pH values of 6.7 to 7.8. Ferric hydroxide coagulation can be carried out at very much higher pH values and consequently usually gives better removal of hydrolysable ions[17]. A decontamination factor of about 10 is usually possible, depending on the radionuclides present in the water. Polyvalent cations of Group III and above, including the rare-earths, are usually well removed. The α-emitting transuranium elements, such as

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* Decontamination Factor (DF) is the ratio of the initial amount of activity to residual activity after treatment.

plutonium, are very efficiently removed, particularly by the high pH iron floc[19]. Group II metals are only poorly removed[19] and the monovalent Group I metals are virtually not removed. Among the more important radionuclides that pass through these effluent treatments are caesium-137, strontium-90, radium, ruthenium-103 and -106, cobalt-60, iodine-131, phosphorus-23 and sulphur-35.

The presence of any chelating agents such as citric acid or EDTA in the waste can markedly affect the efficiency of the process. This can be important in wastes arising from processes such as decontamination and solvent extraction. The use of an iron floc with a high pH is more efficient in overcoming the chelation effects[7].

The type of equipment used in these processes in nuclear establishments differs little from modern conventional systems[17]. The radioactivity is concentrated into the floc, and it is vital that carry-over of floc particles be avoided if a high removal efficiency is to be obtained. For this reason, the use of sludge blanket clarifiers is greatly favoured. Passage of the water through the sludge blanket increases filtering efficiency and the presence of a high volume fraction of floc greatly enhances the probability of particle collisions, and hence, the completeness of removal. Filters are frequently used on the outlet to remove any entrained floc.

Contact times and sludge volumes are more important than in normal water treatment plants. Polyelectrolyte flocculating agents are used in the nuclear industry[17,20,21] and result in an increased throughput and a slightly greater decontamination factor. Some clays have ion exchange as well as coagulating properties and can be used both to aid coagulation and to improve the removal of certain radionuclides. Alkali and alkaline earth metals are selectively ion exchanged by clay minerals and the addition of these during normal flocculation processing is used for caesium removal[22]. One problem of additions of this type is that they can greatly increase the final volume of the sludge.

4.3 Coagulation-flocculation with Phosphates

Since heavy and alkaline earth metal phosphates are less soluble than the hydroxides, it is possible to obtain a better removal with basic phosphate flocs[3,7,17]. If the water supply is hard, addition of phosphate followed by alkali to give a pH of 10 will precipitate basic calcium phosphate, which forms a good floc and acts as a carrier for precipitated radionuclides.

Phosphate coagulation has been used extensively in the treatment of low level wastes in Europe[7,23] and found to give decontamination factors of 100 for α-activity and about 10 for β-activity with wastes containing mixed fission products. Strontium is well removed by these flocs but caesium removal is poor, and that of ruthenium depends upon the state of the metal.

4.4 Specific Precipitation Processes

Coprecipitation processes are sometimes used to improve the removal of specific nuclides[17]. Precipitants may be used alone or as additives to the standard coagulation-flocculation treatment. In the latter case, the precipitates formed are scavenged by the floc giving improved settling and removal of finely divided solids. The method is used particularly to improve the removal of anions and those cations mentioned earlier which are difficult to remove by standard hydroxide and phosphate precipitation processes.

Caesium decontamination can be achieved by coprecipitation with metal ferro-cyanides[24]. Radium-containing wastes may be treated using coprecipitation of barium carbonate or barium sulphate[25-27]. The removal of radioiodine, normally present in the anionic state, can be improved by the addition of silver salts[3]. Barium and lead chlorides have been used to precipitate sulphur-35 as sulphate and tellurium-52 as tellurate[17].

4.5 Treatment of Chemical Sludge Concentrates

The chemical sludges arising from the treatment of low- and medium-level waste effluents contain only one to ten per cent solids. The sludges are sometimes stored in this state, but more often, they are subjected to a primary de-watering treatment for volume reduction, usually to about a 20-40 per cent solids content. The processes that have been used include thermal and solar evaporation, pressure and vacuum filtration and centrifugation. The AAEC Research Establishment, Lucas Heights, pioneered the use of solar evaporation for this purpose and has probably the only plant in routine use. Depending on the amount and type of radioactivity in the sludge concentrates, they may be further immobilised by incorporation into concrete or bitumen before storage or disposal. The treatment methods for these chemical sludges are well described by Burns[28] and in an IAEA technical report[29].

5. ION EXCHANGE

Ion exchange processes are widely used in the nuclear industry[30]. Compared to chemical processing, ion exchange offers the possibility of removal of nuclides down to very much lower levels of concentration and, depending on the nuclides being removed and the nature of the waste solution, decontamination factors of greater than 10^3 are common. Ion exchange is applicable only to water with relatively low suspended and total solids, and is normally used as a final polishing step in conjunction with other processes. A major application of ion exchange is the purification of power reactor coolant and blowdown waters where it is used essentially as a polishing process to treat large quantities of relatively clean water[31,32].

Spent resins containing the radioactive impurities are either regenerated in the usual way or treated as solid waste for storage. Very high concentration factors are obtained with ion exchange resins and it may be very convenient to use the process to 'fix' radioactivity in the solid state. Depending on the half-life of the radionuclides, they are either allowed to decay or the resins are immobilised in cement or asphalt and stored[28]. A further concentration of the radioactivity may be obtained by incinerating the resins.

A choice between regeneration and disposal is made on the relative costs of resin replacement and regeneration. Regeneration costs include not only the cost of chemicals but also those of treating the radioactive spent regeneration solutions and rinse waters.

5.1 Synthetic Organic Ion Exchangers

Most applications in the nuclear industry are met by the use of conventional synthetic organic ion exchange resins which have the advantage of a very highly developed technology[30]. Depending upon the nuclide to be removed, single- or dual-column arrangements of cation- and anion-exchange are used. Where maximum decontamination and demineralisation are required, mixed-bed columns are preferred. These are frequently preceded by a cation-exchange bed which has the advantage of increasing the life of the mixed-bed and minimising radiation decomposition in the mixed bed. Although fixed bed operation is normally used, moving bed, continuous counter-current or pulsed bed contactors have found some application[30]. The resin beds concentrate radioactivity and have to be remotely operated and shielded to protect operators. Resin regeneration can also be carried out by remote operation and any resin movements are accomplished hydraulically.

Removal efficiency is governed not only by the form of the radionuclides but also by the type and concentration of non-radioactive ions present in the water. In high ionic strength solutions, competition for exchange sites is much greater and exchange efficiencies of specific radionuclides, present in minute chemical concentrations, are greatly impaired. Thus monovalent ions such as caesium and, to a lesser extent, divalent ions such as strontium are often difficult to remove from many waste solutions; exchangers specific for these elements must be used. For most efficient resin use, total solids should be less than 1000 mg ℓ^{-1} and water with levels greater than 2500 mg ℓ^{-1} is not suitable for ion exchange treatment[30].

Although synthetic organic resins are the most widely used class of ion exchange resin, they have a number of important limitations which include limited radiation and thermal stability, relatively high cost and poor selectivity.

5.2 Synthetic Inorganic Ion Exchangers

To overcome the limitations of the earlier inorganic exchangers[30] such as zeolites and of the synthetic organic exchangers, the nuclear industry has, over the last fifteen years, pioneered the development of high selectivity, inorganic ion exchange materials resistant to chemicals, temperature and radiation. Most of the earlier work was centred on the hydrous oxides of polyvalent metals and the insoluble salts of polybasic acids with polyvalent metals[33,34], such as zirconium hydrous oxide and zirconium phosphates. The range of materials investigated has since been greatly extended[35,36] but so far they have not found any widespread or large-scale use in waste water processing. In general, they are not commercially available in quantity, are mechanically weak and frequently of low capacity and their exchange groups are not stable at high pH. At this stage of development, it would appear that they are likely to find use only in small-scale specialised applications, probably in medium- or high-level waste treatment.

5.3 Naturally Available Ion Exchangers

The use of natural mineral ion exchangers and sorbents has been investigated in a number of countries with a view to making ion exchange treatment economic[7,13,30,37]. The IAEA[37] has reviewed the use of local minerals in the treatment of radioactive wastes, a possibility of great interest in underdeveloped countries. These materials have found uses based primarily on their selectivity and low cost which favours disposal of solid waste rather than regeneration.

The IAEA[37] lists about 150 natural materials such as clays, zeolites, oxides and phosphates which have been evaluated for treating waste water. Clay or zeolite materials, for instance, are commonly used in cation exchange columns but an alternative use, as additives to increase the decontamination obtained in chemical precipitation processes, was mentioned above. Illite, for instance, is used as an additive to fix caesium-137 in the lime-soda process[18].

Certain of the materials commonly classified with these natural ion exchangers actually function via an adsorption/surface precipitation of the contaminant followed by incorporation into the crystal lattice. The use of barytes to remove radium is an example of this type of process; effluent containing radium is passed through high-rate trickling filters containing beds of barytes[25,37] and radium is retained in the barite crystals.

These natural inorganic exchangers suffer in general from a relatively low capacity, chemical instability in acidic solutions, slow exchange kinetics, poor mechanical stability (zeolites) and a tendency to peptise (clays).

Naturally occurring organic ion exchange substances such as humus, lignite, sulphonated bitumen and sawdust have been studied for use in waste treatment and have shown selective removal of various anions[37]. The organic materials, however, suffer from low capacities, chemical and radiation instability and excessive swelling in water with a tendency to peptise.

In general, the synthetic organic resins are preferable for low-level waste treatment but the low cost, selective, natural materials will continue to find applications in specific treatment applications where they are used to fix radionuclides selectively into the solid state.

6. EVAPORATION

Evaporation is used to treat radioactive wastes where a very high level of decontamination is required; factors of 10^4 to 10^5 are possible[38]. The process is well suited to the treatment of wastes which have a high

total solids content (and are thus not suitable for processing by ion exchange), small volume and a relatively high activity.

The range of types of evaporator used for concentrating radioactive waste is very wide, varying from the simple coil or pot evaporators, often used for small installations, to those using natural and forced circulation, vapour-compression, multiple-effect, film and flash evaporation. Spray-drying has also been used for the evaporation and solidification of high solid wastes. The design, operation and relative merits of these evaporators for concentrating radioactive wastes have been described in an IAEA report[38]. Evaporation has the advantage of a very large concentration factor so the volumes of radioactive solids produced are very much lower than those from chemical or ion-exchange treatment.

7. DEVELOPMENTAL PROCESSES

7.1 Foam Separation

Foam separation takes advantage of the preferential adsorption of surface-active solutes at the gas-liquid interface and enables surface-active agents to be removed from solution by forming stable aqueous foams which are continuously separated from the water phase. Ion flotation of inorganic ions from solution can be achieved by adding foaming agents, such as dodecyl benzene sulphonate[39-41], and has been applied to the separation of caesium, strontium and cerium[42-45]. Other ions such as calcium, magnesium and phosphate interfere with the extraction processes and chemical pre-treatment is often necessary. Decontamination factors as high as 1000 have been reported for strontium-90[16].

An alternative variation is precipitate flotation where the nuclide is separated as a precipitate or as a colloid adsorbed onto a carrier such as ferrous hydroxide[46-48].

Foam separation is attractive because of its applicability to very dilute solutions and the good volume reductions which should be possible. The process has been found 'interesting for preliminary decontamination'[16] but probably needs to show an advantage in removal efficiency over ion exchange and chemical precipitation, together with better volume reduction, before it is widely accepted.

7.2 Electrodialysis

Electrodialysis refers to ion transport through selective ion exchange membranes under the influence of an applied electric field. The process separates anions and cations leaving the aqueous solution with any non-electrolytes. The process has been claimed to be economically competitive with ion exchange or evaporation for de-salting solutions containing 500-30,000 mg ℓ^{-1} dissolved solids[49]. At electrolyte concentrations below 500 mg ℓ^{-1}, operating efficiency of electrodialysis falls rapidly because of high solution resistance and membrane polarisation[49]. When strongly ionised ion exchange resin is placed in the electrodialysis compartments it serves as an ion conducting medium[49] and overcomes the increased resistance. This technique is called 'electro-deionisation' and depends on polarisation being sufficient to achieve electrolytic dissociation of water to regenerate membranes and resins. In effect, it can be regarded as a mixed-bed column continuously regenerated by the electric current.

Electrodialysis has been of interest to the nuclear industry in various countries[13-16] because it can deal with total ion concentrations far in excess of those practicable for ion exchange. It is a continuous process which does not require regeneration by the addition of chemicals and so gives rise to only low volumes of radioactive concentrate. Pilot plant work at Harwell, UK[7,50,51] showed that good removal efficiencies were possible, but considerable problems were experienced through membrane failures[52].

The major present interest in the technique appears to be in Russia[14,32] where a 100 m^3d^{-1} electrodialysis pilot-plant facility[53] for the purification of effluent from the Moscow power station has been constructed. The plant has two stages with primary desalting in an electrodialysis unit, followed by polishing in electro-deionisation cells containing mixed ion exchange resins. Each unit has 500 cells. The feedwater is pre-treated by flocculation-coagulation before desalting. The product is stated[54] to be of 'distilled water quality with a radioactivity below the maximum permissible level'. The only radioactive waste is the concentrate product which is less than one per cent by volume of the feed. The results are claimed to demonstrate that purification by two-stage electrodialysis is cheaper than ion exchange because of the better concentration factor obtained and the savings in regeneration chemicals.

7.3 Reverse Osmosis

Reverse osmosis is a pressure-driven membrane process and, like electrodialysis, was originally of interest as a method of desalinating water supplies, but has been receiving increasing attention as a method of treating waste water. Although industrial electrodialysis plants have been available for a considerable time, it is only relatively recently that well-engineered and reliable reverse osmosis equipment has appeared. Considerable effort is still being expended on membrane development[55], currently an area which has the most problems, and reverse osmosis would thus appear to have a great deal of development potential still available as a process for water treatment. Strangely, although several commercial reverse osmosis systems have arisen from the work of the nuclear energy industry on desalination, there appears to be little published data on its evaluation as a possible treatment method for radioactive waste water. Recent Russian work[56] has indicated that very high separations of a variety of fission products are possible even when they are present in extremely low chemical concentrations in high ionic strength solutions.

Reverse osmosis is finding applications in the conventional power industry[57,58] for boiler feedwater make-up

and for treatment and recycle of boiler and cooling-tower water blowdown. Similar applications are being evaluated for nuclear power plants both for make-up water and for the treatment and recycle of radioactively contaminated blowdown water[58,59].

Reverse osmosis appears to hold promise as a method for the deionisation and concentration of wastes in the nuclear industry. It has the advantage of being compact, simple to operate, does not use regeneration chemicals, and appears to have good rejection properties for most nuclides of interest. Disadvantages include its lack of specificity, a tendency to lose flux when treating waste streams containing fouling constituents, and a sensitivity to extremes of acidity or alkalinity.

The future use of the technique in the nuclear industry is probably very dependent on the development of more economic module designs and membrane systems which show an improved resistance to loss of flux through fouling. Greater experience is also required of the long-term behaviour of reverse osmosis systems when treating typical nuclear waste streams.

8. CONCLUDING REMARKS

As stated in the Introduction, the basis of all these processes is the removal of low concentrations of a wide variety of chemical species from water. Because these species are often radioactive when they occur in the nuclear industry, it is necessary to upgrade process performance to achieve the desired decontamination. This same improved performance is available to conventional chemical industry faced with the requirement to treat its waste water streams to more rigid standards of purification.

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