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APPLICATION OF POLYMER MATERIALS FOR ORGANIC LIQUID RADIOACTIVE WASTE IMMOBILIZATION

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The article gives an overview of published data on the experience of polymer materials usage for liquid organic radioactive waste treatment. Advantages and disadvantages of incineration, cementing and other methods are noted. Data on polymer polymeric materials usage for liquid organic radioactive waste treatment and the results of experiments conducted in V.G. Khlopin Radium Institute are presented. The advantages of polymers consist in the use of simple equipment, minimum number of technological operations and the possibility of immobilizing liquid organic radioactive waste into polymer material for subsequent incineration (pyrolysis).

Keywords: *radioactive waste, liquid organic radioactive waste, liquid organic radioactive waste immobilization, polymer materials.*

Introduction

Immobilization of liquid organic waste is believed to be a quite challenging issue deserving some special attention due to a number of reasons. Compared with other radioactive waste, the volume of generated organic waste is much smaller, nevertheless, its safe management requires some special technologies to be applied usually not being considered compatible with already known methods of aqueous solution treatment.

Liquid organic RW is generated at many nuclear fuel cycle facilities, in nuclear research centers, as well as due radioisotope applications in medicine. Most typical organic waste involves: lubricants from NPP operation, diluents from spent fuel regeneration, scintillation fluids from analytical laboratories and several others.

Spent radioactive oils generated at NPPs originate from primary coolant pumps and turbines. These are usually low-level waste (LLW) containing small amounts of radionuclides, mainly such as ^{137}Cs, ^{134}Cs, ^{58}Co, ^{60}Co, ^{65}Zn and others. Tributyl phosphate (TBP) and its mixtures with hydrocarbon solvent used in spent fuel reprocessing is believed to be the second important source of organic LRW generation. Due to irradiation extraction capacities of the components deteriorate: after a series of cycles the extractant is considered unsuitable for use and is categorized as intermediate-level waste (ILW). This waste may contain uranium, plutonium and fission products.

Organic decontamination liquids resulting from various procedures and scintillation liquids should be also considered as sources of organic LRW generation. The latter consisting of a three-component mixture involving dissolved substance (sorbate), solvent and test sample (sample) are used in radiochemical analysis.

Long-term storage is being considered as a possible organic RW management option, nevertheless, causing a number of problems, both environmental

and economic. In addition, during storage, organic waste can decompose releasing radiolytic gases, thus, constituting a fire hazard. In each specific case, an adequate method for such waste processing should be identified based on a careful analysis followed by the selection of the cheapest option enabling to eliminate the waste.

Criteria enabling the decision making on the selection of relevant organic RW processing methods are based on economic aspects of waste treatment (or storage) processes, commitment to waste volume reduction and the complexity of the applied methods and equipment. The impact of any criteria or all of the criteria on the selected waste treatment option may be quite specific for each particular waste generator and long-term storage or disposal conditions.

Conducted study and experiments show that waste immobilization involving the use of polymer materials can be considered as a promising method for organic LRW treatment. The logic of certain technologies development is often prompted by the desire of processing a maximum possible amount of waste. We do not anticipate that polymers can change the established practice in a fundamental way, nevertheless, in many cases polymer materials may be viewed as an alternative option and may help to address some particular issues arising at different facilities.

Organic LRW management technologies

Discussion of polymer material applications seems worth to be preceded by a summary of most commonly applied methods for organic LRW processing.

RW incineration is certainly viewed as a well-developed, albeit rather sophisticated technology. Attractiveness of this method is associated with the combustibility of organic waste and the possibility of achieving a high volume reduction rate. Nevertheless, it should not be forgotten that the incineration process itself can only be considered as a component a complex waste management system. Depending on the ash residue activity, it can be simply packaged or further treated enabling its immobilization into a waste form being considered more suitable for waste storage and/or disposal.

Commissioning of Karlsruhe (Germany) incineration facility in 1977 can be viewed as an earliest example of its industrial application [1—3]. Oganic LRW stream having the following composition was fed to the incineration facility: 40 % of oil, 34 % of solvent, 10 % of scintillation fluid and 16 % of water. A total of 360 m^3 of waste was processed in the first 5 years of its operation.

A facility designed for oil, chlorine-containing solvent, scintillation fluid and TBP incineration was operated in Cadarache (France). Coefficients of volume reduction from 30 to 300 were achieved there [1, 2]. During facility's operation, 95 % of the active ash was captured by filters with only 5 % remaining in the furnace.

A fluidized bed furnace was tested in Rocky Flats (USA) [4, 5]. The unit was designed to process plutonium containing waste. Organic liquid waste consisting of TBP, spent oils and solvent mixtures was fed into the incineration unit. Some issues associated with clogged nozzles due to uneven heating were noted during facility testing.

Extensive knowledge on waste incineration has been accumulated during the long-term operation of MosNPO Radon facilities. The incineration facility designs were based on a two-chamber furnace: one chamber is used for waste incineration in a fire grate, the other is used for precipitation and afterburning of coarse aerosol fractions. Flue gases are treated in a dry-type system involving cooling, coarse and fine filtering stages. The furnace capacity amounts to 80 kg/h, the temperature in the incineration chamber reaches 900—950 °C, the specific consumption rate of liquid fuel is up to 0.25 kg per 1 kg of waste with a 30—100 volume reduction coefficients being achieved.

Several types of secondary waste products being generated (ash, soot, condensate, exhaust gases) are considered as a disadvantage of the method. These must be conditioned with the cementing method being usually applied as such. It should be noted that in addition to processing, any pre-disposal technology requires waste conditioning with glass or cement compounds being most commonly considered as a matrix material.

Recently, much attention has been paid to catalytic oxidation to gaseous and solid compounds as a promising organic LRW treatment method. [6] discusses the oxidation of organic waste containing tributyl phosphate (TBP).

The experiments showed that out of 10 synthesized catalysts lanthanum-cobalt catalyst has the highest activity. TBP conversion at a temperature of 170 °C amounted to 80 %. Global practice shows that thermocatalytic method is constantly upgraded with a trend suggesting the application of some new catalysts (not containing any precious metals) and hardware optimization.

As it comes to the incineration method being applied at industrial sites, combination of organic waste incineration with a high-temperature vitrification process is believed to be a most promising option. Obviously, in this case no particular facilities should be purposely developed, with already available ones being put in place.

A method described in the patent [7] can be considered as an example of such an approach. A radioactive mixture of tributyl phosphate and n-paraffins (melt temperature 800—1200 °C) is fed into the apparatus containing aluminophosphate melt of vitrified RW. Oxidizing agent (oxygen) is introduced into the combustion zone simultaneously with water vapor, gaseous carbon oxides and a solid phase predominantly containing phosphorus oxides and the majority of radionuclides being formed due to the oxidation of organic substances. In this case, the phosphorus oxides from TBP incineration are almost entirely (88—100 %) melted into aluminophosphate glass.

The process of organic waste mineralization, mainly involving the residues from distillation purification of the extractant (TBP) is discussed in [8]. The process starts with waste pre-treatment by adjusting its composition, mixing $Mg(OH)_2$ and emulsifying the mixture. Further, due to TBP pyrolysis and water evaporation, mineralization occurs in the reactor and, finally, magnesium phosphate precipitate is formed. The resulting ash residue is subject to cementing.

Certainly, incineration and pyrolysis processes are characterized by a number of benefits with a significant waste volume reduction being considered as a most important one. The required rather sophisticated equipment can be viewed as its disadvantage. In addition to the furnaces themselves, the units may feature afterburners, heat exchangers, a cold air supply system, filters, gas scrubbers and dust collectors. Thus, this method requires large capital investments with high operation and maintenance costs. However, even a mature technology can result in some problems associated with incomplete combustion, corrosion of equipment elements and purification of exhaust gases.

Any decision on the use or rejection of organic RW incineration method should be probably based on the specific economy of each individual waste storage/disposal scenario. On the other hand, if the waste storage costs are so high that the volume reduction procedure seems to be attractive, the incineration process can be selected as a preferred option even despite the high costs involved.

Among other methods applied for organic LRW treatment, the processes of "wet" oxidation, adsorption and cementing should be mentioned as well. "Wet" oxidation process was studied in Japan as a potential option enabling the decomposition of low-level organic waste (solvents). Some papers discuss a possible option of this process application: waste processing takes place at a temperature of 100 °C and under an atmospheric pressure using a copper catalyst and hydrogen peroxide [9, 10]. Since "wet" oxidation requires strict control of chemical reactions at high temperatures, it can be assumed that the industrial scale application of this process would require the development of a complex control and process water treatment system.

Introduction of organic waste sorption process seems to be a rather simple solution being applied in many areas. There are many absorbents ranging from very simple (such as sawdust) to rather complex (alkyl styrene polymers). The use of adsorbents can increase the waste volume to 300 %. Suitability of absorbents alone as organic LRW hardeners seems to be quite minor: their effectiveness decreases significantly if water or other ionic impurities are available, whereas a change in the viscosity can cause a significant decrease in the amount of absorbed liquid.

As it comes to cementing, it should be specifically noted that Portland cement appears to be ineffective in immobilizing any liquid organic waste. Normally, only about 12 % (vol) of oil can be contained inside the cement waste form with an acceptable compressive strength of the final solid product. However, waste containment capacity can be significantly increased by introducing suitable moisturizing agents. Laboratory studies of organic LRW cementing were carried out at the Brookhaven National Laboratory [11]: several options enabling the industrial implementation of the process are known. Envirostone method involves the use of a patented gypsum cement with the addition of a polymer and a special emulsifier to incorporate organic liquid into the emulsion [11]. Under this process, organic LRW is mixed with an emulsifier and then gypsum cement is added. The ingredients are stirred until a homogeneous mass is formed. The latter one is poured into containers, where the solidification occurs. This method results in solid waste forms being filled to up to 30—50 % by organic ingredients.

Similar operations were performed at Hanford to immobilize pump oils, mineral alcohols and TBP mixture with solvents [12].

A comprehensive method for radioactive oil cementing has been developed by the State Unitary Enterprise MosNPO Radon with an additional module added to already existing pilot-industrial facility designed to perform pre-cementing operations. The module is designed to mix up a suspension made up of liquid inorganic radioactive waste (LRW), treated radioactive oils and powder additives providing for an even distribution of organic components within the hardened cement compound. The mixed-up radioactive oil being a homogeneous suspension is fed to the cementation unit. A complex additive ("Bizon") made up of bentonite, fine-ground quick-hardening cement and

microbiological compound protection is proposed to be fed into the suspension [13].

Pilot-industrial tests showed that the oil content in the compound can be brought up to 15—20 % and the process behavior of the final cement compound will not suffer any deterioration [14].

A combination of absorption and cementation processes can be applied to treat liquid organic waste. In this case, instead of being converted into an emulsion, liquid organic waste is absorbed by an adsorbent resulting in a solid that can be further incorporated into cement.

The resulting waste forms may contain up to 56 % of organic waste by volume. Absorbent saturation with an organic liquid followed by its mixing with cement and water in an amount required for cement hydration is seen as common procedure. If the organic waste already contains water, it may not be further added. However, if the amount of water is insufficient for normal cement hydration, the resulting product will have a low strength. Despite some advantages of this method enabling to achieve an increased waste containment capacity, it still has an important disadvantage: the liquid should be immobilized before curing base is added.

It should be also emphasized that none of the processes considered above is universal, i. e. it is impossible to use the same type of equipment and the same processes to treat all types of organic LRW.

Experience of applying polymer materials in organic LRW processing

IAEA technical report [15] provides an in-depth discussion of polymer applications in RW processing. However, for a number of reasons due attention has not been given to this matter with the successful industrial implementation of the cementing method that could be primarily noted as one of these reasons.

This section describes organic LRW management with polymers of various compositions being applied in one way or another [15-18].

[19—22] provides some case studies suggesting the use of polyethylene in RW immobilization at NPPs.

Polyester resins were used in a process developed at the Grenoble Nuclear Research Center (CEA) [23] enabling industrial-scale processing of waste from Aden NPP. Later on, this method was reproduced in Japan [24]: it was modified to immobilized concentrated waste and spent ion exchange resins from site No. 2 at Fukushima NPP. Similar process was also developed independently by Toshiba and several other Japanese companies [25].

So-called IREP waste immobilization process suggesting the use of unsaturated polyesters was developed [26]. IREP results in a waste emulsion with unsaturated polyesters and was used to immobilize intermediate-level waste (concentrates from the evaporation plant, as well as residues and ion-exchange resins). Similar methods have been investigated in Canada [27] and in the USA to immobilize waste from nuclear power plants (Dow process [28]).

Successful implementation of the process requires the formation of an oil-in-water emulsion being further slowly introduced by stirring into a polyester polymer to form a secondary emulsion. Immobilization is triggered by adding a catalyst. The process was used to immobilize concentrates from evaporation plants, ion-exchange resins and post-decontamination solutions. It was adapted to enable its application at European facilities [29]. However, further studies were discontinued: it appeared that under a waste loading of over 5 %, the waste-containing emulsion deteriorates inside the polyester polymer preventing the product from hardening.

As for other polymer materials applied, special consideration should be given to urea-formaldehyde resin (UF resin). Viscous emulsion of urea and formaldehyde is completely miscible with water, but cannot be mixed with non-polar solvents. Due to condensation, addition of an acid catalyst results in a cross-linking. By-water is captured by matrix during polymerization. An aqueous solution of sodium bisulfate is usually applied to stimulate the process.

No chemical reaction can occur between UF resins and waste: they polymerize forming a microstructure similar to comb cells with their voids providing waste containment. The volume of the product obtained is the sum of the waste and UF resin volumes. Liquid radioactive waste is mixed with reagents capable of forming solid products with chemically stable products being ultimately formed.

Restrictions on the chemical composition of the waste that can be recycled is seen as a disadvantage of this method (it cannot be applied to treat solutions with high acidity and some other types of waste that cannot be easily mixed with UF resins).

In 1970—1980, UF resins were considered as the main curing agent used in the USA for waste immobilization purposes, such as dehydrated filter sludge and spent resins from demineralization facilities. However, due to newly introduced regulations restricting free liquid content in waste packages these were abandoned since the amount of liquid in the final product exceeded the standard values suggested by regulations [15].

A mobile unit for spent ion-exchange resin immobilization using a styrene-divinylbenzene mixture was developed in Germany [30]. Later on, such facilities were used in a number of countries [31].

Polyvinyl chloride (PVC) was used from 1971 to 1980 to immobilize spent extractants (TBP in kerosene) at WAK plant in Germany [15]. TBP presumably acts on PVC as a plasticizer. A solid product with mechanical properties similar to the rubber ones (product density of some 1.12 g/cm^3) is formed upon TBP diffusion into PVC pellets. Containment capacity of PVC with a high molecular weight is higher than the one of polyvinyl chloride with lower molecular weights, which, however, can be mixed more easily at a room temperature and absorbs TBP faster.

Quite a long time ago, a process enabling the immobilization of all RW types and spent ion-exchange resins using polyurethane was developed in Sweden [15—18]. It was proposed to use hydrocarbon oils as cheap additives to polyurethane. However, this method has not been commercialized.

A number of studies on the application of polymers in the immobilization of organic liquid radioactive solutions was also performed in Russia [32]. Experiments were conducted to study the immobilization of spent extractants and spent engine oil using a diene copolymer. High efficiency of the polymers in the immobilization of LRW having complex composition was demonstrated. For spent oil (emulsion) solidification, the rate of tritium leaching from the samples amounted to 10^{-3}—10^{-4} g/cm^2·day. The leaching rate of cesium and americium in the experiments was found to be equal to 10^{-5}—10^{-6} g/cm^2·day.

[33] summarizes the study on the immobilization of organic LRW using polymer and cement matrices. According to the authors, despite a significant increase in the volume of the final product to be stored, immobilization with a polymer matrix may be considered as an acceptable option taking into account the increased environmental safety and relatively small volumes of organic LRW generated by enterprises.

Quite recently, developed was a new class of polymer materials (corporation "Nochar", USA): over the past 15—20 years relevant research has been performed to study the use of these polymers for LRW immobilization purposes, including organic waste [34—37].

The research has shown that the polymers are able of bonding the organic liquid with a soft porous rubber-like material being obtained as the final product. This product can be sent for long-term storage or incorporated into a cement matrix.

Summary of experiments on the application of Nochar polymers

Nochar PetroBond polymer No. 910 was used to immobilize organic waste. Since liquid waste, in addition to organic liquids, contains an aqueous phase, a combination of different polymer materials was used to immobilize the entire LRW mass (No. 910, No. 960).

Waste from the decontamination unit and waste from laboratories was involved in the experiments (Table 1). Figure 1 shows the samples after the immobilization took place.

Table 1. Inventory of waste used in the experiments

Product name	Specific activity
Vacuum oil	No analysis was performed
Organic sediment from LRW storage tank	$A_{\Sigma\alpha}$ – $6.6\cdot10^6$ Bq/l, $A_{\Sigma\beta}$ – $1.1\cdot10^7$ Bq/l
Liquid scintillator (ZhS-8) containing tritium water	No analysis was performed
30% TBP in a hydrocarbon diluent	$A_{\Sigma\beta}$ – $1.8\cdot10^7$ Bq/l
Organic discharges from laboratories. Aqueous phase is available (from 3 to 10 %)	$AA_{\Sigma\beta}$ – $3.6\cdot10^6$ Bq/l,

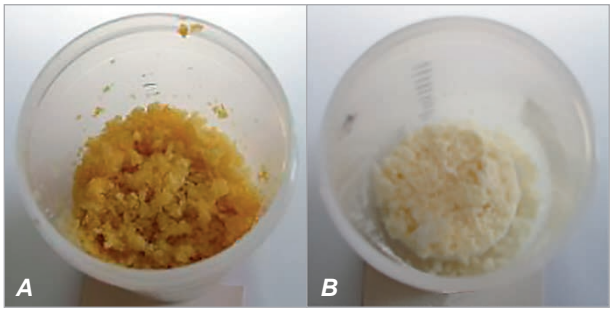

Figure 1. Samples after vacuum oil (A) immobilization and spent scintillator (B) immobilization. Polymer No.910 was applied with the ratio of the polymer mass to the liquid mass (T:W) of 1:5

Introduction of polymers into LRW container was performed either by stirring or the polymer material was simply impregnated with the solution. It should be noted that the immobilization process can be implemented also directly inside waste containers containing the waste subject to processing which are further used as transportation containers for subsequent waste shipment from the site.

In addition to the experiments with solutions, the composition of which is given in Table 1, some 25 liters of organic liquid waste of unknown composition with high specific activity were immobilized as well. These measures were purely practical and

were performed to clean the repair areas of "hot" chambers from accumulated solutions that could not be recycled via cementation.

Additional isolation of solidified waste involved mixing of the resulting compositions with a cement solution. Immobilized waste was packaged into polypropylene containers and poured with a cement solution.

We believe that a comprehensive technology involving organic (or mixed aqueous-organic) RW immobilization into a polymer matrix with subsequent high-temperature treatment of the solidified waste could be seen as a quite interesting option [38].

Our experiments suggested the application of high-temperature treatment (carbonization) process implemented in a confined space (temperature range of 600—700 °C) as an alternative to the combustion process. After its completion, the mass of the residue did not exceed 5—15 % of the initial LRW mass. Resulting ash residue was cemented.

The above data shows that the high-tech polymers proposed by Nochar have a wide range of applications when it comes to addressing organic LRW immobilization challenges.

Conclusion

None of the methods used for organic RW processing can predominantly overmatch the others. A most effective method should be selected based on a preliminary analysis and cost estimates only then followed by a feasibility study of particular methods.

This approach involves an objective comparison of options enabling relevant decision making with the environmental risks and implementation costs being considered as the main focus areas with due account of the subsequent costs associated with the isolation of the final products in a repository.

The conducted studies demonstrate that the immobilization of organic waste using polymer materials should be seen as a promising area in organic LRW processing and deserves some careful attention.

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