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Advanced concepts for uranium and plutonium multi-recycling

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Abstract – The multiple recycling of plutonium and uranium is a main stake to gradually achieve a fully closed nuclear fuel cycle in the future once first Generation IV fast reactors will be deployed into the fleet. To reach this objectives, optimizations of current processes and technologies and, in some cases, development of innovative concepts are required to meet the scientific, technical and economical challenges imposed by the treatment of spent fuels containing increasing Pu content. Based on the significant past experience acquired in France in LWR and FR MOX fuel treatment, R&D studies are in progress at CEA to develop new efficient processes for Pu recycling and for FR MOX fuels fabrication. More compact and economically competitive processes, ensuring the safety/security of the facilities (regarding in particular criticality and proliferation resistance) have to be developed where as plutonium amounts will be one order of magnitude higher than in UOX fuels. New concepts for the head-end operations for MOX dissolution, for the separation of plutonium and uranium by solvent extraction with higher Pu/U ratios, for their conversion into mixed oxides, for MOX fabrication avoiding powder milling, and for optimized management of effluents and waste have been put forward to propose an ambitious program for the 4th generation plants.

I. INTRODUCTION

In the present closed fuel cycle, the buildup of non fissile Pu isotopes in thermal neutronic spectrum limits the multi-recycling of Pu to only one or two cycle in Light Water Reactors (LWR). Used LWR MOX fuels are thus currently not recycled and are waiting for next generation reactors and reprocessing plants. Indeed, the potential progressive deployment of Generation IV fast reactors (FR) into the fleet paves the way for the multi-recycling of plutonium as all Pu isotopes are fissile in fast neutron spectrum. The full closure of the fuel cycle would thus be achievable and allow a much better use of natural uranium resources than today by optimizing the energy potential of natural uranium as well as benefits in terms of reduced environmental impact [1].

The french strategy in this field consists first in recovering plutonium from used MOX fuels unloaded from LWR with the objective to make the first cores for fast reactors. At a longer term, the goal will be to reprocess these spent FR MOX fuels in order to multi-recycle plutonium in these reactors, without any limits imposed by the Pu isotopic composition [2]. Plutonium, recovered

from used MOX fuels, will substitute enriched ²³⁵U and will be generated as well in the reactor by neutronic capture on fertile ²³⁸U. Uranium ores could be closed and energy production will arise from plutonium and uranium multi-recycling from spent nuclear fuels completed by depleted uranium.

To reach such industrial goal, treatment and recycling processes will have to be adapted or developed to take into account increasing Pu content in future spent nuclear fuels (1% in UOX, ~ 8% in LWR MOX and up to 30% in FR fuel) and specificities of FR assemblies. Based on the significant past experience acquired in France in LWR and FR MOX fuel treatment/recycling [3], R&D studies are in progress at CEA to develop new efficient processes for Pu recycling from used MOX fuels. In addition to the capacity to process high Pu content fuels, these processes will have to address the following objectives :

- Be more efficient in order to develop more simple and compact processes ;
- Be more reliable and available in order to optimize the maintenance and limit as much as possible the plant stoppages, keeping in mind that

in a closed fuel cycle the recycling products will be the unique source for loading the reactors ;

- Be flexible in order to process LWR and FR MOX fuels as well as UOX fuels during the transition periods between LWR and FR reactors ;
- Increase the global safety of the facilities, especially in terms of proliferation resistance and criticality risks ;
- Limit effluents and waste generation in a growing regulatory and environmental context ;
- Limit as much as possible the environmental footprint of the overall recycling process.

To fulfill these different criteria, the innovative concepts under study should:

- Allow a co-management of uranium and plutonium at every steps of the process to reinforce the resistance towards proliferation ;
- Select compact technologies and fast chemical processes for the different operations to reduce the residence time in the industrial units ;
- Avoid the use of redox reactions for plutonium separation and conversion ;
- Increase the process efficiencies to minimize Pu losses while increasing Pu content fuels will be processed ;
- Limit the radioactive and chemical releases in liquid and gaseous effluents ;
- Be selected by an integrated approach of the whole operations (dissolution, separation, conversion, fabrication, waste management) in order to reach the best compromise between efficiency, cost (investment and operation) and environmental impact.

Based on existing processes already proposed or designed for FR MOX treatment in the past, the program leads to develop efficient and innovative processes able to bring sufficient potential gains once technological issues will be resolved. This new R&D phase closely linked to the Sodium FR ASTRID project [4-6] has been recently launched at CEA [7-8] and some recent orientations and first results are presented here.

II. DESCRIPTION OF NEW CONCEPTS DEVELOPED FOR PU MULTIRECYCLING

Based on the different criteria to fulfill for the future industrial multi-recycling of plutonium and uranium, concepts have been proposed for each of the main operations of the treatment/recycling plant:

- Head-end step including new mechanical operations for the fuel disassembly and a compact fuel dissolution unit ;

- Separation and purification of uranium and plutonium by solvent extraction ;
- Conversion of uranium and plutonium into oxides and MOX fuel fabrication ;
- Waste conditioning and effluents management ;
- Process monitoring and control.

II.A. Head-end operations and fuel dissolution

Recent R&D work in this field has been driven by the AREVA TCP project, a new shearing and dissolution facility in the La Hague plant for dissolution of LWR/FR MOX fuels [9]. The TCP process developed by CEA includes complementary steps dedicated to recover the residual Pu still contained in the fine undissolved particles arising from the dissolution of challenging fuels [10].

To go beyond this process and considering an industrial process able to treat up to hundreds of tons of FR fuels per year at a longer term, new operations have been proposed in order to optimize the whole shearing and dissolution operations. The concept is focused on a full separation of the U-Pu irradiated oxide from the cladding in nitric acid. This separation would allow reducing drastically the size of the dissolution unit and is especially interesting with FR fuels as metallic waste are more important compared to LWR fuels and partially soluble in concentrated nitric acid solutions.

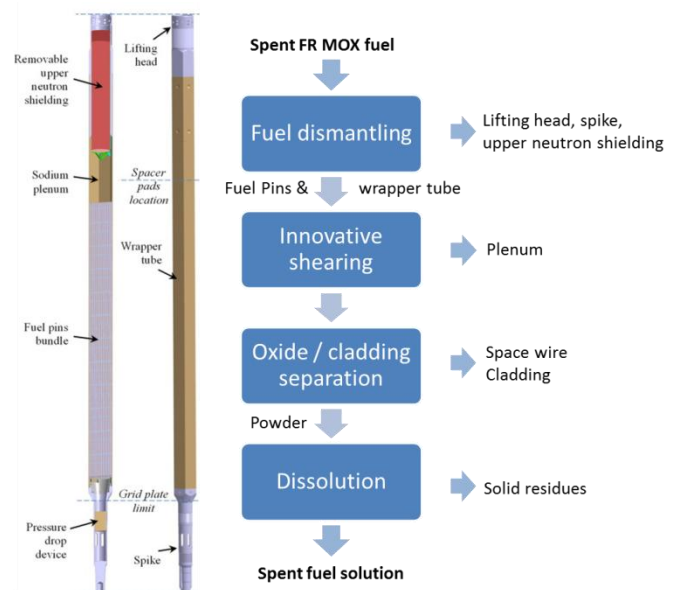


Fig. 1: New head-end operations in the case of FR ASTRID spent fuel treatment

Innovative technologies have thus been proposed for the shearing step and the objective will be to select the one insuring the best separation of the powder from the cladding. After separation, the oxide fuel will be dissolved in nitric acid in a compact unit. A gain up to a factor 10 is

expected on the size of the dissolver unit compared to the industrial unit implemented at La Hague plant. The sequence of operations is reported on Fig.1.

II.B. U & Pu separation

The objective is to recover and purify uranium and plutonium by solvent extraction after dissolution of used MOX fuels. Co-management of uranium and plutonium in the process is encouraged by Generation IV recommendations [11-12] in order to never produce separated Pu and increase the resistance towards proliferation risks.

The PUREX process based on the use of the TBP solvent has already been used to separate U and Pu from LWR MOX and even FR MOX fuels at a large scale (more than 60 t of LWR MOX and up to 25 t of FR MOX fuels treated in the La Hague plants and in the Marcoule Pilot Plant) [3,13]. However, several extraction and purification cycles were required to reach the specifications on uranium and plutonium products. Furthermore, the principle of a PUREX TBP process needs to use redox reagents to control Pu oxidation state during U/Pu separation. This redox is controlled with a reducing and an anti-nitrous reagent (e.g. U(IV), hydrazine,...) or oxidizing reagent (nitrous gas or NOx). If the use of these reagents is managed and optimized at the industrial scale with UOX fuels, the increase of Pu content in LWR and FR MOX fuels will drastically increase the consumption of these reagents if the same process is applied in the future (Table I).

TABLE I

Estimation of the quantity of redox reagents required for the treatment of LWR/FR MOX fuels with a PUREX TBP process

Redox reagent	U(IV)	Hydrazine	NOx
UOX (present)	Ref.	Ref.	Ref.
LWR/FR MOX	× 10 to 15	× 5 to 7	× 5 to 7

In addition to a significant economic and environment gain, the search of alternative extracting systems allowing U/Pu separation without redox is driven by the willingness to design a more simple and compact process for MOX fuel treatment. N,N-dialkylamides have been studied in France [14] and elsewhere [15-16] for their ability to extract uranium and plutonium from concentrated nitric acid solutions and to separate U(VI) and Pu(IV) without redox by decreasing the HNO₃ concentration. Monoamides show also high U/FP selectivity giving the opportunity to reduce the number of extraction cycles to only one (Fig. 2).

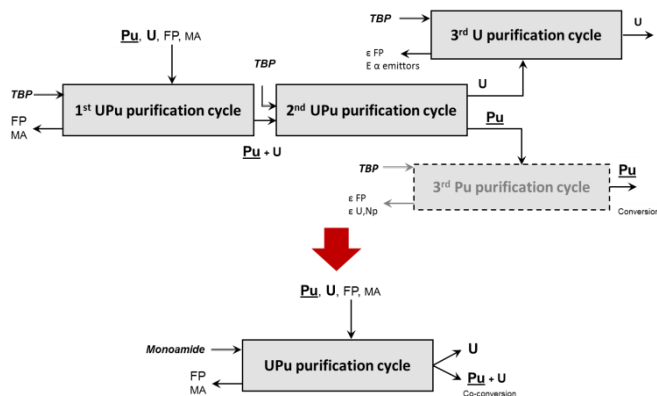


Fig. 2: Towards an innovating separation process of U and Pu; left: the TBP process applied in the past to Phenix FR MOX dissolution liquor, right: the process under development (“one single cycle without redox”).

Based on the experience acquired at CEA on these molecules, the mixture of DEHBA (N,N-di-2-ethylhexylbutanamide) and DEHiBA (N,N-di-2-ethylhexylisobutanamide) was selected for the process development [17]. After parametric batch experiments, a chemical model was elaborated and implemented in the PAREX simulation code. The flowsheet was tested in 2015 in a genuine UOX fuel HA solution in laboratory-scale mixer-settlers in the ATALANTE facility. The scientific feasibility of the “1 single cycle process without redox control” was demonstrated by a quantitative recovery of uranium and plutonium, sufficiently purified from the fission products to reach ASTM specifications [18].

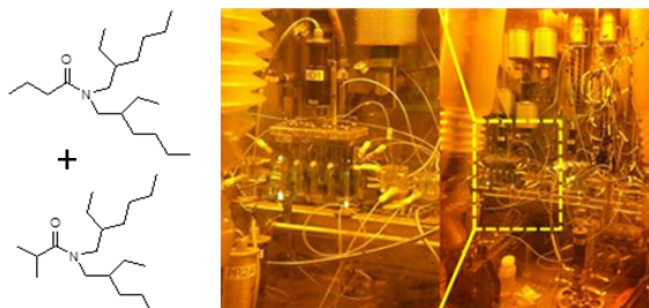


Fig. 3: Structures of DEHBA and DEHiBA monoamides (left) and view of the mixer-settlers used to demonstrate the scientific feasibility of the “one single cycle process without redox” separation process (ATALANTE CBP shielded cells) (right).

R&D is now carried out to adapt this process to MOX fuels with higher Pu content. Less viscous molecules with high Pu+U loading capacity (Limit Organic Concentration before 3rd phase formation) are under development to fulfill the different criteria required for Pu and U multi-recycling.

II.C. U & Pu conversion and MOX fabrication

The conversion step aims to convert U and Pu present in nitric solution (after separation by solvent extraction) into mixed U, Pu oxide or mixture of highly reactive actinide oxides.

In the frame of U and Pu multi recycling for Generation IV systems, the objective is to develop a flexible and efficient process able to manage higher metal flows (due to an increased amount of Pu in the cycle and the addition of U for U-Pu co-management) and without redox adjustment of uranium and plutonium coming from the separation step. The conversion process will also have to precisely control the oxide morphology in order to synthesize actinide precursors suitable to the fuel fabrication step.

Two different conversion routes have been identified to fulfill these criteria and are currently under study:

- The first route, based on our scientific expertise and on the industrial feedback (AREVA La Hague plant) on oxalic precipitation, aims to identify one organic ligand or a mixture of organic ligands leading to the quantitative precipitation of U(VI) and Pu(IV) in a mixed compound or an intimate mixture of compounds, respectively.
- The second route does not involve any precipitation step but is based on a direct denitration of the U(VI) and Pu(IV) solution in presence of organic additives to control the final morphology of the synthesized powder.

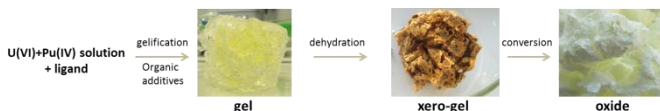


Fig. 4: Scheme of the direct denitration concept in presence of organic additives

Depending on the fabrication process, the conversion parameters should be optimized during (e.g. temperature / atmosphere, morphology, particle size distribution) or after the synthesis (e.g. powder granulation / grinding, specific surface area adjustment) in order to provide the best materials for MOX pellets fabrication.

The main objective of the fabrication step is to produce annular dense pellets at high rate and by minimizing or even suppressing the powder handling in order to limit the dust build-up and thus the radiation exposure risk due to potential retention in glove-boxes. Various routes are considered: from the most traditional way with the uniaxial pelletizing to more innovative “wet” routes such as casting or extrusion from liquid suspension or plastic dough. The “wet routes” have the advantages to suppress the milling step and to offer a better homogeneity

of the U/Pu oxide but will require a prepared suspension of the oxide precursors at the head-end of the fabrication process.

First promising results have already been obtained for the “liquid” fabrication route on a surrogate suspension (mixture of TiO_2/Y_2O_3 to simulate UO_2/PuO_2). Full spherical and homogenous granules were obtained in these conditions (Fig. 5) and could be used to produce dense pellets after dedicated pressing and sintering. Experiments are now planned in the next months on U, Pu samples in ATALANTE facility to validate the concept feasibility.

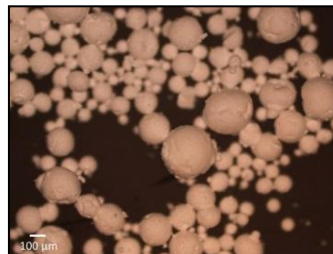


Fig. 5: MEB image of surrogate TiO_2/Y_2O_3 granules prepared by a new “liquid” fabrication route

II.D. Waste management

FR MOX spent fuels have several characteristics that will impact the waste management and thus strongly influence the technical solutions to implement for the back-end treatment of the future Generation IV reprocessing plants.

- First, production of intermediate-level waste will increase between LWR UOX/MOX and FR MOX spent fuels reprocessing due to a higher quantity of stainless steel pieces in FR MOX subassemblies.
- Then, a larger quantity of metallic fission products such as platinum group metals (Ru, Rh, Tc, Pd, Mo,...) will be present in the solid residues recovered after dissolution and clarification. Because these metallic elements are responsible of partial sedimentation in the matrix glasses, their incorporation in the glass, as in the current process, could involve difficulties during the vitrification step.
- Finally, the higher Pu content (up to a factor 20 compare to UOX fuels) will require a much better recovery of Pu from the cladding and the solid residues in order to limit the losses of Pu in the ultimate waste as low as what is currently done at La Hague plant with UOX fuels.

To tackle these issues, a melting process has been proposed and is currently under development at CEA for the conditioning of stainless steel cladding and FP solid residues. This process has the following advantages:

- Better homogeneity and higher density of the metallic waste compare to the compacting process currently used at La Hague plant for LWR fuels ;

- Possibility to incorporate the solid residues with the cladding in the melting process, avoiding any vitrification issues ;
- Recovery of residual plutonium from fuel cladding and undissolved particles after decontamination of the cladding by addition of a dedicated oxide slag in the melting process for oxide recovery.

Based on previous lab-scale experiments, a decontamination factor of alpha-emitter up to 2000 was obtained through the use of a dedicated slag [19]. Experiments are now in progress at laboratory-scale on FR representative waste (stain-less steel with metallic FP residues and cerium as Pu surrogate) to support the previous experiments and confirm the potential advantages of this melting process. The incorporation of solid residues in the melt and the selective recovery of cerium in the slag will be particularly studied before validating the concept on genuine irradiated cladding in presence of Pu.

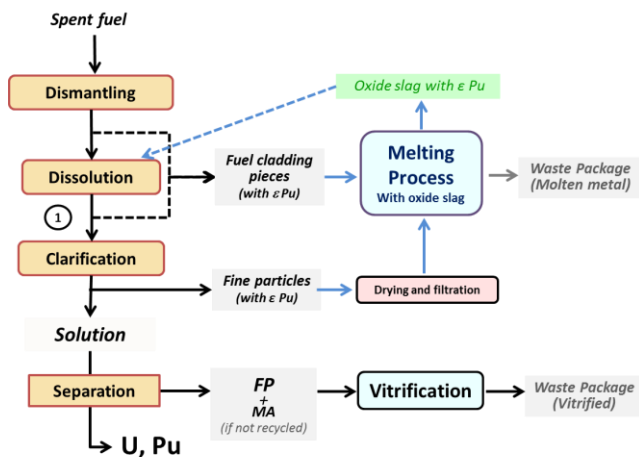


Figure 6: Example of an optimized flowsheet for FR MOX waste management

Thanks to a common management of FP solid residues with stainless steel cladding, the vitrification step could be optimized, developing new glass matrices, able to incorporate higher quantities of fission products and / or a better incorporation flexibility of problematic waste. Glasses such as new peraluminous formulations for example are currently under study at CEA [20-21].

Efforts are also done on the trapping and conditioning of volatile radionuclides in order to minimize gaseous and effluent releases at the future treatment facilities and to develop suitable matrices to immobilize them safely and without added impact. Adsorption of ⁸⁵Kr on zeolite and conditioning of iodine (¹²⁹I) in different matrices (hydroxyapatite [22] or silver phosphate glasses [23]) are studied at laboratory scale in order to propose new conditioning processes for future Generation IV reprocessing plants.

II.E. Process Monitoring and Control

The development of more efficient, compact and fast processes, able to manage increasing Pu flows, reinforce the need to develop quasi on-line process monitoring to guarantee the best control and safety of the process. The main objective is to improve the connection between on-line measurements and simulation codes in order to increase reactivity and provide a rapid and automatic response to a process drift or disturbance.

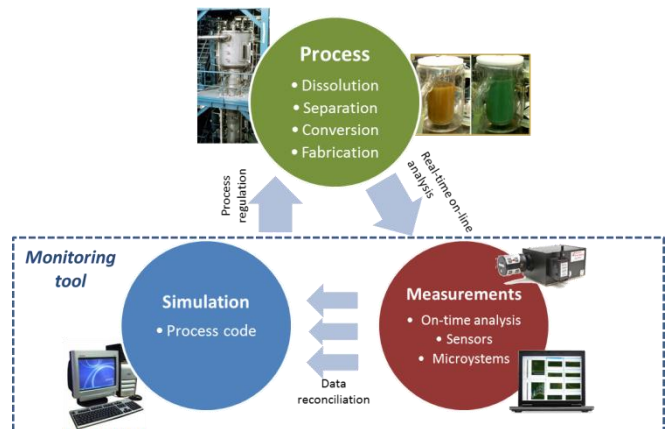


Figure 7: Advanced Process Monitoring and Control system based on a simulation code, real-time measurements and data reconciliation

If the final goal is to apply this approach to each step of the process, tools are already available for the monitoring of the separation process. The feasibility of this approach has been recently assessed at the laboratory scale during a solvent extraction test in mixer-settlers. A specific monitoring tool integrating the real-time on-line analysis of uranium concentration by UV-visible probes and a transfer function calculated by the PAREX simulation code was conceived and used to correct a voluntary malfunction during the continuous test. After a disturbance of the organic flow rate, the feed flow rate was successfully automatically monitored to stabilize the process (Fig. 8).

Microsensors should also be developed to broadening the range of real-time measurements of the key elements in the process ([U], [Pu], [H⁺],...) with an additional objective to reduce the volumes of the sample required for analysis. Microsensors combining microfluidics and detection by visible absorptiometry in a single chip [24] is for instance under development and promising results have been recently obtained on Pu(VI) measurements.

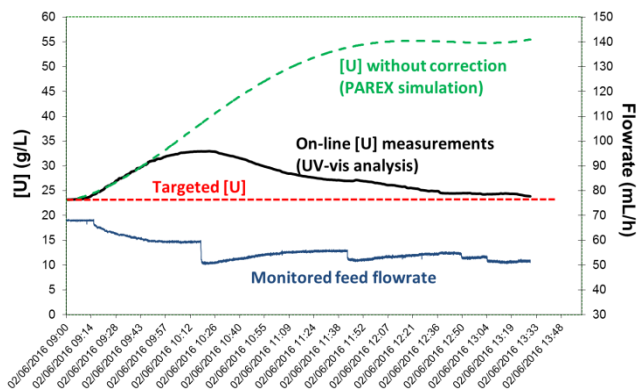


Figure 8: Illustration of the automatic correction of the uranium concentration by the monitoring tool after a process maloperation

III. CONCLUSIONS

An ambitious R&D program has been launched at CEA to develop efficient and innovative processes for the multi-recycling of uranium and plutonium from MOX fuels. This program aims to support the progressive full closure of the nuclear fuel cycle involving that the processes will have to be flexible and adapted to different kind of fuels (LWR UOX, LWR MOX and FR MOX) in order to manage the transition periods during the progressive deployment of fast reactors in the fleet.

The specificities of FR fuels compared to LWR fuels and the increase of Pu content in the fuel require some optimizations and innovations of the existing processes. Based on the experience acquired on LWR and FR fuels recycling, and on new innovative developments arising from upstream R&D, technical solutions have been proposed for each of the main steps of the future Generation IV treatment/recycling unit. The main innovations are listed below :

- Oxide/cladding separation to optimize head-end operations, improve the dissolver compactness and accelerate dissolution kinetics;
- “1 single cycle process without redox” for uranium and plutonium separation by solvent extraction with N,N-dialkylamides molecules ;
- U(VI) and Pu(IV) conversion into mixed oxides without redox control by selective precipitation or direct denitration with organic additives ;
- MOX fuel fabrication using “wet routes” avoiding powder milling ;
- Melting process for the conditioning of fuel cladding pieces and solid residues in addition with a dedicated slag for Pu recovery and decontamination ;
- Direct connection between real-time measurements and the simulation code to better monitor and control the process and development

of regulation algorithms to manage non-linear and time-delay disturbances.

First promising results have been obtained on these different steps and growing studies are now under progress at laboratory scale to validate the technical option selected for each step. Based on these results, a general system analysis will be made by an integration of the different processes in a future plant in order to check the links between the different steps, to confirm the benefits of each options and to be sure that all the selected options can be integrated together in the same flowsheet.



ACKNOWLEDGMENTS

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NOMENCLATURE

- LWR: Light Water Reactor.
- FR: Fast Reactor
- UOx: Uranium Oxide fuels.
- MOx: Mixed uranium-plutonium Oxide fuels
- FP: Fission Products
- MA: Minor Actinides
- TCP: Traitement des Combustibles Particuliers or Flexible Recycling Unit for special fuels
- PUREX: Plutonium Uranium Refining by EXtraction
- TBP: Tri Butyl Phosphate
- DEHBA: N,N-di-2-ethylhexyl-butanamide
- DEHiBA: N,N-di-2-ethylhexyl-isobutanamide
- TPH: Hydrogenated Tetrapropylene diluant

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