GENIORS

Research and Innovation Action (RIA)

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 755171.

Start date : 2017-06-01 Duration : 48 Months
http://geniors.eu/

WPASR 1

Authors : Mr. Bastien DUPLANTIER (LGI)

GENIORS - Contract Number: 755171
GEN IV Integrated Oxide fuels recycling strategies Roger Garbil

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Author(s)	Mr. Bastien DUPLANTIER
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Summary

This is the WPs activity summary report of the 1st six months of the project.

Approval

Date	By
2018-03-05 07:31:50	Mr. Stéphane BOURG (CEA)
2018-03-05 07:31:58	Mr. Stéphane BOURG (CEA)
2018-03-05 07:40:10	Mr. Stéphane BOURG (CEA)

Content

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DOMAIN1 - WP1

DOMAIN	1	WP	1	WP Leader	G. Modolo
Manpower dedicated to this WP on the period				1	

Contribution to Deliverables (number and title of each deliverable)

D1.1 : Report on the speciation and extraction of key fission products [due month 36]

Contribution to Milestones (number and title of each milestone)

MS1 : provide information on WP activity for the Work-Package Activity Summary Report 1 (month 7)

Main achievements - Progress

Task 11 Problematic fission products - extraction chemistry

Distribution data for the extraction of Sr(II) into TODGA solvents (containing 1-octanol or TBP as modifier) were determined as a function of HNO3 and TODGA concentrations

An attempt at establishing an equilibrium model for Sr(II) extraction into TODGA was made

Main difficulties - Delays

Partners CEA, ICHTJ, Juelich, KIT, Twente and UREAD are involved within WP 1. However, only KIT reported the first work.

CEA will start this work on month 12.

Juelich is planning to initiate the work on month 11. A Ph.D. student will be hired for this WP.

Scientific publications, patents (beneficiary name and type of publication)

Journal:
Int. Conference Oral:
Int. Conference Poster:
Proceedings:
Patent:

Effective collaboration between Beneficiaries (put crosses in boxes)

	CEA	CHALMERS	CIEMAT	CNRS	CTU	ICHTJ	IIC	IRSN	JRC-ITU	JUELICH	KIT	LGI	NNL	POLIMI	SCK-CEN	TWENTE	UEDIN	UNIMAN	UNIPR	UNIVLEEDS	UREAD	ULANC	EDF	AREVA
CEA																								
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INTRODUCTION

WP1 aims at investigating the solution and extraction chemistry of key fission products. This research is important to improve the actinide/fission product separation and then to further optimize the separation processes. It includes extraction chemistry of problematic fission products (Task 1.1) and the optimization of scrubbing steps during co-extraction (Task 1.2).

TASK 1.1

MANPOWER

1 pm was realized this semester by KIT. It is only 2.5 % of effort which is planned for the complete project (40.2 pm).

MAIN PROGRESSES

Extraction of Sr(II) into TODGA

Distribution data for the extraction of Sr(II) into TODGA solvents (containing 1-octanol or TBP as modifier) were determined as a function of HNO3 and TODGA concentrations. An attempt at establishing an equilibrium model for Sr(II) extraction into TODGA was made.

DIFFICULTIES

none

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ACTION PLAN

TASK 1.2

No action for this semester

LIST OF PUBLICATIONS

No publication so far

CONCLUSIONS

Since the start of the GENIORS project only KIT performed R&D related to the fission product chemistry. It is expected that other partners involved will smoothly initiate their planned work.

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DOMAIN 1 – WP2

DOMAIN	1	WP	2	WP Leader	Hitos Galán
Manpower dedicated to this WP on the period				10.37	

Contribution to Deliverables (number and title of each deliverable)

D2.1 Stability and safety studies of hold extraction systems (1 for each CyMe4BTBP, HidroBTBP, TODGA & PTD) (due m39, IIC).
D2.2 Stability studies of stripping agents (due m24, UNIPR).
D2.3 Destruction of organic (due m30, CEA).
D2.4 Impacts on process safety of gas generation (due m32, POLIMI).
D2.5 Molecular modelling of particular degradation mechanism of extracting and complexing agents (due m36, CTU).

Contribution to Milestones (number and title of each milestone)

MS1: provide information on WP activity for the Work-Package Activity Summary Report 1. CEA (due m7)

Main achievements - Progress

Task 2.1 Radiolysis & degradation products (9.17 mp)

- A new fluorinated diluent – BK-1, a carbonate of fluorinated alcohol (bis(2,2,3,3-tetrafluoropropyl) carbonate), has been tested by CTU (2 pm). Am/Eu extraction from HNO_3 solutions by $CyMe_4$-BTBP or $CyMe_4$-BTPhen in BK-1.
- $CyMe_4$BTBP degradation products in samples irradiated in FS-13, GANEX solvent and octanol were studied by HPLC and MS in detail by IIC (2.37 pm). Semi-preparative conditions by HPLC have been set up and peaks corresponding to degradation compounds (identified as hydroxyderivatives of BTBP) have been isolated.
- Study of the behaviour CDTA degraded samples has been started by CIEMAT (0.8 pm). After 1 MGy samples are completely degraded. After 200 -500 kGy, a white precipitate was formed in samples irradiated in presence of 10% of a simulated HAR solution. Influence on An/Ln/FP co-extraction by TODGA/DMDOEHMA system is being studied.
- Degradation chemistry study of hydrophilic DGAs has been carried out by Julich (3 pm) with very interesting results about degradation pathways. This work has been carried out in collaboration with Bruce Mincher (INL, USA) and Steve Mezyk (California State University Long Beach, USA).
- Review of radiolytic results on PyTri-Diol (PTD) already obtained has been performed to continue its stability studies (POLIMI, 1 pm).

Task 2.2 Destruction of organics

No activities.

Task 2.3 Gas generation (0.7 pm)

Revision of past work (SACSESS) and literature about degradation has been carried out by NLL (0.2 pm) and POLIMI (0.75 pm) in order to define experimental conditions protocols as well as identified gaps about gas generation due to radiolysis.

Task 2.4 Radiolysis modelling (0.5 mp)

CTU (0.5 pm) has started the studies of radiolysis modelling by initial simulations of $CyMe_4$-BTBP. Comparison of the energy of formation of the optimized structures revealed the possible role of intra-molecular H-bond. Further deeper analysis of the proposed products by means of stability indicators is in progress.

Main difficulties - Delays

No major difficulties have been reported. Although there was some delays due to:

- TODGA *in situ* alpha radiolysis and degradation study will start next semester (CEA).
- CIEMAT reported no availability of ^{60}Co-sources at Náyade facility for all irradiation experiments.
- IIC will need larger irradiated samples in FS-13 from partners to perform isolation of degradation products.

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Scientific publications, patents (beneficiary name and type of publication)
Journal: 1 accepted (work performed during SACSESS project). Int. Conference Oral: 2 Int. Conference Poster: Proceedings: Patent: 1 DOE report

Effective collaboration between Beneficiaries (put crosses in boxes)

	CEA	CHALMERS	CIEMAT	CNRS	CTU	ICHTJ	IIC	IRSN	JRC-ITU	JUELICH	KIT	LGI	NNL	POLIMI	SCK-CEN	TWENTE	UEDIN	UNIMAN	UNIPR	UNIVLEEDS	UREAD	ULANC	EDF	AREVA
CEA																								
CHALMERS																								
CIEMAT											■		■											
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Julich: INL and USA and California State University Long Beach, USA.

INTRODUCTION

The general objective of WP2 is to ensure a safe long-term performance of a chemical system submitted to radiation. To improve the resistance of the ligands as well as of the systems where they are involved will be the main goal. Solvent degradation may lead to many undesirable effects, for that, the identification of loses of efficiency, the behaviour troublesome degradation products or any mal operation situation due to degradation will be

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the key issues. Work is divided in four task: T21 Radiolysis & degradation products; T22 Destruction of organics; T23 Gas generation and T24 Radiolysis modelling.

TASK 2.1 RADIOLYSIS & DEGRADATION PRODUCTS (TASK LEADER: JULICH)

MANPOWER

9.17 mp realized this semester (2 pm CTU, 2.37 pm IIC, 0.8 pm CIEMAT, 3 pm Julich, 1 pm POLIMI).

MAIN PROGRESSES

New fluorinated diluent called BK-1 (bis(2,2,3,3-tetrafluoropropyl) carbonate), a carbonate of fluorinated alcohol) has been tested by CTU. Diluent does not solve the problem of limited solubility of the $CyMe_4$-BTBP and $CyMe_4$-BTPhen extractants. However, even at such a low concentration good extraction of Am(III) and high Am/Eu separation factors were achieved at higher HNO_3 concentrations.

IIC has continued with the analysis of samples of $CyMe_4$-BTBP irradiated during extension of previous Project. Degradation products formed in samples irradiated in FS-13, GANEX solvent ($CyMe_4$-BTBP in FS-13 + TBP) and octanol were studied by HPLC and MS in more details, since the composition depends on samples were irradiated in neat solvent, in contact with HNO_3 or in GANEX solvent. Evidences from HPLC and MS were obtained about two main degradation products formed in GANEX solvent, probably mono- and dihydroxy derivatives. Peaks from chromatograms were isolated and confirmed by MS analysis. Semi-quantitative measurement of concentrations of both degradation compounds could be performed as well. More detailed analysis of series F ($CyMe_4$-BTBP in FS-13) and J ($CyMe_4$-BTBP in FS-13 in absence and presence of aqueous phase) seem to indicate divergent degradation pathways, at least partly.

CIEMAT has continued with basics stability studies of the aqueous phases containing CDTA or the stripping agent SO_3PhBTP. Despite of these aqueous phases will not be recycled, the effects and interaction between the corresponding phases must be checked, since this could affect to the behaviour of the organic phase along the process. CDTA aqueous phases have been irradiated up to 200, 500 and 1000 kGy in presence and absence of simulate a HAR solution. White suspension or precipitates were found just after 200 kGy in presence of HAR solution. Assessment of An/Ln/FP co-extraction by TODGA and DMDOEHMA system will be addressed next semester. Moreover, the composition of organic phases will be studied.

Julich, in collaboration with Bruce Mincher (INL, USA) and Steve Mezyk (California State University Long Beach, USA) has carried out the radiolysis study of hydrophilic diglycolamides TMDGA, TEDGA, Me-TEDGA and Me_2-TEDGA. The radiolytic decrease (in pure water) in all the hydrophilic DGAs examined was exponential with respect to absorbed dose (0-150 kGy). The degradation rates decreased with the increasing molecular weight, following the trend TMDGA > TEDGA > Me-TEDGA > Me_2-TEDGA, and in general calculated dose constants are similar and higher than the values reported previously for the lipophilic DGAs. Analogous

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degradation products were identified for the radiolysis of all hydrophilic DGAs examined, and they are partly consistent with the products found for the irradiation of lipophilic DGAs. However, interesting differences have been observed due to irradiation have been carried out in water, as for example the hydroxylation of DGA or DCs; as well as due to the structure of DGA, particularly for TMDGA allowing to understand better degradation pathways because of the cleavage of O_{ether}-C_{ether} bond.

POLIMI focused the attention on the previously obtained radiolytic results on PyTri-Diol (PTD) solutions, in order to plan new irradiation campaigns aiming at identifying PTD degradation compounds and their impact on extracting properties. According to the systematic review, new gamma irradiation experiments have been planned (dose rate of 2.5 $kGy.h^{-1}$ up to an absorbed dose of 500 kGy) on 0.2-0.5 M PTD solutions in 0.44 M. The irradiated solution will pass through a preparative column in order to separate PTD and recover the fraction containing the mix of degradation products for HPLC-ESI-MS and NMR analyses.

DIFFICULTIES

No major difficulties have been reported. Although there will be some delays due to:

- CIEMAT reported delay due to no availability of ^{60}Co-sources at Náyade facility for all irradiation experiments.
- IIC will need larger irradiated samples in FS-13 from partners to perform isolation of degradation products during next semesters.

ACTION PLAN

Studies about in situ alpha radiolysis of TODGA due start on month 6 (CEA).

Study about behaviour of irradiated CDTA samples will be completed in semester 2 (CIEMAT). Testing of extraction properties of radiolysis products that are being synthesized at University of Reading will start by CTU. Preliminary testing of the BK-1 diluent extracts will be finalized.

New gamma irradiation experiments have been planned by POLIMI on 0.2-0.5 M PTD solutions in 0.44M HNO_3, according to the systematic review of the work already done. Where, they will try to separate the main radiolytic by-products in order to confirm the hypothesized degradation products.

Currently, it is being discussed the possibility to perform an irradiation loop to study the viability of PTD (POLIMI, CEA and INL), and compare with previous results obtained for Hydro-BTP in INL (Idaho National Laboratory). This work is related to WP2 and WP5.

TASK 2.2 DESTRUCTION OF ORGANIC (TASK LEADER: CEA)

No activities

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MANPOWER

No activities.

DIFFICULTIES

No reported.

ACTION PLAN

No action plan.

TASK 2.3 GAS GENERATION (TASK LEADER: POLIMI)

MANPOWER

0.7 mp realized this semestre (NNL, POLIMI).

MAIN PROGRESSES

During this first semester NLL has been focused on reviewing past work (SACSESS) and discussions with other partners to define common protocols and key gaps for experimental studies of gas generation.

POLIMI has started the study of gas generation in irradiated PTD solutions also by a literature review on the topic, mainly focused on defining experimental conditions that enable to fruitfully compare data with existing literature.

DIFFICULTIES

No reported.

ACTION PLAN

Gas generation experiment planned by NNL will start in semester 2.
Experimental Gas generation studies on irradiated PTD solutions will begin in semester 2.

TASK 2.4 RADIOLYSIS MODELLING (TASK LEADER: CTU)

MANPOWER

0.5 mp realized this semestre (CTU).

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MAIN PROGRESSES

During the last six month CTU has started the studies of Radiolysis modelling by initial simulations of $CyMe_4$-BTBP. Equilibrium conformations and adducts of CyMe4-BTBP were identified and modeled by MD and DFT methods. Application of MD conformation scan resulted in finding energetically more stable structures. Comparison of the energy of formation of the optimized structures revealed the possible role of intra-molecular H-bond. Further deeper analysis of the proposed products by means of stability indicators is in progress.

DIFFICULTIES

No reported.

ACTION PLAN

Further theoretical DFT analysis of the obtained degradation products of CyMe4BTBP will be done by CTU.

LIST OF PUBLICATIONS

Publications :

1. "Behaviour of the extractant Me-TODGA upon gamma irradiation: quantification of degradation compounds and individual influences on complexation and extraction." V. Hubscher-Bruder V. Mogilireddy, S. Michel, A. Leoncini, J. Huskens, W. Verboom, H. Galán, A. Núñez, J. Cobos, G. Modolo, A. Wilden, H. Schmidt,M.-C. Charbonnel, P. Guilbaud and N. Boubals. New J. Chem., 2017, 41, 13700-13711. (Studies performed within the SACSESS project / writing an submission performed within the GENIORS project).

Oral communications:

1. "Influence of gamma radiation on extraction systems based on diglycolamides and water soluble SO_3-Ph-BTP such the Euro-GANEX process." H. Galán, A. Núñez, I. Sánchez, D. Munzel, U. Müllich, J. Cobos and A. Geist ISEC 2017, Miyazaki, Japan 5-10 November 2017.
2. "Estudio de la influencia de la radiación en sistemas de extracción de ciclos avanzados del combustible nuclear". H. Galán, A. Núñez, I. Sánchez, J. Cobos. Spanish Nuclear Society 2017, SNE2017, 4-6 October 2017, Málaga, Spain.

DOE Report

CONCLUSIONS

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Since GENIORS project is just starting it can be reported a good beginning of WP2 activities (10.37 pm reported / 13.4 pm estimated average), particularly in Task1, where 9.17 pm were reported. Activities in Task 3 and 4 already have started, however activities related to Task 2 will start during second semester

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DOMAIN 1 – WP3

DOMAIN	1	WP	3	WP Leader	P. Guilbaud
Manpower dedicated to this WP on the period				15.4	
Contribution to Deliverables (number and title of each deliverable)					
D3.1 : Status on the PyTri-Diol properties [due m36] *D3.2 : Status on Distribution data and chemical modelling [due m40]* *D3.3 : Status on TODGA organic phase loading [due m36]* *D3.4 : Status on solvent clean-up & recycling [due m40]*					
Contribution to Milestones (number and title of each milestone)					
MS1 : provide information on WP activity for the Work-Package Activity Summary Report 1					
Main achievements - Progress					
Task 3.1 Optimisation of systems Studies on hydrophilic complexing agents such as SO_3-Ph-BTP or Pi-Tri-Diol (PTD) have been performed, in the extension of what has already been done in the former SACSESS project. Task 3.2 Distribution data and chemical modelling TRLFS studies have been done in order to characterize the stoichiometries of the $M(NO_3)_3(HNO_3)_m(DMDOHEMA)_n$ complexes both in monophasic and biphasic solutions. Task 3.3 Physico-chemical & loading TODGA organic phases loading studies have been taking into account the degradation products effect. Task 3.4 Solvent clean-up & recycle A review of past work (SACSESS) and discussions with other partners have been initiated in order to organize the experimental studies.					
Main difficulties - Delays					

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Task 3.3 Physico-chemical & loading

Some TODGA degradation compounds are not available up to now, which make their studies difficult to carry on.

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Scientific publications, patents (beneficiary name and type of publication)
Journal: 1 Int. Conference Oral: 2 Int. Conference Poster: 0 Proceedings: 0 Patent:0 The corresponding list is given at the end of this document.

Effective collaboration between Beneficiaries (put crosses in boxes)

	CEA	CHALMERS	CIEMAT	CNRS	CTU	ICHTJ	IIC	IRSN	JRC-ITU	JUELICH	KIT	LGI	NNL	POLIMI	SCK-CEN	TWENTE	UEDIN	UNIMAN	UNIPR	UNIVLEEDS	UREAD	ULANC	EDF	AREVA
CEA	■					■								■		■			■					
CHALMERS																								
CIEMAT			■								■		■											
CNRS				■																				
CTU																								
ICHTJ	■					■										■								
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JRC-ITU																								
JUELICH										■														
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EDF																								
AREVA																								

INTRODUCTION

The aim of this work-package is to improve the understanding and optimization of the reference chemical systems for advanced solvent extraction separation processes: grouped separation of the actinides (EURO-GANEX), separation of the minor actinides (i-SANEX), or separation of americium only (EURO-EXAM). It includes (i) the understanding of extraction chemistry to support concept process flowsheets (Tasks 3.1 & 3.3), (ii) the acquisition of extraction data to support the conception of process flowsheets (Task 3.2), and (iii) the identification of process options for clean-up of solvents to allow recycle on plant (Task 3.4).

TASK 3.1 OPTIMISATION OF SYSTEMS (TASK LEADER: POLIMI)

MANPOWER

7.5 pm realized this semester (ICHTJ, POLIMI, UNIPR).

7.5 pm realized since the beginning of the project, corresponding to 9.6 % of the 78 pm planned for the complete project.

MAIN PROGRESSES

Hydrophilic complexation:

1) BTP based complexing agents

Tests of Am(III) and Eu(III) stripping by a hydrophilic SO_3-Ph-BTP^{4-} ligand from the organic phase containing a nonadentate extractant T-DGA allow us to reject the hyphothesis on the formation of extractable heteroleptic complexes of Am(III) with TODGA and SO_3-Ph-BTP in the two-phase systems studied.

2) Pi-Tri-Diol (PTD)

A new batch of Pi-Tri-Diol (PTD) was produced by UNIPR to feed the studies. A sample was sent to POLIMI that used it, as a continuation of the complexation studies previously performed on PTD solutions within the SACSESS project, to perform new complexation titration experiments with Nd(III) by UV-Vis spectrophotometry, in order to further confirm PTD selectivity for Actinides towards Lanthanides.

DIFFICULTIES

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ACTION PLAN

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TASK 3.2 DISTRIBUTION DATA AND CHEMICAL MODELLING (TASK LEADER: KIT)

MANPOWER

4 pm realized (KIT)

4 pm realized since the beginning of the project, corresponding to 29.4 % of the 13.6 pm planned for the complete project.

MAIN PROGRESSES

Both monophasic and biphasic speciation studies in malonamide systems were performed usinf TRLFS to help identifying the different $M(NO_3)_3(HNO_3)_m(DMDOHEMA)_n$ complexes (M = Eu(III) or Cm(III)) postulated from solvent extraction experiments performed in the EUROPART project.

DIFFICULTIES

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ACTION PLAN

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TASK 3.3 PHYSICO-CHEMICAL & LOADING (TASK LEADER: CIEMAT)

MANPOWER

3.5 pm realized (CIEMAT, KIT)

3.5 pm realized since the beginning of the project, corresponding to 10.3 % of the 34 pm planned for the complete project.

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MAIN PROGRESSES

The loading of TODGA organic phases studies have been started:

1) A TODGA solvent (0.1 mol/L TODGA + 5% octanol in TPH) was loaded with La(III), Nd(III) or Yb(III) ;
2) The accumulation of the main degradation compounds (diglycolamic acid, V (DODGAA); Oct2NH, VII (DOA)) has been studied; and
3) The Influence of detrimental DCs (diglycolamic acid, V (DODGAA); Oct_2NH, VII (DOA)) on the extraction at high nitric acid concentration (4 mol/L HNO_3) and the back-extraction of Ln/FP of TODGA systems has been assessed.

DIFFICULTIES

No availability of some degradation compounds (DCs) to continue their studies.

ACTION PLAN

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TASK 3.4 SOLVENT CLEAN-UP & RECYCLE (TASK LEADER: NNL)

MANPOWER

0.4 pm realized (NNL)

0.4 pm realized since the beginning of the project, corresponding to 1.9 % of the 21.5 pm planned for the complete project.

MAIN PROGRESSES

Initial planning and preparation of paperwork for experimental studies in NNL is ongoing. A review of past work (SACSESS) and discussions with other partners is ongoing in order to define common protocols and key gaps for experimental studies. Specifically, interactions with WP23 are being considered to maximise results across both WPs from solvent radiolysis experiments.

DIFFICULTIES

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ACTION PLAN

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LIST OF PUBLICATIONS

Publications :

2. "Behaviour of the extractant Me-TODGA upon gamma irradiation: quantification of degradation compounds and individual influences on complexation and extraction." V. Hubscher-Bruder V. Mogilireddy, S. Michel, A. Leoncini, J. Huskens, W. Verboom, H. Galán, A. Núñez, J. Cobos, G. Modolo, A. Wilden, H. Schmidt,M.-C. Charbonnel, P. Guilbaud and N. Boubals. New J. Chem., 2017, 41, 13700-13711. (studies performed within the SACSESS project / writing an submission performed within the GENIORS project).

Oral communications:

3. "Influence of gamma radiation on extraction systems based on diglycolamides and water soluble SO_3-Ph-BTP such the Euro-GANEX process." H. Galán, A. Núñez, I. Sánchez, D. Munzel, U. Müllich, J. Cobos and A. Geist ISEC 2017, Miyazaki, Japan 5-10 November 2017.
4. "Estudio de la influencia de la radiación en sistemas de extracción de ciclos avanzados del combustible nuclear". H. Galán, A. Núñez, I. Sánchez, J. Cobos. Spanish Nuclear Society 2017, SNE2017, 4-6 October 2017, Málaga, Spain.

CONCLUSIONS

The GENIORS project is just starting and most of the studies were planned to begin after month 6 in this WP3. However, 15.4 pm were realized in WP3 during the first semester, corresponding to 10.5 % of the 147.1 pm planned for the complete project.

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

DOMAIN 1 – WP4

DOMAIN	1	WP	4	WP Leader	CNRS
Manpower dedicated to this WP on the period					
Contribution to Deliverables (number and title of each deliverable)					
No deliverable during the period. 4 deliverables in WP4: D4.1 Understanding the evolution of an interface during dissolution of actinide oxide materials by macro/microscopic approaches combination [**month 40**] – **CNRS** D4.2 Innovative dissolution routes for highly plutonium doped (U,Pu)O_2 and (U,Pu,MA)O_2 samples [**month 45**] – **CEA** D4.3 Studies of non-powder routes for the synthesis of MOX fuels materials, potentially bearing minor actinides and blanket fuel materials [**month 36**] – **SCK.CEN** D4.4 Impact of the precursor “history” (nature, structural, microstructural and morphological parameters) during the conversion and sintering to actinide based dioxide materials [**month 45**] – **CNRS**					
Contribution to Milestones (number and title of each milestone)					
MS1 : provide information on WP activity for the Work-Package Activity summary Report 1					
Main achievements - Progress					

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T41 Study of the solid/liquid interfaces during dissolution [1-48] – Task Leader : CNRS

Multiparametric study of the dissolution of $(U,Ce)O_2$ and $(U,Ln)O_{2-x}$ solid solutions – CNRS/ICSM

In order to develop the multi-parametric dissolution tests, the preparation then characterization of a large defined panel of uranium-lanthanide solid solutions has begun in month 6.

Innovative dissolution routes for highly plutonium doped $(U,Pu)O_2$ and $(U,Pu,MA)O_2$ samples – CEA

No work done during this semester : a PhD started in Nov 2017.

T42 Study of the solid/liquid interface during conversion [1-48] – Task leader : SCK.CEN

Studies of non-powder routes for the synthesis of MOX fuels materials, potentially bearing minor actinides and blanket fuel materials – SCK.CEN

The sol-gel process via internal gelation was successfully applied to fabricate particles containing cerium (Ce) and uranium (U). Compositions with Ce contents of 5%, 10%, 15%, 20%, 25% and 30% were prepared using an acid deficient uranyl nitrate solution and cerium (III) nitrate solution as precursor. Moreover, pure undoped uranium reference particles were fabricated applying the same technique and parameters.

Hydrothermal precipitation of uranium oxides for simplified fuel fabrication – CNRS/ICSM

The direct precipitation of uranium oxides was undertaken under hydrothermal conditions through the *in situ* conversion of uranium oxalate above 180°C. SEM observations of the resulting samples revealed the loss of the classical square platelet shape of the oxalate during the treatment, while the residual carbon contents were significantly lowered compared to that usually obtained through thermal conversion.

Innovative precursors for morphology-controlled UO_2 and $(U,Ln)O_2$ oxides – CNRS/ICSM

Monodisperse microspheres of $UO_2 . nH_2O$ were obtained through hydrolysis of uranium(IV) in the presence of asparagic acid under hydrothermal conditions at 160°C. Their size was monitored through operating parameters (time, temperature, reactant concentrations, …). Moreover, their morphology was maintained throughout thermal treatment up to 800°C which allowed to ensure the preparation of UO_2 with controlled morphology.

Conversion of uranium nitrate into uranium oxide by Solution Combustion Synthesis – CNRS/ICSM

Actinide surrogate systems $GdNO_3$/fuel (fuel = glycine, urea, citric acid) were investigated to propose an experimental protocol for the conversion of uranium nitrate into uranium oxide by Solution Combustion Synthesis (SCS). The fuel over gadolinium nitrate ratio appeared as a critical parameter during the conversion, and may induce phase transitions of the final product. The highest temperature of conversion was obtained for a ratio of 1 leading to the formation of monoclinic form of Gd_2O_3. For higher ratios, the conversion temperature was lowered and the cubic form of Gd_2O_3 was observed. The end-products exhibited interesting characteristic for sintering (specific surface area typically of 10-20 m^2/g).

Main difficulties - Delays

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Scientific publications, patents (beneficiary name and type of publication)

Journal: No
Int. Conference Oral: No
Int. Conference Poster: No
Proceedings: No
Patent: No

Effective collaboration between Beneficiaries (put crosses in boxes)

	CEA	CHALMERS	CIEMAT	CNRS	CTU	ICHTJ	IIC	IRSN	JRC-ITU	JUELICH	KIT	LGI	NNL	POLIMI	SCK-CEN	TWENTE	UEDIN	UNIMAN	UNIPR	UNIVLEEDS	UREAD	ULANC	EDF	AREVA
CEA	X			X																				
CHALMERS																								
CIEMAT																								
CNRS	X			X											P									
CTU																								
ICHTJ																								
IIC																								
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JRC-ITU																								
JUELICH																								
KIT																								
LGI																								
NNL																								
POLIMI																								
SCK-CEN				P											X									
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UNIMAN																			X					
UNIPR																								
UNIVLEEDS																								
UREAD																								
ULANC																								
EDF																								
AREVA																								

P : some discussions will be done soon for collaborations in the field of materials elaboration

INTRODUCTION

The main objectives of the WP solid/liquid interface chemistry is to better understand the phenomena occurring at the solid/liquid interfaces during spent nuclear fuel reprocessing in order to support potential processes. It is divided in two main topics. The first is devoted to dissolution step (Task 4.1). It will be examined not only considering direct interactions between the chemical species coming from the solid and the solution, but also through the development of catalytic reactions at the interface. The second topic is focused on the conversion of

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

actinides by precipitation of original precursors coming from new chemical processes based on wet chemistry routes (Task 4.2).

TASK 4.1 STUDY OF THE SOLID/LIQUID INTERFACES DURING DISSOLUTION

MANPOWER

0.3 pm (CNRS)

MAIN PROGRESSES

Multiparametric study of the dissolution of $(U,Ce)O_2$ and $(U,Ln)O_{2-x}$ solid solutions – CNRS/ICSM

In order to develop the multi-parametric dissolution tests, the preparation then characterization of a large defined panel of uranium-lanthanide solid solutions has begun in month 6.

Innovative dissolution routes for highly plutonium doped $(U,Pu)O_2$ and $(U,Pu,MA)O_2$ samples – CEA

No work done during this semester : a PhD started in Nov 2017.

DIFFICULTIES

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ACTION PLAN

CNRS/ICSM

The first dissolution experiments on $(U,Ce)O_2$ and $(U,Ln)O_{2-x}$ solid solutions are planned to begin during the next semester

CEA

A PhD work started in Nov. 2017 to study the Innovative dissolution routes for highly plutonium doped $(U,Pu)O_2$ and $(U,Pu,MA)O_2$ samples

TASK 4.2 STUDY OF THE SOLID/LIQUID INTERFACE DURING CONVERSION

MANPOWER

1.3 pm (CNRS/ICSM) + 2.5 pm (SCK.CEN)

MAIN PROGRESSES

Studies of non-powder routes for the synthesis of MOX fuels materials, potentially bearing minor actinides and blanket fuel materials – SCK.CEN

The sol-gel process via internal gelation was successfully applied to fabricate particles containing cerium (Ce) and uranium (U). Compositions with Ce contents of 5%, 10%, 15%, 20%, 25% and 30% were prepared using an acid deficient uranyl nitrate solution and cerium (III) nitrate solution as precursor. Moreover, pure undoped uranium reference particles were fabricated applying the same technique and parameters.

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The direct precipitation of uranium oxides was undertaken under hydrothermal conditions through the *in situ* conversion of uranium oxalate above 180°C. SEM observations of the resulting samples revealed the loss of the classical square platelet shape of the oxalate during the treatment, while the residual carbon contents were significantly lowered compared to that usually obtained through thermal conversion.

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DIFFICULTIES

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ACTION PLAN

SCK.CEN

Within the next semester, the particles will be analysed using inductively coupled plasma mass spectrometry, thermogravimetry and high-temperature X-ray powder diffraction.

Furthermore, a thermal treatment under reducing conditions is foreseen to end up with UO_2 based material. The final products will be studied with X-Ray powder diffraction and scanning electron microscopy.

CNRS/ICSM

Direct precipitation of uranium oxides under hydrothermal conditions will be extended to $(U,Ln)O_2$ mixed oxides.

The sintering capability of UO_2 microspheres will be studied through preliminary dilatometry tests and further SEM examinations.

The experimental protocol developed for the conversion of gadolinium nitrate to gadolinium oxide by Solution Combustion Synthesis will be transposed to uranium oxide powders.

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

LIST OF PUBLICATIONS

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CONCLUSIONS

Several ways of preparation of precursors are now under progress. They will allow to provide a large variety of materials, with various compositions and microstructures. This will allow to developed multiparametric dissolution experiments on the prepared samples (planed to begin during the next semester). No big difficulties encountered by the partners for both tasks : T4.1 and T4.2.

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DOMAIN 2 – WP5

DOMAIN	2	WP	21	WP Leader	C. Ekberg
Manpower dedicated to this WP on the period				10.4	
Contribution to Deliverables (number and title of each deliverable)					
D211 Experimental and modelling studies of transfer kinetics in the reference separation processes (Juelich, ULANC, CNRS) D212 Impacts of solvent degradation and recycle on the EURO-GANEX process (CIEMAT) D213 Performances on microelectrode for online monitoring of relevant parameters () D214 contribution of computational multi-disciplinary approaches to the design of solvent extraction devices (ULEEDS) D215 Status of flowsheet calculations made for WP22 process testing (Juelich, KIT)					
Contribution to Milestones (number and title of each milestone)					
Main achievements - Progress					
The on-line radioplysis studies got a leap forward by the design and adjustment for the implementation of an irradiation loop at Náyade irradiation facility at CIEMAT. This together with the MARCEL loop at CEA and the loop at INL makes it possible to compare different on-line measurements to increase reliability in the results.					
Main difficulties - Delays					

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

No major difficulties although some smaller ones as detailed in the separate task reports.

Scientific publications, patents (beneficiary name and type of publication)

Oral communication: “The EURO-GANEX process: Stability studies of GANEX system”.
ENYGF (European Nuclear Young Generation Forum), Manchester (UK) 11-16 June, 2017.

Andreas Geist, Giuseppe Modolo, Udo Müllich, Robin Taylor, Daniel Whittaker, Andreas Wilden, David Woodhead, The extraction of An(III), Ln(III) and HNO3 by a TODGA solvent: equilibrium modelling, presented at ISEC 2017, Japan.

“The Effects of Nitric Acid on Extraction Properties of TODGA During Fission Product Management”, M.A.Bromley, C.Boxall in “The Scientific Basis of Nuclear Waste Management”, N.C.Hyatt, R.Ewing, Y.Inagaki, C.Jantzen (Eds), Cambridge University Press, Cambridge UK, MRS Advances., 6 pages (2017) DOI: 10.1557/adv.2016.624

Submitted
“A Study of Cerium Extraction Kinetics by TODGA in Acidified and Non-Acidified Organic Solvent Phases in the Context of Fission Product Management”, M.A.Bromley, C.Boxall, Progress in Nuclear Science & Technology, submitted (2017).

Presentation at Actinides 2017 in Sendai, Japan during the week 10th – 14th July 2017:
“The Effects of Nitric Acid on the Extraction Properties of TODGA During Fission Product Management” M.A.Bromley, C.Boxall

Effective collaboration between Beneficiaries (put crosses in boxes)

	CEA	CHALMERS	CIEMAT	CNRS	CTU	ICHTJ	IIC	IRSN	JRC-ITU	JUELICH	KIT	LGI	NNL	POLIMI	SCK-CEN	TWENTE	UEDIN	UNIMAN	UNIPR	UNIVLEEDS	UREAD	ULANC	EDF	AREVA
CEA																								
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INTRODUCTION

It is clear that with the investment of 10.5 PM out of the about 155 PM dedicated in total the work in this WP did not really start yet. However, as seen above some important progress has been made with respect to the irradiation loop and modelling of column performance.

There is an ongoing collaboration with INL which does not show in the collaboration table even if the work is ongoing. During this period only Juelich did report on this.

UNILEEDS did wrongly report their work in Task 1 but the work belongs to Task 2 as presented here.

TASK 5.1

MANPOWER

- CIEMAT : 3 pm
- ULANC: 6 pm
- CNRS: 0 pm

MAIN PROGRESSES

- CIEMAT:
  - Designs and adjustment for the implementation of an irradiation loop at Náyade facility:
    - Reactor design development to contact phases during the irradiation inside and outside of the irradiation device.
    - Design of a new opened irradiation device to carry out dynamic experiments.
  - First studies to find the relevant conditions to simulate effects of radiation along the process: TODGA/SO_3PhBTP systems irradiated up to 200 and 500 kGy alone, two phases in contact and two phases in contact with air flow sparging (Work related to WP2 task 1).
    - Assessment of the An/Ln extraction properties of irradiated samples.

- ULANC
  - Studies have been conducted into the solvent extraction kinetics of lanthanide fission products by TODGA and the effects of solvent phase acidity on the effectiveness of this organic extractant molecule. We have observed a reduction in the rate of cerium extraction as solvent phase acidity increases, attributed to aggregation of the extractant. The effect is shown to be reversible through neutralization while a permanent prevention method is the subject of further work.
  - A new PhD student, Alex Jackson, has been recruited and began study at Lancaster in Oct 2017. He is currently undergoing an intensive 3 month training course and will being his research in earnest in Jan 2018.

DIFFICULTIES

- CIEMAT:
  - Not availability of 60Co-sources at Náyade facility to submit samples at low integrated dose.

ACTION PLAN

- CIEMAT
  - On-going activities about:
  - Building a new device for the Náyade facility.
  - Designing and testing new devices for an irradiation loop.
  - Material evaluation to radiation, organic solvents and high nitric acid concentrations to
  - Build a robust irradiation loop.
  - Relevant conditions to simulate effects of radiation along the process.
- ULANC
  - Alex Jackson to have completed training course and to have begun work on validating existing analytical model (developed during SACSESS) of the Rotating Diffusion Cell (RDC).
- CNRS
  - the kinetics of stripping of Am(III) cation will be investigated by using the rotating membrane cell (RMC) technique. The extractants will be TODGA (+5% octanol) and $CyMe_4BTBP$ (+ TODGA).The concentrations of the extractants will be varied in order to determine the optimum values and the effect of varying its concentration.

TASK 5.2

MANPOWER

- ULEEDS: 0.3 pm

MAIN PROGRESSES

- ULEEDS
  - The CFD model of the pulsed column has been expanded now to cover two distinct liquid phases, representing the aqueous and organic.
  - The column model is still over 2 plates, but has been re-built to represent a size and geometry that is closer to that expected in a real facility.

DIFFICULTIES

- ULEEDS
  - The re-mesh of the model resulted in problems of scale up around the sieve plates and delaying running of the models
  - A delay in the other modelling project on centrifugal contactors has occurred as the Post Doctoral Researcher needed to be replaced and recruitment is still ongoing

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ACTION PLAN

- ULEEDS
  - Once the 2 phase models are running in a stable manner, we will explore expanding the detail to cover more plates or investigate hold-up as a direct relation to residence times.

TASK 5.3

MANPOWER

- JUELICH: 1 pm
- NNL: 0.1 pm

MAIN PROGRESSES

- JUELICH
  - Data from single centrifugal contactor tests (AmSel system) were shared with KIT.
- NNL
  - Review of progress made under SACSESS

DIFFICULTIES

-

ACTION PLAN

- JUELICH
  - Further single centrifugal contactor tests will be conducted and data will be shared with flowsheet developers.
- NNL
  - Modeling will start in semester 2

LIST OF PUBLICATIONS

None

CONCLUSIONS

None

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

DOMAIN 2 – WP6

DOMAIN	2	WP	6	WP Leader	Robin Taylor
Manpower dedicated to this WP on the period					

Contribution to Deliverables (number and title of each deliverable)

Write here the N° (Dx.y) of the deliverables concerned by the work done in the semester. Mention when achieved.

D6.1: Assessment of applications of PTD in i-SANEX and EUROGANEX processes

- Contributions from POLIMI

D6.2: Optimized flowsheets for homogeneous recycling of advanced MOX fuel in GEN IV reactors

- Contributions from JRC

D6.3: Optimized flowsheets for heterogeneous recycling of advanced MOX fuel in GEN IV reactors

- Contributions from JUELICH, POLIMI

Contribution to Milestones (number and title of each milestone)

Write here the N° (Dx.y) of the milestones concerned by the work done in the semester. Mention when achieved.

MS1: provide information on WP activity for the Work-Package Activity Summary Report 1

- Completed via this WPASR report

MS2: provide information on WP activity for the Work-Package Activity Summary Report 2
MS3: provide information on WP activity for the Work-Package Activity Summary Report 3
MS4: provide information on WP activity for the Work-Package Activity Summary Report 4
MS5: provide information on WP activity for the Work-Package Activity Summary Report 5
MS6: provide information on WP activity for the Work-Package Activity Summary Report 6
MS7: provide information on WP activity for the Work-Package Activity Summary Report 7
MS9: EURO-GANEX trial complete and TRU nitrate product ready for conversion studies

Main achievements - Progress

JRC – Key highlight:

- A new solvent is proposed to simplify the EURO-GANEX process and published paper

JEULICH – Key highlight:

- Analysis of precipitates from a previous EURO-EXAm test using TPAEN
- Single stage contactor tests of the AmSel system

POLIMI – Key highlight:

- Discussions and planning for essential data acquisition for flowsheet modelling (TODGA-PTD system)

No progress this semester at: CEA, Chalmers, CIEMAT, ICHTJ, KIT, NNL (except presentation at ISEC 2017 conference) TWENTE, UNIPR, UREAD (assumed state as no HYPAR available for downloading)

Main difficulties - Delays

The work package concerns flowsheet development of key processes for homogeneous and heterogeneous recycling so it is expected that most studies will commence later in the project. No significant delays or causes for concern were raised by the partners in the HYPARs.

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

Scientific publications, patents (beneficiary name and type of publication)

Journal:

1. R. Malmbeck et al., J. Radioanal. Nucl. Chem, Modified diglycolamides for grouped actinide separation, 2017, in press.

Int. Conference Oral:

1. R. Taylor, S. Bourg, C. Boxall, M. Carrott, A. Geist, B. Hanson, R. Malmbeck, G. Modolo, C. Rhodes, M. Sarsfield, C. Sharrad, T. Tinsley, D. Whittaker, A. Wilden, Progress towards reference routes for recycling actinides in future nuclear fuel cycles: presented at ISEC 2017, Japan.

Int. Conference Poster: None
Proceedings: None
Patent: None

Effective collaboration between Beneficiaries (put crosses in boxes)

	CEA	CHALMERS	CIEMAT	CNRS	CTU	ICHTJ	IIC	IRSN	JRC-ITU	JUELICH	KIT	LGI	NNL	POLIMI	SCK-CEN	TWENTE	UEDIN	UNIMAN	UNIPR	UNIVLEEDS	UREAD	ULANC	EDF	AREVA
CEA																								
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JRC-ITU											x													
JUELICH	x										x													
KIT																								
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NNL											x													
POLIMI										x	x													
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INTRODUCTION

The objectives of WP6 are to optimise the efficiencies and safety of separation processes developed under the SACSESS project (i.e. i-SANEX, EURO-GANEX, EXAm, CHALMEX) The main emphasis is on process development through flowsheet testing. WP22 focuses on optimisation of the reference separation processes, particularly where significant simplification is possible or replacement of non-CHON ligands by CHON molecules can be proposed. It also includes extension of process envelopes to more challenging GenIV feeds and integration of the minor actinide SX cycles with upstream and downstream stages, particularly where interfaces may cause issues.

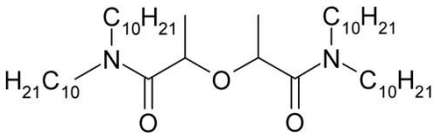
TASK 6.1: HOMOGENEOUS RECYCLING

MANPOWER

JRC was the only partner to book time against Task 1.1 and achieved 3 man-months cf. 2 expected.

MAIN PROGRESSES

A simplification of the organic formulation of the EURO-GANEX process has been tried out, replacing the two extractants with a single modified diglycolamide. Batch experiments were carried out with the new organic solvent formulation based on 0.5 M m-TDDGA in dodecane (below), investigating the extraction behaviour of transuranic elements and important FPs under high Pu loading, > 30 g/L. The new solvent system seems to have potential for application in the EURO-GANEX process, thus simplifying the organic phase chemistry, pending further studies.



DIFFICULTIES

Availability of the ligand in sufficient quantities has been highlighted as a potential problem.

ACTION PLAN

Discussions between JRC, KIT and NNL should focus on development requirements for this ligand to enable possible use in flowsheet testing later in WP6.

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

TASK 6.2: HETEROGENEOUS RECYCLING

MANPOWER

	task 2	
	expected	achieved
JRC		
JUELICH	4	4
POLIMI	0.25	0.5
total	4.25	4.5

MAIN PROGRESSES

During this semester POLIMI is discussing with colleagues from KIT and FZJ, in order to evaluate the availability of all the data needed to perform the calculations of a flow-sheet for the system PTD-TODGA (Innovative-SANEX process with PTD replacing SO3-Ph-BTP in the aqueous phase to maintain the CHON principle). The data required for the extraction and scrubbing steps are available from literature, while the data provided by POLIMI and FZJ are enough to calculate the stripping step based on PTD ligand. Data concerning the purification of the An loaded PTD phase from lanthanides are still missing, therefore focused extraction tests will be performed in the next semester.

SEM images and EDX measurements at JUELICH suggest that the solid precipitate collected from the second H_4TPAEN centrifugal contactor test (made in the SACSESS project) are composed of the complexant itself. No indication has been found that lanthanide or other complexes with the TPAEN ligand precipitated.

A single centrifugal contactor experiment was carried out using the AMSEL system, which is based on the i-SANEX process but aims to separate americium alone. The stage efficiencies in the tests were relatively low, as stage efficiencies of 25% and 35% were reached for Am(III) at the higher and lower flow-rate, respectively. The selectivity of the system was good, with Eu/Am separation factors of ca. 400 and Cm/Am separation factors of 2.5 – 2.6. As already mentioned above, the stage efficiency was lower than expected and therefore, the distribution ratios of Am and Cm were too high. Therefore, a second single centrifugal contactor test was conducted using 0.70 mol/L HNO_3 in the Am stripping solution containing SO_3-Ph-BTBP and even lower flow-rates of 40/20 and 20/10 ml/h (org./aq.). The data acquisition from the second test is not finished yet and will be reported in the next HYPAR.

DIFFICULTIES

ACTION PLAN

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

LIST OF PUBLICATIONS

1. Malmbeck, R., Magnusson, D., Geist, A., Modified diglycolamides for grouped actinide separation, (2017) Journal of Radioanalytical and Nuclear Chemistry, 314 (3), pp. 2531-2538.

2. R. Taylor, S. Bourg, C. Boxall, M. Carrott, A. Geist, B. Hanson, R. Malmbeck, G. Modolo, C. Rhodes, M. Sarsfield, C. Sharrad, T. Tinsley, D. Whittaker, A. Wilden, Progress towards reference routes for recycling actinides in future nuclear fuel cycles: presented at ISEC 2017, Japan.

CONCLUSIONS

The work package concerns flowsheet development of key processes for homogeneous and heterogeneous recycling so it is expected that most studies will commence later in the project. No significant delays or causes for concern were raised by the partners in the HYPARs.

In fact, despite the low levels of manpower dedicated to WP6 in semester 1 there has been already one significant development – the promising results obtained by JRC on the modified DGA ligand that may be a candidate to replace the TODGA+DMDOHEMA combination in the EURO-GANEX process. A journal paper is already published on this subject.

DOMAIN 2 – WP7

DOMAIN	2	WP	7	WP Leader	Leturcq (CEA)
Manpower dedicated to this WP on the period				6.2	
Contribution to Deliverables (number and title of each deliverable)					
D7.1 Report on dissolution model development [month 36] (**CEA**) D7.3 Report on the development of the different MABB precursor synthesis routes and their contribution on MA homogenity, dust reduction, lowering sintering temperature [month 45] (**CEA**) D7.4 Product quality, characterization and testing (**NNL**)					
Contribution to Milestones (number and title of each milestone)					
M7.1 “availability of model compounds” **achieved.**					
Main achievements - Progress					
<u>T71 Dissolution of model compounds</u> Synthesis of 18 ($U_{1-x}Pu_xO_2$) model compounds with Pu amount ranging from 0 to 100% and different morphologies. <u>T73 Uranium/minor actinide based oxide conversion dedicated to transmutation</u> Dense pellet fabrication of Americium-based oxides from co-converted oxalates					
Main difficulties - Delays					
None					
Scientific publications, patents (beneficiary name and type of publication)					
Journal: Int. Conference Oral: Int. Conference Poster: Proceedings: Patent:					
Effective collaboration between Beneficiaries (put crosses in boxes)					

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	CEA	CHALMERS	CIEMAT	CNRS	CTU	ICHTJ	IIC	IRSN	JRC-ITU	JUELICH	KIT	LGI	NNL	POLIMI	SCK-CEN	TWENTE	UEDIN	UNIMAN	UNIPR	UNIVLEEDS	UREAD	ULANC	EDF	AREVA
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INTRODUCTION

The objective of WP7 is to provide relevant dissolution and conversion processes linkable with the separation processes. When necessary, interfaces between dissolution/separation and separation/conversion are considered. For dissolution, the focus is put on the completion of data relevant for defining a dissolution model. The conversion issues of solutions from SX-process are the development of safe synthesis routes, the destruction of prejudicial organics from SX-process and characterization of synthesized MABB precursors. The workpackage is divided into 4 tasks: Task7.1 on dissolution, Task7.2 on destruction of organics, Task7.3 on development of safe synthesis routes and Task7.4 on characterization of MABB precursors. This semester major progresses were achieve in Task 7.1 with the synthesis of the differents $(U,Pu)O_2$ samples that will be used to obtain dissolution data to feed the definition of a dissolution model, and in Task 7.3 with the dense pellet fabrication of Americium-based oxides from co-converted oxalates.

TASK 7.1

MANPOWER

Expected effort for the period 3 men.month

Actual effort spent during the period 3 men.month

MAIN PROGRESSES

Syntheses of $(U,Pu)O_2$ model compounds

In order to be able to model the dissolution of $(U,Pu)O_2$ different single phase oxide powders were synthetized using different conversion routes, two oxalic routes, a sol/gel route and a calcination of PuO_2 colloids. Using different routes permitted to obtain oxides with the same Pu amount but exhibiting different morphologies, in order later to quantify the influence of this parameter on dissolution kinetics. Up to 18 different oxide powder batches were synthetized for a total of seven different compositions as shown in Fig1. Typical morphologies obtained using the 4 different synthesis routes are shown in Fig.2. Depending on the oxalate precipitation parameters, needle morphology or platelet morphology can be targeted for the UO_2 and PuO_2. Then, the oxalic route permits only to obtain needle morphology mixed oxides for Pu amount less than 50% explaining why all Pu-richer samples were only obtained using sol/gel routes.

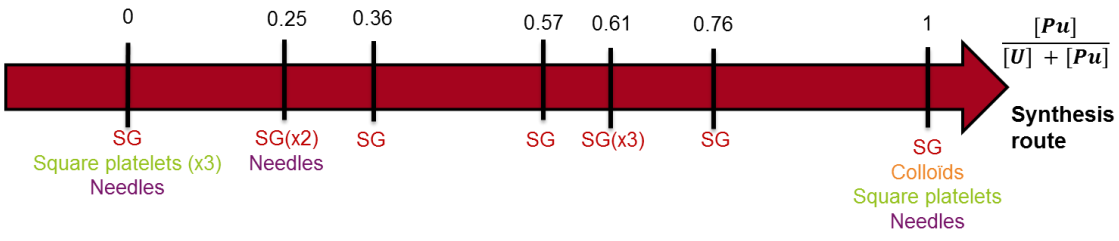

Fig.1: Model compounds synthetized for dissolution studies.

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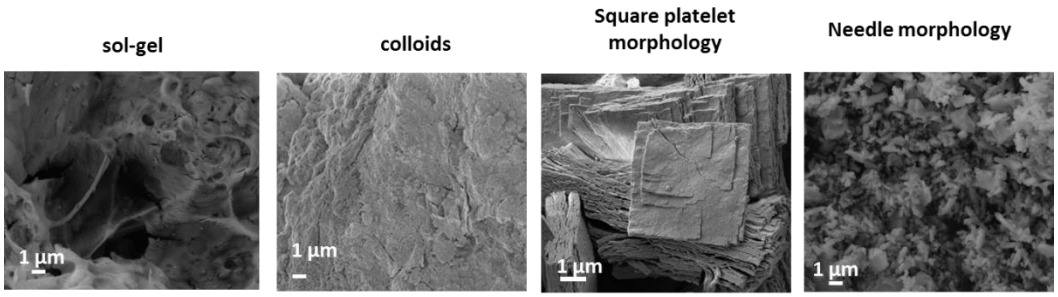

Fig.2: morphologies of (U,Pu)O_2 powders obtained using the 4 different synthesis routes.

In order to modify the microstructure different calcination temperatures or atmospheres were applied to some compositions.

In a first paragraph the different synthesis routes are exposed, and then characterizations of the 18 samples are exposed in a second paragraph.

DIFFERENT SYNTHESIS ROUTES USED

The different synthesis routes used are known to produce single phase oxides and were chosen for this reason.

The oxalic synthesis route.

The oxalic precipitation apparatus is shown in Fig3. Three reagents are needed; the first one is composed of the cations to precipitate in a nitric solution. A total actinide concentration of about 30 $g.L^{-1}$ was used. The two other solutions are composed of a mixture of oxalic and nitric acids in defined proportions. The addition of the two first reagents inside a vortex created by stirring the third reagents leads to the precipitation of a single phase actinide oxalate. Thus, after filtering and rinsing the precipitate, calcination under a controlled atmosphere converts the precipitate onto the oxide.

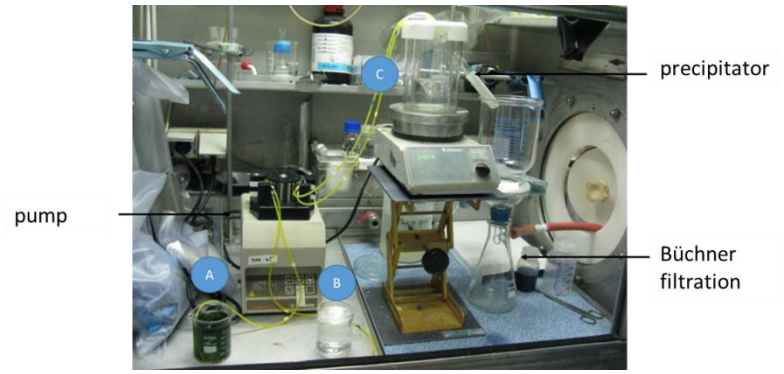

Fig 3: Oxalic precipitation apparatus (example of uranium (IV) precipitation)

Square platelet morphology

In industrial conditions, actinides (+IV) meanly precipitate as a $An^{IV}(C_2O_4)_2$, 6 H_2O P-1 triclinic oxalate with a shape of agglomerated square platelets. To form this oxalate $[HNO_3] > 2M$ and an oxalic excess of less than 0.1M have to be used. This synthesis route was used to fabricate UO_2 and PuO_2 samples.

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Needle morphology

Under different precipitation conditions ([HNO_3] < 2M, oxalic excess > 0.1M and addition of a monovalent cation NH_4^+ or $N_2H_5^+$), it is possible to precipitate $An^{IV}_2M_2^+(C_2O_4)_5,nH_2O$ (with An^{IV} = U ou Pu et M^+= H_3O^+, $N_2H_5^+$ or NH_4^+) exhibiting a hexagonal structure. Such compounds precipitate as agglomerated needles (Fig4). The addition of M^+ on top of the use of different precipitation conditions allows the precipitation of these hexagonal oxalates for the synthesis of UO_2 and PuO_2 needle samples. For the synthesis of $(U,Pu)O_2$ by oxalic route, hydrazinium ions $N_2H_5^+$ were added to reduce and stabilize plutonium at its lower oxidation state (i.e. +III). In this case; the actinide solution, composed of a mixture of U^{IV}-Pu^{III}-$N_2H_5^+$, must have a plutonium ratio $\frac{[Pu]}{[U+Pu]}$ identical to the ratio targeted in the final oxide.

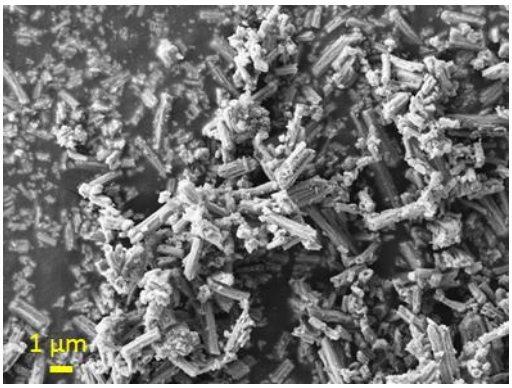

Fig.4: SEM image of $Pu^{IV}_2(NH_4)_2(C_2O_4)_5.nH_2O$ synthetized. (secondary electron mode)

The sol/gel synthesis route

The sol/gel route that consists in the formation of a gel followed by calcination under a controlled atmosphere to form the oxide is commonly used [1]. This route allows the synthesis of mixed $(U,Pu)O_2$ for all Pu contents contrarily to the oxalic route. The protocol followed is slightly different to the one described in the literature [1] as the aim was to obtain powder and not beads. So, no silicon oil was used. The protocol is described in Fig.5. It requires very concentrated actinide solutions with as less as possible free nitric acid. Urea is used to complex Pu in order to delay its hydrolysis. HMTA decomposes at high temperature leading to NH_3 generation that causes actinide hydrolysis.

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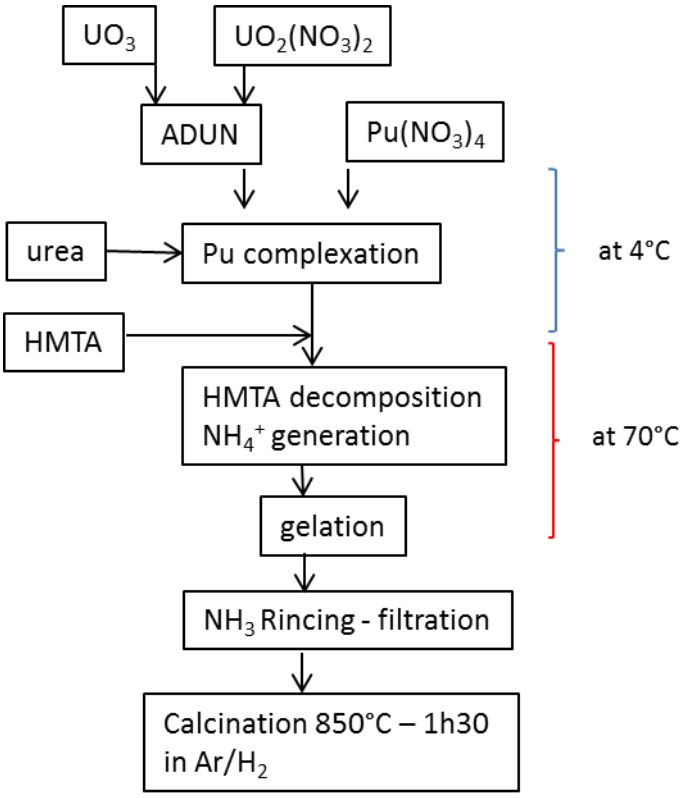

Fig.5 : sol/gel process for $(U,Pu)O_2$ synthesis.

[Urea]/[U+Pu] and [HMTA]/[U+Pu] ratios of 1.7 and 2 respectively were used except for one sample (Pu/U+Pu = 55%). A [HMTA]/[U+Pu] ratio of 1.7 was used to make the gelation in a different pH. One sample was also calcined at 1000°C under Ar+100ppm O_2 to obtain an evolution of the microstructure.

Acid Deficient Uranium Nitrate solution preparation

In sol/gel route, uranium is added using ADUN solution. Such solution has a uranium concentration close to 700 $g.L^{-1}$ and a free acidity very low as its pH is around 1.8. To obtain such solution, some ammonium uranate is precipitated by addition of ammonia in uranium nitrate solution. UO_3 is then obtained by calcination under air atmosphere at 430°C of the precipitate. Then, this uranium trioxide is dissolved in a uranium nitrate solution.

The Plutonium rich-solution :

In order to concentrate a Pu nitrate solution without concentrating the free nitric acidity, a distillation column was used associated with many additions of as less acidic as possible nitric solution. Ideally, water should be used but the addition of water would cause Pu hydrolysis locally prior to the mixing of the solution. As shown in Fig. 6, the more acidic the solution to be evaporated, the more acidic the vapor and the effective denitration [2]. Therefore, it is better to add little volumes of low acidic solution frequently than bigger volumes less often to keep the acidity of the solution to distillate as high as possible during the process of denitration.

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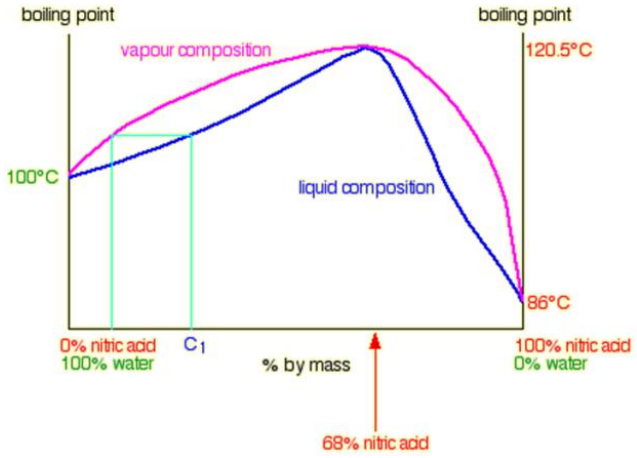

Fig.6 : Distillation of nitric acid, link between acidity of the solution to be evaporated and the acidity of the vapor produced.

The addition of nitric solution of concentration below 0.5 M leads to Pu hydroxide colloids generation. Nitric acid of 0.5M was then used. As described in Fig.6, the acidity of the solution to evaporate has to be higher than 3 M in order to evaporate more nitric acid than the 0.5 M added. Using this method, a Pu solution with [Pu] =510 $g.L^{-1}$ and [HNO_3] = 1.85 M was obtained. A picture of the nitric distillation apparatus is shown in Fig7.

Fig.7: nitric distillation for concentration and denitration of a Pu solution

The colloid synthesis route

As mentioned previously, addition of nitric acid less acid than 0.5M in a Pu solution leads to colloid formation. [HNO_3] 0.1M was added to some Pu solution and then the colloidal suspension was filtered. Calcination at 850°C under air was then done to obtain another PuO_2 exhibiting a different morphology. Another calcination of a part of the batch was done at 1500°C under Ar+500 ppmO_2 in order to modify the morphology.

CHARACTERISATIONS OF THE MODEL COMPOUNDS SYNTHETIZED

Chemical composition of the samples

Total dissolutions of a small quantity of each (U,Pu)O_2 sample were done carried out using boiling HNO_3/HF (4M/0.05M). Then, $\frac{[Pu]}{[U+Pu]}$ ratio was determined for all the samples using TIMS (Thermal Ionization Mass Spectrometry) with a precision of 2 rel%. The results are given in Tab.1. This determination was only done on sample calcined at 850°C. For samples calcined at different temperatures, it is assumed than no modification of the composition occurred during the second calcination.

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Excepted for two samples, the analyzed chemical compositions were in agreement with respect to the targeted compositions. It has to be noted that using highly concentrated actinide solutions, little deviation in the volume used has a huge impact on the composition.

Tab.1 : chemical composition of the $(U,Pu)O_2$ model compounds.

Synthesis route	N°	Targeted oxide	Obtained oxide
Needle oxalic route	1	$U_{0.75}Pu_{0.25}O_2$	$U_{0.75}Pu_{0.25}O_2$
Sol-gel route	2	$U_{0.75}Pu_{0.25}O_2$	$U_{0.75}Pu_{0.25}O_2$
	3	$U_{0.65}Pu_{0.35}O_2$	$U_{0.64}Pu_{0.36}O_2$
	4	$U_{0.45}Pu_{0.55}O_2$	$U_{0.43}Pu_{0.57}O_2$
	5	$U_{0.45}Pu_{0.55}O_2$	$U_{0.39}Pu_{0.61}O_2$
	6	$U_{0.35}Pu_{0.65}O_2$	$U_{0.39}Pu_{0.61}O_2$
	7	$U_{0.25}Pu_{0.75}O_2$	$U_{0.24}Pu_{0.76}O_2$

X-ray diffraction.

All the oxides synthetized were analyzed by XRD. All the samples are single phase oxides with a fluorine cubic structure and are well crystallized. As examples, XR diffraction patterns of oxides obtained via sol-gel route are shown in Fig.8. No impurities were observed.

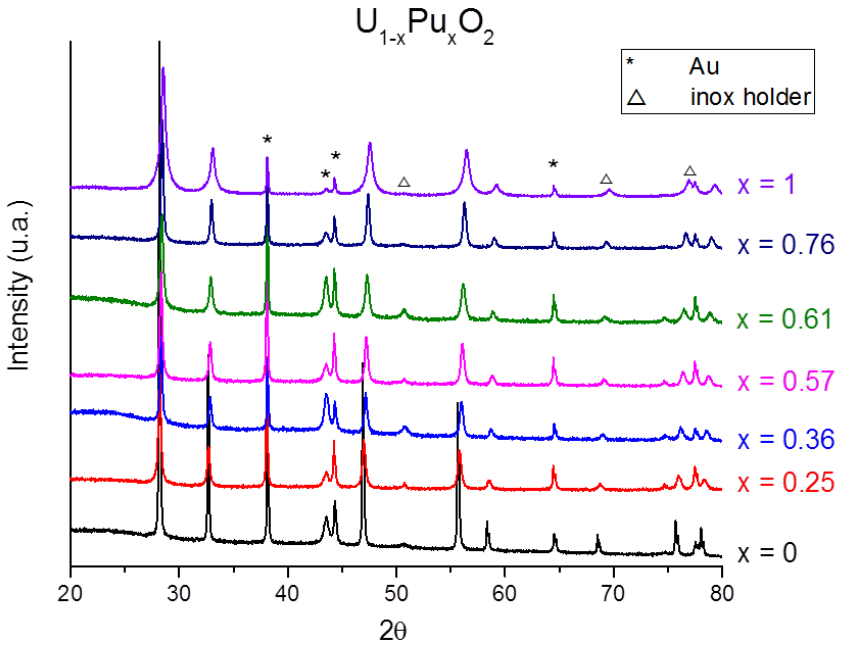

Fig.8 : X-Ray Diffraction patterns on oxides made using sol-gel route

The lattice parameters were determined by Le Bail refinement using the TOPAS software (TOtal Pattern Analysis Solutions) from BRUKER AXS **Erreur ! Source du renvoi introuvable.**] where only the profile parameters (cell dimensions, peak shapes, background, zero point correction) were refined. The microstrains and average crystallite sizes were calculated with the fundamental parameters method [4]. Results are given in Tab.2.

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All the crystallite sizes are on the same order of magnitude. (from 40 to 100 nm) excepted for UO_2, that exhibits higher crystallite sizes, and PuO_2 synthetized by sol-gel route that has smaller crystallites. These results are in agreement with the observations made on SEM

The O/M ratio of the different $U_{1-x}Pu_xO_{2+\partial}$ oxides were determined using the following equation:

$$\Leftrightarrow \partial = (1-x) * y = (1-x) * \frac{5{,}467\ (1-x) + 5{,}396x - a_{exp}}{0{,}094}$$

with a_{exp} the cell parameter.

Excepted one sample (61 % sol-gel) the O/M ratios were found to be close to 2.00. For this specific sample the oxide appears to be under stoichiometric indicating a partial reduction of Pu.
Microstrains are very low and all in the same order of magnitude.

Tab.2: XRD refinements on model compounds.

Compound	Synthesis route	Sample Name	Cell parameter (Å)	Crystallite size (nm)	microstrains (%)	O/M
UO_2	Needle oxalic route	UO_2 P Ar	5.4688 (1)	161 (4)	0.1 (0)	2.02 ± 0.02
	Square platelet oxalic route	UO_2 P Ar/H_2	5.4704 (1)	167 (3)	0.1 (0)	2.01 ± 0.02
	Square platelet oxalic route	UO_2 P air+Ar/H_2	5.4686 (2)	360 (8)	0.1 (0)	2.03 ± 0.02
	Needle oxalic route	UO_2 B	5.4704 (2)	129 (8)	0 (0)	2.01 ± 0.02
	Sol-gel	UO_2 SG	5.4694 (1)	164 (2)	0.1 (0)	2.02 ± 0.02
$U_{0.75}Pu_{0.25}O_2$	Needle oxalic route	25% B	5.4497 (2)	66 (1)	0.2 (0)	2.01 ± 0.02
	Sol-gel	25% SG YZ	5.4512 (1)	46 (1)	0.3 (1)	2.01 ± 0.02
	Sol-gel	25% SG GL	5.4493 (2)	34 (1)	0.1 (0)	1.99 ± 0.02

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$U_{0.64}Pu_{0.36}O_2$	Sol-gel	36% SG	5.4412 (2)	48 (1)	0.2 (0)	2.00 ± 0.02
$U_{0.45}Pu_{0.57}O_2$	Sol-gel	57% SG	5.4276 (1)	49 (1)	0.3 (1)	2.02 ± 0.02
$U_{0.39}Pu_{0.61}O_2$	Sol-gel	61% SG a	5.4321 (1)	43 (1)	0.2 (0)	1.92 ± 0.02
$U_{0.39}Pu_{0.61}O_2$	Sol-gel	61% SG b	5.4228 (1)	33 (1)	0.3 (0)	2.01 ± 0.02
$U_{0.39}Pu_{0.61}O_2$	Sol-gel 1000°C	61% SG c	5.4241 (1)	69 (2)	0.1 (0)	2.00 ± 0.02
$U_{0.24}Pu_{0.76}O_2$	Sol-gel	76% SG	5.4131 (1)	56 (2)	0.3 (1)	2.04 ± 0.02
PuO_2	Sol-gel	PuO_2 SG	5.3980 (1)	17 (1)	0.1 (0)	1.98 ± 0.02
	Needle oxalic route	PuO_2 B	5.3958 (2)	75 (2)	0.2 (0)	2.00 ± 0.02
	Square platelet oxalic route	PuO_2 P	5.3960 (1)	92 (3)	0.2 (0)	2.00 ± 0.02
	Colloïd route	PuO_2 C	5.3969 (1)	270 (15)	0	1.99 ± 0.02

Specific surface areas determined by BET method

The specific surface areas of all the oxides were determined using BET method with nitrogen (Tab.3), all the values range from 0.5 to 6 $m^2.g^{-1}$.
These low specific surface areas are in agreement with published values for actinide oxides calcined over 800°C. [5-6].

Tab.3: Specific surface area determined by BET method using nitrogen.

Compound	Synthesis route	Sample name	SSA ($m^2.g^{-1}$)
O_2	Square platelet oxalic route	UO_2 P Ar	1 ± 0.2

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	Square platelet oxalic route	UO_2 P Ar/H_2	1 ± 0.3
	Square platelet oxalic route	UO_2 P air+Ar/H_2	1 ± 0.3
	Needle oxalic route	UO_2 B	2 ± 0.2
	Sol-gel	UO_2 SG	2 ± 0.2
$U_{0.75}Pu_{0.25}O_2$	Needle oxalic route	25% B	2.8 ± 0.2
	Sol-gel	25% SG YZ	1 ± 0.2
	Sol-gel	25% SG GL	0.2 ± 0.2
$U_{0.64}Pu_{0.36}O_2$	Sol-gel	36% SG	0.4 ± 0.2
$U_{0.45}Pu_{0.57}O_2$	Sol-gel	57% SG	0.4 ± 0.3
$U_{0.39}Pu_{0.61}O_2$	Sol-gel	61% SG a	1 ± 0.1
	Sol-gel	61% SG b	1.5 ± 0.3
	Sol-gel 1000°C	61% SG c	0.9 ± 0.2
$U_{0.24}Pu_{0.76}O_2$	Sol-gel	76% SG	2.2 ± 0.2
PuO_2	Sol-gel	PuO_2 SG	6.3 ± 0.5
	Needle oxalic route	PuO_2 B	2.7 ± 0.4
	Square platelet oxalic route	PuO_2 P	3.3 ± 0.4
	Colloïds	PuO_2 C	0.2 ± 0.2

SEM images

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The different UO_2 microstructures obtained are presented in Fig. 9. Relative observations on these samples are summarized in Tab. 4. For all these oxides the crystallite sizes observed using SEM are in agreement with the crystallite sizes determined by XRD refinements. The UO_2 synthetized using sol-gel route is composed of big porous aggregates. For the three compounds obtained by conversion of $U^{IV}(C_2O_4)_{2,6}H_2O$ the square platelet sizes vary from 4 to 8 µm except for the sample calcined twice for which the grain size is increased by a factor two due to the increase of the total calcination duration. For this powder an intergranular porosity is also observed, probably due to the U_3O_8 reduction onto UO_2. U_3O_8 crystallizes in a 30% bigger cell than UO_2 [7]. Thus, the second thermal treatment may have not been long enough or been done at a too low temperature to totally remove this intergranular porosity. The last UO_2 sample is composed of big agglomerates of little hexagonal needles as expected for the conversion of $U_2M_2(C_2O_4)_5,nH_2O$ (with $M^+ = H_3O^+$, $N_2H_5^+$).

Tab.4: SEM observations on UO_2 samples

	Synthesis route and calcination	Name	Aggregate size (µm)	Grain size (nm)	Comments
1	Sol-gel Ar/H_2 (96/4)	UO_2 SG	216 ± 56	~ 200	Big porous aggregates
2	Square platelets oxalic route Air + Ar/H_2 (96/4)	UO_2 P Air+Ar/H_2	6 ± 2	300-800	Intergranular porosity
3	Square platelets oxalic route Ar	UO_2 P Ar	6 ± 2	150-400	Ø
4	Square platelets oxalic route Ar/H_2 (96/4)	UO_2 P Ar/H_2	6 ± 2	150-400	Ø
5	Needles oxalic route Ar	UO_2 B	18 ± 15	~ 100	Agglomerated needles

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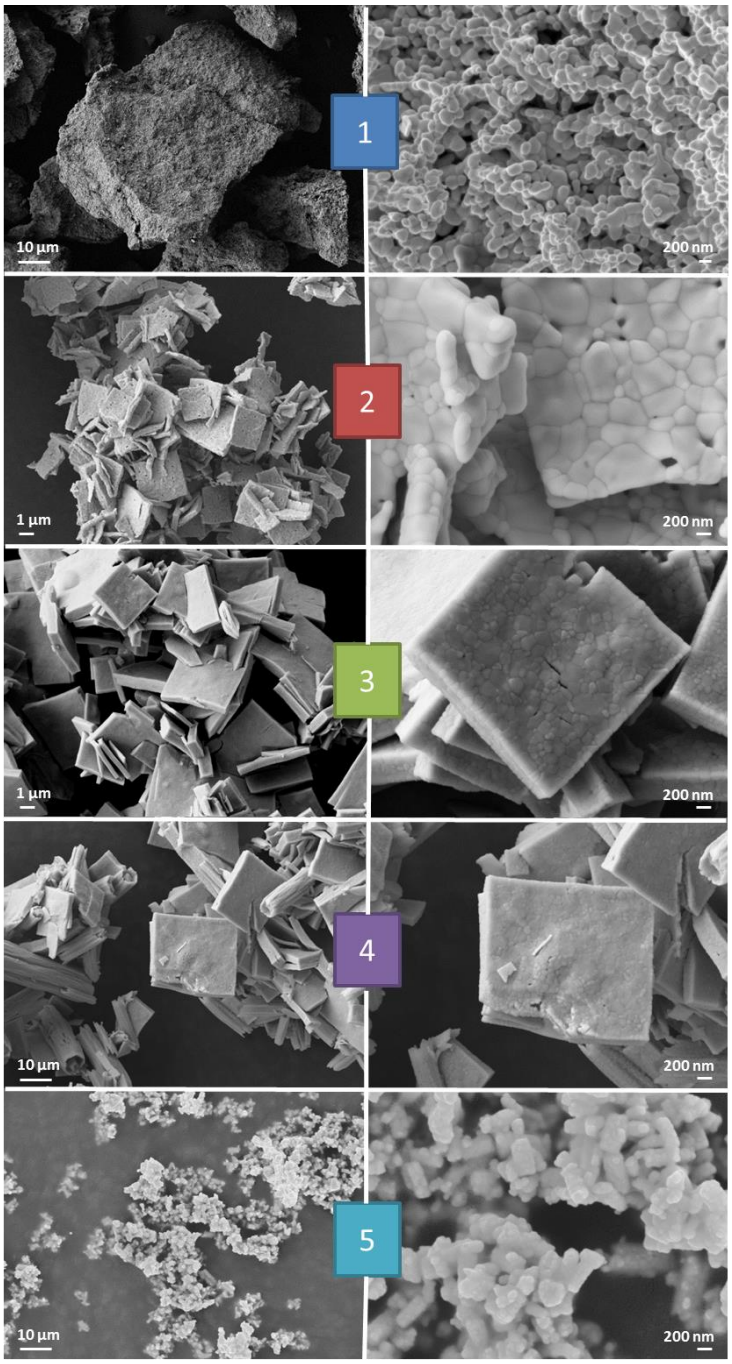

Fig.9 : secondary electron micrographies of UO_2 synthetized using the different conversion routes. Numbers being those reported in Tab.4.

Tab.5 and Fig.10 are relative to the other samples made using oxalic routes. The square platelets of PuO_2 have similar sizes to those observed on UO_2. However, the grain size looks smaller. The two other samples are composed of hexagonal needles of several tens of µm. Their grain sizes are in agreement with the crystallite sizes determined by XRD refinements (tens of nm).

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Tab.5: SEM observations linked to Fig.10.

	Synthesis route and calcination	Name	Aggregate size (µm)	Grain size (nm)	Comments
1	PuO_2 Square platelet oxalic route Ar	PuO_2 P	7 ± 1	50 - 100	Square platelets
2	PuO_2 Needles oxalic route Ar	PuO_2 B	25 ± 5	< 100	Hexagonal needle agglomerates
3	$U_{0.75}Pu_{0.25}O_2$ Needles oxalic route Ar	25% B	108 ± 55	n.d.	Hexagonal needle agglomerates

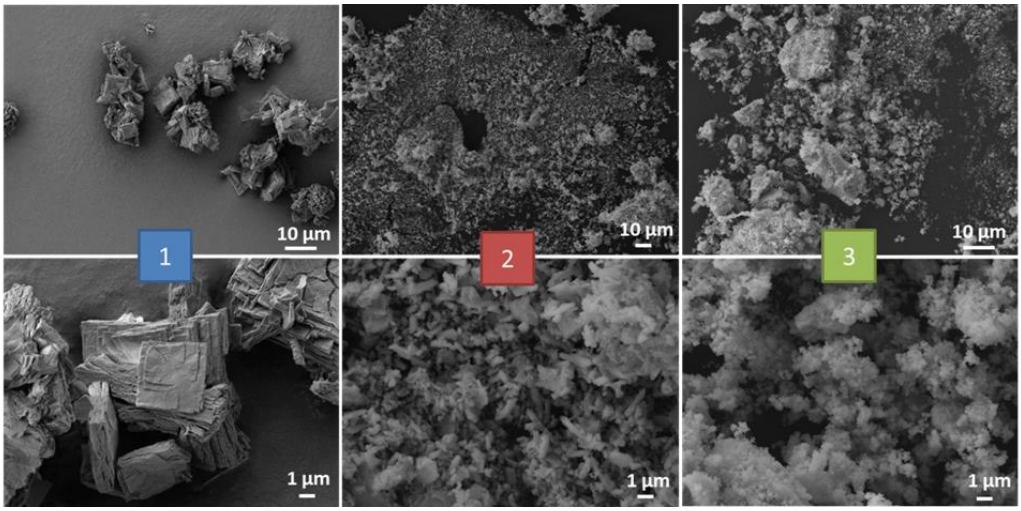

Fig.10: secondary electron micrographies of other samples synthetized using the oxalic routes. Numbers being those reported in Tab.5.

Oxide compounds of Fig.11 and Tab.6 are mixed oxides or PuO_2 made using sol-gel route. For all these samples, crystallite sizes are too small to be observed by SEM (< 100nm). This is in agreement with the values determined by XRD refinements. All these samples are composed of big porous agglomerates (several hundreds of µm long).

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Tab.6: SEM observations linked to Fig.11

	Synthesis route and calcination	Name	Aggregate size (µm)	Grain size (nm)	Pore size (µm)	Comments
1	$U_{0.75}Pu_{0.25}O_2$ Sol-gel Ar/H_2 (96/4)	25% SG YZ	207 ± 33	Ø	2	Big porous aggregates
2	$U_{0.43}Pu_{0.57}O_2$ Sol-gel Ar/H_2 (96/4)	57% SG	97 ± 55	Ø	0.90	Big porous aggregates
3	$U_{0.24}Pu_{0.76}O_2$ Sol-gel Ar/H_2 (96/4)	76% SG	75 ± 75	Ø	0.75	Big porous aggregates
4	PuO_2 Sol-gel Ar/H_2 (96/4)	PuO_2 SG	63 ± 41	Ø	Ø	Big porous aggregates

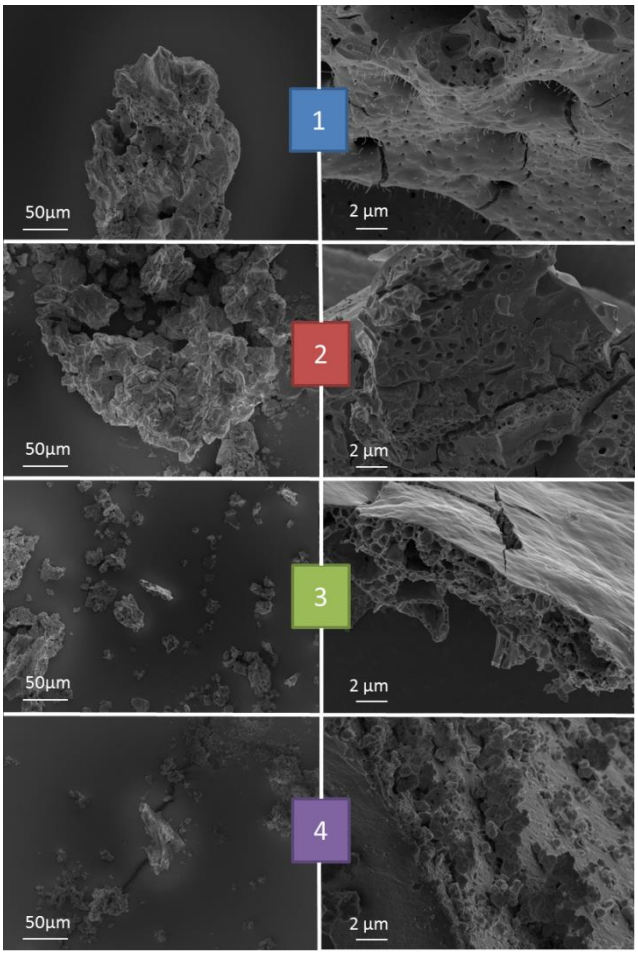

Fig.11: secondary electron micrographies of samples synthetized using the sol-gel routes. Numbers being those reported in Tab.6.

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EDX analyses were also done to verify the homogeneity of all the samples. All the results are in agreement with the composition analyzed by TIMS on total dissolution liquors within an uncertainty of 5% due to a lack of flatness of unpolished powder samples. It is then possible to conclude on the homogeneity of all the samples.

SEM images in secondary electron mode of PuO_2 obtained by calcination at 1500°C of colloids are shown in Fig.12. The sample is composed of sintered grain agglomerates with a size over 1mm long. Some intra and intergranular pores of several hundreds of nm long are also observed.

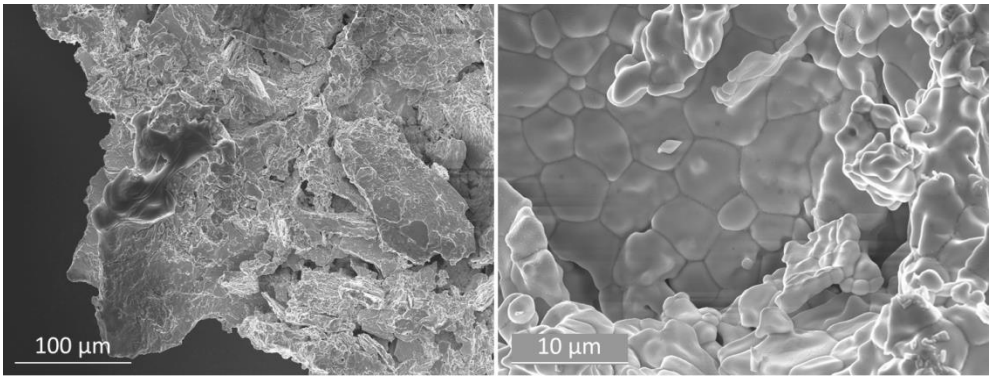

Fig.12: secondary electron micrographics of PuO_2 made by calcination at 1500°C of colloids.

Conclusion

Eighteen samples of $(U,Pu)O_2$ were synthetized and characterized with a Pu amount ranging from 0 to 100 % and with different morphologies in order to be able to collect data on dissolution kinetics on single phases depending on the dissolution parameters in one part and on the solid parameters (Pu amount, morphology parameters: crystallite size, SSA,…). This synthesis work constitutes the Milestone 7.1 which is therefore achieved.

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DIFFICULTIES

None

ACTION PLAN

Dissolution experiments will now be carried out on theses samples in order to collect data to build a dissolution model.

TASK 7.2

Nothing done on this Task during this semester

TASK 7.3

MANPOWER

Expected effort for the period 3 men.month

Actual effort spent during the period 3 men.month

MAIN PROGRESSES

Dense pellet fabrication of Americium-based oxides from co-converted oxalates

1. Introduction

In advanced nuclear fuel cycles which intend to manage recycling of the main long-lived radionuclides, such as plutonium and minor actinides (MA: americium, neptunium and curium), the use of mixed-oxide fuels will be required. In the case of plutonium, MOX fuels, composed of uranium–plutonium mixed oxides, are currently used in PWRs (pressurized water reactor), whereas for minor actinides, several types of fuels are being studied. These notably include MA-MOX, MOX fuels containing several percent of minor actinides [1], or MABB (minor actinide bearing blankets), uranium–minor actinide oxide fuels with up to 20 at.% of one or several minor actinides and dedicated to the periphery of the core [2,3]. In this context, several processes are being developed to produce mixed-oxide fuels complying with specifications in terms of dimensions, density, open porosity, grain size, homogeneity, etc. [3–6]. Currently, the most common processes, i.e., those employed at an industrial scale, are based on a reactive sintering of single oxide powders and only include dry steps such as milling, grinding and thermal treatments. They are particularly suitable for single-oxide fuels, such as UOX (UO_2) but, in the case of mixed oxides, such as MOX or MABB fuels, the efficiency of these processes in terms of microstructure (notably the fuel pellet densities) and homogeneity control remains limited [3, 4, 7, 8]. Besides, these dry steps are generally associated with a major drawback: the formation of large amounts of radioactive dust. With the UMACS process, a solution was proposed to overcome limitations in terms of density and homogeneity in MABB fuels [4, 9], but it requires additional steps including a long, thus dust-generating, grinding step and two thermal

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treatments at high temperatures [4]. In the context of the GEN-IV International Forum [10], the recycling of the valuable actinides and/or the transmutation of the minor ones has motivated the development of innovative and robust synthesis methods. Among them, oxalic (co-)conversion has been developed over several years by CEA and AREVA NC to obtain various compositions of highly reactive actinide mixed-oxide precursors. The advantages of such a synthesis route are mainly reduced dust generation, improved cationic homogeneity [11–13] and enhanced microstructural properties (especially homogeneity of pore distribution and grain size) for ceramic fuels. A fabrication of americium-bearing blanket (AmBB) fuels from $U^{+IV}_{1.7}Am^{+III}_{0.3}[N_2H_5^+,H_3O^+]_{2.3}(C_2O_4)_5,nH_2O$ oxalate thermally converted into $U_{0.85}Am_{0.15}O_{2\pm\delta}$ oxide powder has been done during this 1rst Geniors' 6 month period. The synthesis and characterization of the $U_{0.85}Am_{0.15}O_{2\pm\delta}$ mixed oxide powder is first presented and discussed in this activity report. Then, densification of a pellet prepared from the co-converted powder is studied by dilatometry and under different conditions. Finally, obtained fuels are compared to those fabricated using the UMACS process including a solid-state synthesis and a similar conventional sintering.

2. Powder synthesis and characterization

2.1. Synthesis

U (+IV) and Am (+III) solutions were prepared separately via dedicated protocols. U (+IV) monometallic nitrate solution was obtained through catalytic reduction of a U (+VI) nitrate solution by H_2 on a Pt/Si backing. Hydrazinium nitrate ($N_2H_5^+$, NO_3^-) was employed as an anti-nitrous agent to stabilize the (+IV) oxidation state of uranium. Am (+III) nitrate solution was prepared by dissolution of the corresponding oxide powder in concentrated hot nitric acid, with addition of hydrogen peroxide as a catalyzer. Americium oxide dissolution was carried out in a hot cell to limit the exposure of workers to radiation. The cationic concentration of each solution was determined by UV–visible absorption spectroscopy. As-obtained actinide (+III) or (+IV) solutions were mixed together in the desired ratio to achieve the selected cationic stoichiometry in the final oxide. This mixture was prepared as late as possible to avoid radiolysis phenomena. This solution and the precipitation agent, concentrated oxalic acid, were then added simultaneously in a vortex effect reactor. The precipitation occurred in free acid nitric media with an excess of oxalic acid. The oxalate precursor precipitated as soon as the reagents were in contact. From XRD (X-ray diffraction), the obtained solid is a mixed-oxalate phase, $U^{+IV}_{1.7}\ Am^{+III}_{0.3}\ [N_2H_5^+,\ H_3O^+]_{2.3}(C_2O_4]_5,\ nH_2O$, which has a hexagonal crystalline structure. The latter was already described in a previous work [11]. The crystallized powder was filtered and dried at room temperature, then treated for 3 h at 1023 K under an argon flow to form the required $U_{0.85}Am_{0.15}O_{2\pm\delta}$ oxide.

2.2. Co-converted powder characterization

2.2.1. Powder composition and residual carbon: TIMS measurements and TGA-µGC

After powder synthesis, thermal ionization mass spectrometry (TIMS) analyses were performed to assess the final Am/(U + Am) ratio. A VG-54 magnetic sector mass spectrometer was used. The obtained oxide was dissolved in nitric acid and diluted in water. An internal standard of known isotopic composition and concentration was added to the sample. Actinide content was determined and assessed from the measured final sample concentrations and the known internal standard concentration. The Am/(U + Am) ratio found equal to 15.8 ± 0.3 at.% is slightly higher than the target value (15 %), but remains in fair agreement with it. The ratio of carbon still remaining in the powder after calcination was estimated by thermogravimetric analysis (TGA) coupled with gas chromatography measurements (IGC). It was found to be equal to 2690 ± 120 ppm. Such high carbon content would presumably be problematic and generate additional porosity during the sintering thermal treatment unless it is properly removed before the densification [14].

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2.2.2. XRD Analysis

XRD analysis of the synthesized powder was performed using a D8 Bruker Advance diffractometer especially equipped for radioactive material measurements. For peak position correction, gold powder was added as a 2h standard. The diffractogram in Fig. 1 shows that a monophasic fluorite-type uranium–americium mixed oxide (space group 225) is present, without any additional phases. Impurities could not be revealed because of the detection limit of XRD. In particular, carbon present in too small quantity or in an amorphous state cannot be observed. A lattice parameter of 5.4615(3) Å was determined from Le Bail refinement using the Fullprof suite [15]. This value is smaller than those typically reported for this composition, i.e., around 5.468 Å [4, 16]. Such a difference, around 0.007 Å, is probably caused by a higher O/M (oxygen to metal) ratio for the co-converted compound. Most of the reported uranium–americium oxide materials were heat-treated at high temperature under reducing atmospheres, inducing lower O/M ratios than for the considered powder, calcinated under argon at a relatively low temperature. From Fig. 1 diffractogram, the mean crystallite size was estimated using the Williamson–Hall plot [17–19] and gives a value of 26 ± 3 nm for the $U_{0.85}Am_{0.15}O_{2\pm\delta}$ powder. From the refined lattice parameter and considering a hypothetical O/M ratio of 2, a $U_{0.85}Am_{0.15}O_2$ theoretical density of 10.993 g.cm^{-3} was established. This value was used to determine densities of the sintered samples.

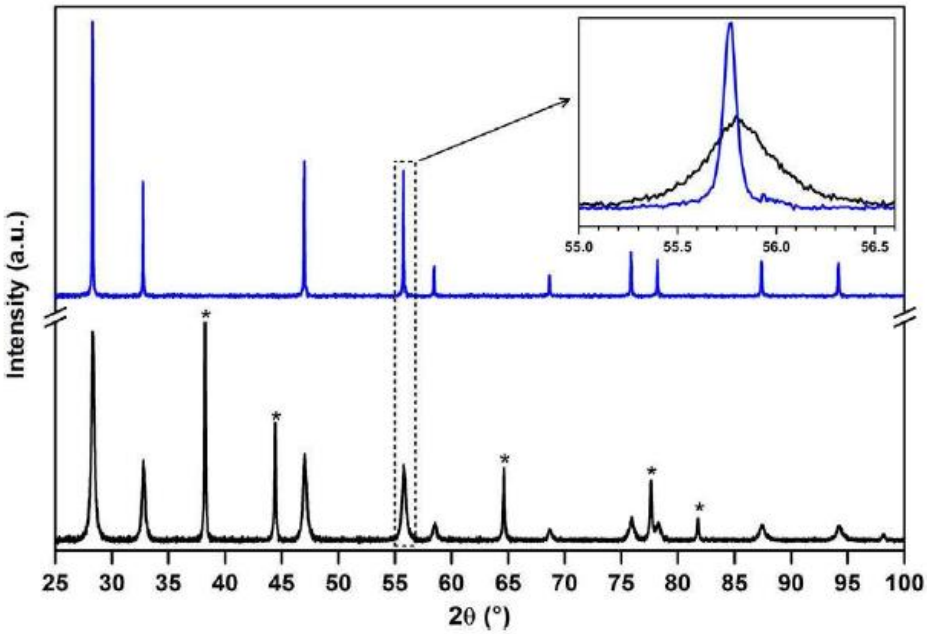

Fig. 1: XRD pattern of ground $U_{0.85}Am_{0.15}O_{2\pm\square}$ (—) after co-conversion (asterisks indicate Au standard diffraction lines) and (blue color) after sintering for 5 h at 1923 K under Ar/H_2 (4 %).

2.2.3. SEM Observations

The powder was characterized using a nuclearized Zeiss SUPRA 55/55VP FEG-SEM (field emission gun scanning electron microscope). SEM micrographs presented in Fig. 2 show a typical morphology for a wet chemical route. The powder is composed of large agglomerates of several hundred micrometers. These soft and porous agglomerates are composed of submicronic particles, similar to those obtained in AmO_2 powder synthesis [20], which includes precipitation in an aqueous environment followed by a filtration/crushing step. Because of this specific particle size distribution, proper laser granulometric measurements could not be performed.

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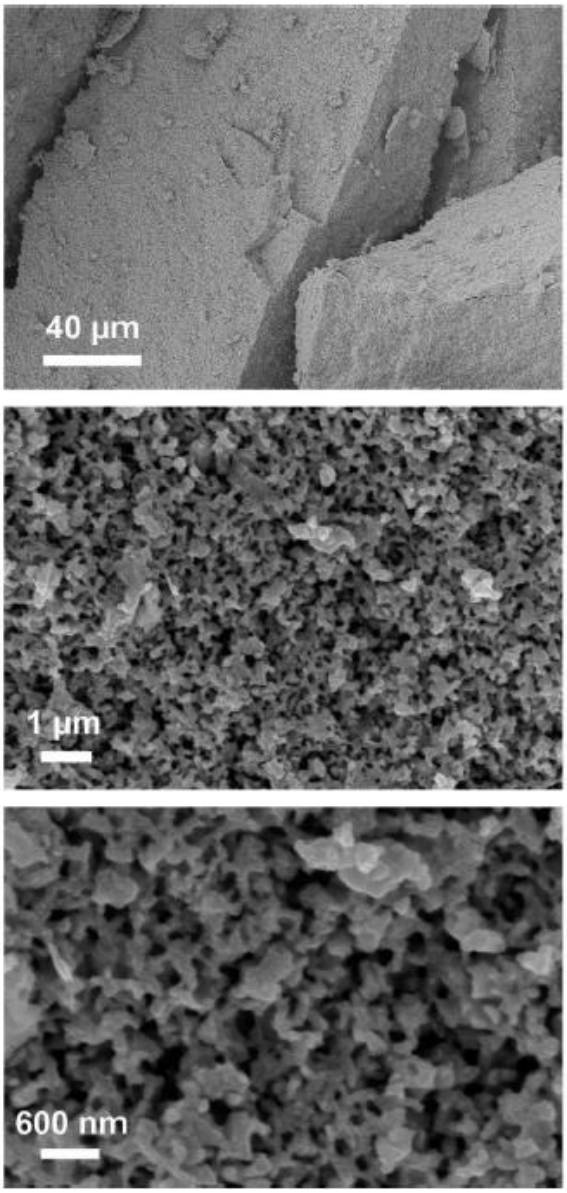

Fig. 2: FEG-SEM micrographs of co-converted $U_{0.85}Am_{0.15}O_{2\pm\square}$ powder in secondary electron mode.

3. Densification by sintering

3.1. Pelletizing Green pellets were prepared from the co-converted oxide powder.

Pelletizing was performed using uniaxial pressing (550 MPa for 10 s) in a 4.9-mm-diameter three-part die. Before adding the powder, the tungsten carbide die was lubricated with a saturated solution of stearic acid in ether. No lubricants or additives were, however, added to the powder. Despite the presence of large agglomerates in the precursor oxide powder, cylindrical green pellets exhibiting neither deformations nor defects were obtained, with a repeatable density of 49 ± 1 % TD.

3.2. Dilatometric analysis

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Dilatometric measurements were carried out using a horizontal Netzsch DIL 402 C dilatometer integrated in a glove box. Its furnace chamber, sample holder and push rod are made of graphite, but all parts in contact with the samples were tungsten to avoid any reactions between the oxide and the carbon at high temperatures. Extensive specifications are detailed in a previous publication [9]. The pellet linear shrinkage was recorded from RT (room temperature) to 2173 K with a heating rate of 3 $K.min^{-1}$ under a constant flow of Ar/H_2 (4 %) of 12 L h^{-1}. The relative variation of the sample length (dL/L0) is reported in Fig. 3, which also contains the shrinkage rate, i.e., the derivative of dL/L0 as a function of time. The graphical plotting of shrinkage rate is convenient for determining the onset temperature of sintering. For the $U_{0.85}Am_{0.15}O_{2\pm\delta}$ sample, the sintering begins around 800 K. The densification is then slowed down from 1200 till 1350 K, due to a possible additional crystallization or carbon loss. The highest shrinkage rate is reached between 1550 and 1750 K. At higher temperatures, the shrinkage rate progressively decreases and becomes null around 2000 K, indicating the end of sintering. Around 1900 K a change in the slope of the shrinkage rate curve is observed, which may indicate a change in sintering mechanism around this temperature. With this first sintering cycle performed without any temperature plateaus, a sintered pellet with a density of 94.3 ± 0.5 % TD was finally obtained. Considering the fast drop-off of the shrinkage rate beyond 1900 K, reaching higher temperatures is not required to achieve high densities, an isothermal plateau might be, however, necessary.

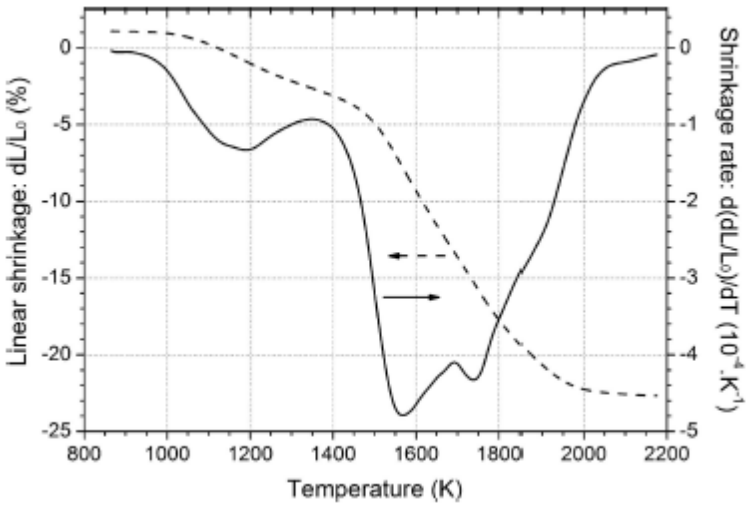

Fig. 3: Dilatometric curve and evolution of the shrinkage rate of the co-converted $U_{0.85}Am_{0.15}O_{2\pm\delta}$ compound at 3 K min^{-1} under Ar/H_2 (4 %).

3.3. Sintering experiments

Thermal treatments for sintering green pellets, coming from co-converted $U_{0.85}Am_{0.15}O_{2\pm\delta}$ powder were selected thanks to data obtained through dilatometric measurement. These sintering processes were carried out in an all-tungsten high-temperature furnace located in a hot cell. The selected process for elaboration of fuel pellets is a "simplified" process composed of only 2 steps: pelletizing and sintering, thus avoiding any grinding/dust generating steps. Three thermal treatments were applied under Ar/H_2 (4 %) on pellets with green densities around 50 % TD. The cycles only differ in their plateau temperatures:

- A: 293 K – 3 K min^{-1} → 1823 K, 5 h – 3 K min^{-1} → 293 K;

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- B: 293 K – 3 K min^{-1} → 1873 K, 5 h – 3 K min^{-1} → 293 K;
- C: 293 K – 3 K min^{-1} → 1923 K, 5 h – 3 K min^{-1} → 293 K.

These sintering processes led to pellets with densities of 93.9 ± 0.5 % TD, 94.7 ± 0.5 % TD and 95.7 ± 0.5 % TD, corresponding to Thermal Treatments A, B and C, respectively. For Cycles B and C, the 5 h plateau allowed reaching higher densities than that of the dilatometric experiment, despite the lower maximum temperature. As densities higher than 94 % TD are considered for AmBB compounds [3, 4, 21], only Treatments B and C appeared to give results complying with this requirement. Thus, only pellets sintered at 1923 K during 5 h were further characterized. It is worth noting that even though the oxygen potential varies with temperature, the variation between 1823 and 1923 K is, in such conditions, very low. The difference in density was thus not caused by the difference in oxygen potential. Fig. 2

3.4. Characterization of the pellets sintered at 1923 K for 5 h

3.4.1. Composition and carbon ratio: TIMS measurements and TGA-µGC

Americium oxides have high oxygen potentials, which induce a significant risk of sublimation that must be taken into account during the high-temperature reducing thermal treatment. TIMS analysis was thus performed on the sintered compound, giving an Am/(U + Am) ratio equal to 15.6 ± 0.2 at.%. This value, similar to that of oxide precursor, indicates that there was no americium sublimation at a significant level during the sintering process. The carbon content of the pellet sintered at 1923 K for 5 h under a reducing atmosphere was measured by TGA-lGC, under air, and found to be 365 ± 120 ppm. A decrease of 86 % of the initial carbon content was thus obtained during the thermal treatment. As this result was obtained without any optimization for this parameter, it may be possible to further lower the final carbon content, if necessary.

3.4.2. Structural aspects: XRD analysis

An XRD analysis was conducted on a powdered fragment of the pellet. After sintering, the obtained diagram (Fig. 1) still corresponds to a sole fluorite-type structure. Within the XRD detection limits, there are thus no additional phases. Besides, a notable improvement of crystallinity is observed, as the diffraction peak FWHM (full-width at half-maximum) are strongly reduced after sintering (Fig. 1). This is accompanied by an increase of crystallite size of at least 0.1 µm according to the Williamson–Hall plot. The lattice parameter was found to be equal to 5.4647(1) Å. This post-sintering increase in lattice parameter can be explained by a decrease of the O/M ratio and maybe by the removal of impurities. Concerning the O/M ratio, no attempts were made in this study to control it because of the lack of thermodynamic data for U–Am mixed oxides. It remains worth noting that this parameter could be controlled during the sintering heat treatment by changing the atmosphere, whether during the whole sintering cycle or only the plateau and/or during cooling. Previous studies reported that U–Am mixed-oxide sintering could be performed in various oxygen potentials ranging for instance between 520 and 325 kJ mol^{-1} [3, 4, 22].

3.4.3. Pellet profiles and morphologies

Before and after sintering, produced pellets were visually examined, and for all of them, no macroscopic defects were present. The pellet profiles were measured using a laser profilometer [23]. As an example, Fig. 4 shows the profile of a sintered pellet, representative of the tested samples. The maximum variation of the diameter is inferior to 12 µm. Moreover, no deformations pre- or post-sintering were observed. Fracture surfaces from sintered pellets were examined with a FEG-SEM in order to observe their microstructure. To gain an increased contrast during acquisition, the selected fragment was tilted. The sample fracture mode is here inter-granular. The micrograph in Fig. 5, representative of the overall sample, reveals a dense and homogeneous microstructure, composed of polyhedral grains of about 5–20 µm. A

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significant grain growth is evidenced by this grain size, compared to that of the initial powder. Only residual and submicronic pores can be observed for this sample. Such microstructural features confirm that the last sintering stage has been reached, in agreement with the sintered density.

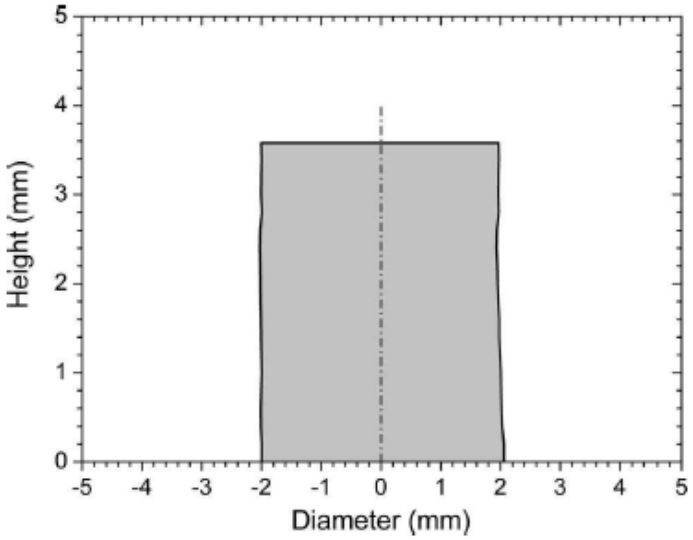

Fig. 4: Profile of a $U_{0.85}Am_{0.15}O_{2\pm\delta}$ pellet sintered for 5 h at 1923 K under Ar/H_2 (4 %)

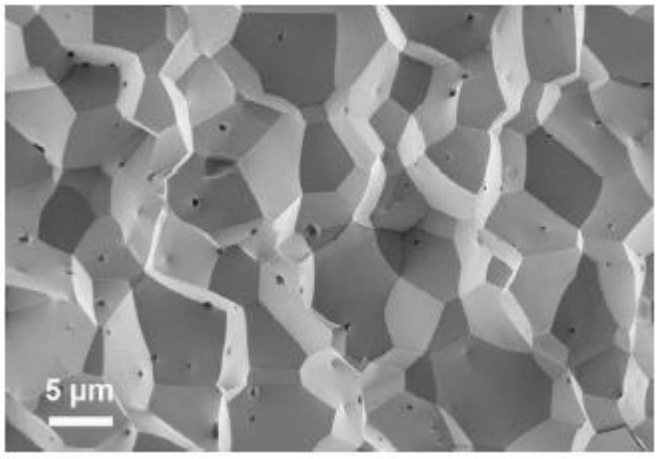

Fig. 5: FEG-SEM micrograph in secondary electron mode of the fracture surface of a $U_{0.85}Am_{0.15}O_{2\pm\delta}$ pellet sintered for 5 h at 1923 K under Ar/H_2 (4 %).

4. Comparison to another fabrication process

In this section, obtained results are compared to those commonly obtained with the UMACS process (Uranium Minor Actinide Conventional Sintering). This powder metallurgy process is used for $U_{1-x}Am_xO_{2\pm\delta}$ dense pellet fabrications from single oxides, and was notably employed for the analytic irradiation program DIAMINO [4]. Its main feature is the synthesis, before the sintering step, of the $U_{1-x}Am_xO_{2\pm\delta}$ compound through a solid-state reaction between single oxides at high temperature, as described in Fig. 6. Though this process makes the fabrication of pellets with densities superior to 94 % TD possible, there is a major drawback. Two high-temperature treatments are required, as well as an

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intermediate long grinding step which is responsible for dust generation. On the contrary, the simplified process based on the use of co-converted powder does not need a milling step, which limits dust creation. Another advantage of co-converted powders is the decrease of the required sintering temperature to reach the same target density of 95 % TD [4]. For this $U_{0.85}Am_{0.15}O_{2\pm\delta}$ compound, this temperature is at least 100 K lower than that used for the UMACS process. This difference is presumably a result of the high chemical reactivity of the co-converted powders due to the fineness of the powder particles, as evidenced by SEM micrographs in Fig. 2 and by the determined mean crystallite size of 26 ± 3 nm (value determined using a modified Thompson Cox Hastings Pseudo Voigt profile function for XRD diagram refinement). In summary, the co-converted route presents several advantages compared to the UMACS route: the simplification of the process through the reduction of the number of steps and the lowering of the sintering temperature and dust production, which could be clinchers for industrialization of this simplified process.

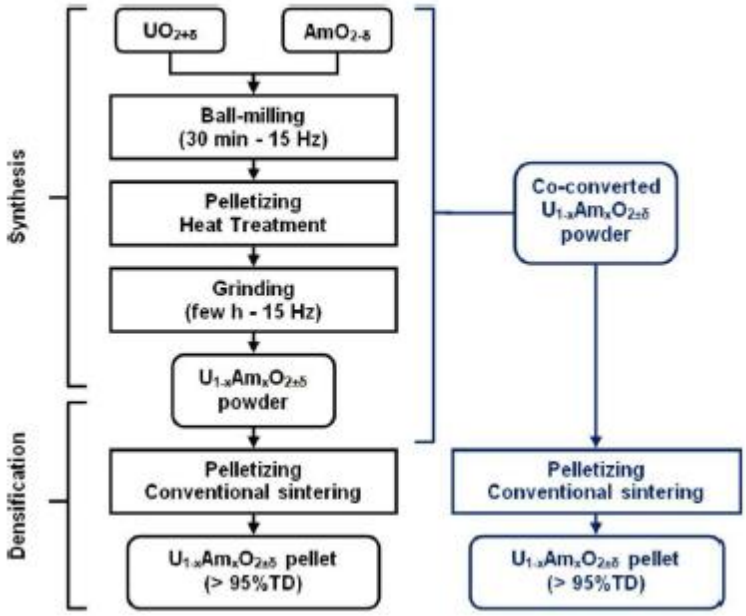

Fig. 6: Comparison between the flowcharts of the UMACS process (left) and that based on the use of co-converted powders (right) for $U_{0.85}Am_{0.15}O_{2\pm\delta}$ dense pellet fabrication.

5. Conclusion

The final goal of this work was to demonstrate the feasibility of the fabrication of U0.85Am0.15O2± dense pellets from co-converted powders. Dense pellets with homogeneous composition and microstructure, a low level of carbon, and a very limited residual porosity were obtained. These pellets were rectilinear, and neither deformations nor particular defects were observed. The selected process for production is a simplified one, with only two steps: pelletizing and sintering. Despite an extensive agglomeration of the particles in the initial powder, no grinding steps were required to reach high densities. This choice makes the reduction of dust production and associated risks possible compared to a conventional method such as the UMACS process. Residual carbon, coming from the initial co-converted oxalate and partially retained after co-conversion (~2700 ppm), was not a limiting factor in obtaining pellets with high densities. Final carbon content in sintered pellets was estimated at around 400 ppm, corresponding to a reduction of 86%. Sintering behavior of the $U_{0.85}Am_{0.15}O_{2\pm\delta}$ co-converted powder was studied under reducing atmosphere by dilatometry. From this study, a 5 h at 1923 K thermal treatment was selected to fabricate pellets. This

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sintering temperature is 100 K lower than that generally used to sinter the same compound synthesized via solid-state reaction, though both routes give similar results in terms of density and microstructure. This important improvement in AmBB elaboration can be attributed to the high reactivity of the co-converted powders compared to the powders obtained via solid-state reaction.

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DIFFICULTIES

None

ACTION PLAN

After this success in the production of dense (U,Am)O_2 pellet with less dust generation than conventional routes, using oxalic co-converted precursors, work will now be focused on another synthesis route avoiding powder precursor generation.

TASK 7.4

MANPOWER

Expected effort for the period 0.2 men.month

Actual effort spent during the period 0.2 men.month

MAIN PROGRESSES

Preparatory experiments to test methods and characterization data obtained when calcining some co-precipitated (U,Th) oxalate simulants are in progress.

DIFFICULTIES

None

ACTION PLAN

LIST OF PUBLICATIONS

None

CONCLUSIONS

A very good start of the project is to be noticed in WP7 with the achievement of M7.1 with the synthesis of the model compounds to be used for dissolution data collection and with the demonstration of dense pellet fabrication of Americium-based oxides from co-converted oxalates exhibiting less dust generation than conventional routes.

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DOMAIN 3 – WP8

DOMAIN	3	WP	8	WP Leader	LGI
Manpower dedicated to this WP on the period				1.8 (NNL) + 1 (LGI)	
Contribution to Deliverables (number and title of each deliverable)					
D8.4 – "SimPlant" – Engineering simulation of integrated plants (due in month 48) D8.3 Mapping of the different status and alternatives for each ESNII concept (due in M18)					
Contribution to Milestones (number and title of each milestone)					
None					
Main achievements - Progress					
D8.4 - SimPlant Flowsheet build of EURO-GANEX process from fuel receipt to powder finishing has begun. It should be noted that this also contributes to T81. D8.3 Mapping Initial work on the state of the art, literature review and putting together all available information. First draft of planning and methodology for deliverable. No work has been reported on deliverables D8.1 and D8.2 with deliverables due in M12. But work is schedule in the second semester of the project to undertake these two deliverables. Only LGI and NNL reported effort on this WP. EDF and AREVA will contribute later in the project.					
Main difficulties - Delays					
None					
Scientific publications, patents (beneficiary name and type of publication)					
None					
Effective collaboration between Beneficiaries (put crosses in boxes)					

	CEA	CHALMERS	CIEMAT	CNRS	CTU	ICHTJ	IIC	IRSN	JRC-ITU	JUELICH	KIT	LGI	NNL	POLIMI	SCK-CEN	TWENTE	UEDIN	UNIMAN	UNIPR	UNIVLEEDS	UREAD	ULANC	EDF	AREVA
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JUELICH																							
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INTRODUCTION

This WP addresses the industrialisation and scale up of some of the chemical projects studied in GENIORS. To develop processes towards industrialisation studies that consider the holistic impacts of the flowsheet are necessary. The tasks in this work package seek to asses and illustrate the holistic effects on the nuclear fuel cycle that occur from fundamental changes to the chemistry at the heart of its key processes. Appropriate technology deployment and consideration of potential issues and impediments to industrialisation will also be assessed.

TASK 8.1

MANPOWER

Total: NNL 3.5pm

MAIN PROGRESSES

Produce a concept design of an entire EURO-GANEX reprocessing plant, from receipt of spent oxide fuel through head-end process and dissolution to chemical separation and the production of oxide powders for re-use. The design pack will include a flowsheeting exercise to determine the mass flows of the main components and isotopes of interest through the process.

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Progress: Flowsheet build of EURO-GANEX process from fuel receipt to powder finishing has begun.

DIFFICULTIES

None

ACTION PLAN

Report to be delivered M12

TASK 8.2

MANPOWER

Total: KIT 2.9pm

MAIN PROGRESSES

Undertake a study comparing the different options for solvent extraction. The comparison will not be made with respect to the TRL of the individual processes but with respect to factors such as compatibility with upstream and downstream processes, compactness (number of stages, size of contactors) and auxiliary steps (solvent clean-up etc.)

Progress: No progress has been reported on this task

DIFFICULTIES

None

ACTION PLAN

Report to be delivered M12

TASK 8.3

MANPOWER

Total: LGI 3.9pm

MAIN PROGRESSES

This task proposes a mapping of the different status and alternatives for each ESNII concept waste treatment process.

The study will undertake a comparative assessment of the different recycling needs and options considered and will identify the facilities that could integrate the reprocessing options at EU level. Building on links with existing gen IV concepts (ASTRID, ALFRED, ALLEGRO) the task will evaluate the possibilities of increasing the capacities of existing EU nuclear facilities

Progress: state of the art, methodology established

DIFFICULTIES

None

ACTION PLAN

Report to be delivered M18

TASK 8.4

MANPOWER

Total: NNL 6.5pm

MAIN PROGRESSES

Develop a "SimPlant" model to illustrate how changes to a baseline could affect the size of the reprocessing and associated plants with which it has close interactions, e.g. waste treatment and vitrification plants. This will allow comparison of the holistic effect of chemical process changes on nuclear plants and site footprints.

Progress: Flowsheet build of EURO-GANEX process from fuel receipt to powder finishing has begun

DIFFICULTIES

None

ACTION PLAN

Report to be delivered M48

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

TASK 8.5

MANPOWER

Total: LGI 3.9pm

MAIN PROGRESSES

The last task of WP8 will assess he impact of implementing a closed cycle across the full nuclear fuel cycle, from mining and fuel fabrication to spent fuel conditioning. The task will use the results from previous tasks and will organise a set of face-to-face semi structured interviews to identify the main changes, main issues, blocking points and impact on the actors involved (some will disappear; some new actors will appear from opportunities in the cycle). The stakeholders

will encompass actors from industry, nuclear associations, policy makers, research centres, etc.

Progress: no progress has been reported in this period

DIFFICULTIES

None

ACTION PLAN

Report to be delivered M36

AREVA (1pm) and EDF (1.5pm) will participate in the whole WP as expert, participating to interviews and reviews of the deliverables.

LIST OF PUBLICATIONS

None

CONCLUSIONS

None

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

DOMAIN 3 – WP9

DOMAIN	3	WP	9	WP Leader	Dave Graham
Manpower dedicated to this WP on the period				6.3pm	

Contribution to Deliverables (number and title of each deliverable)

D9.6 – Assessment of the corrosion vulnerability of common plant materials in the presence of key process streams.

Contribution to Milestones (number and title of each milestone)

N/A

Main achievements - Progress

The objectives of D9.6:

- To perform corrosion assessments for stainless steels SS304L & S316L in the presence of as many of the GANEX inventory permutations as resource permits.
- To perform long term corrosion studies in relevant inventory permutations.
- To conduct corrosion and plant material compatibility tests for extraction and stripping stream compositions proposed for the EXAm process.

In conjunction with electrochemical quartz crystal microbalance (QCM) experiments, linear sweep voltammetry (LSV) studies have been conducted on the corrosion of SS304L and SS316L in the presence of the 1st cycle GANEX ligand, DEHiBA.

The presence of DEHiBA in the organic phase is found to have no effect on the corrosion rate of SS304L and SS316L in Exxsol D80. The presence of DEHiBA in the organic phase when that phase is contacted with nitric acid is found to reduce the rate of SS316L in HNO_3, but has no effect on SS304L.

Main difficulties - Delays

None so far.

Scientific publications, patents (beneficiary name and type of publication)

Journal:
Int. Conference Oral:
Int. Conference Poster:
Proceedings:
Patent:

None as yet for all of the above.

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

Effective collaboration between Beneficiaries (put crosses in boxes)																								
	CEA	CHALMERS	CIEMAT	CNRS	CTU	ICHTJ	IIC	IRSN	JRC-ITU	JUELICH	KIT	LGI	NNL	POLIMI	SCK-CEN	TWENTE	UEDIN	UNIMAN	UNIPR	UNIVLEEDS	UREAD	ULANC	EDF	AREVA
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INTRODUCTION

Work Pack 9 begins to investigate the safety implications of the advanced reprocessing flowsheets to inform future engineering design and flowsheeting chemistry. The work pack will cover both normal and mal-operations across the reprocessing cycle from head-end to powder finishing.

TASK 9.1 – PROCESS SAFETY

MANPOWER

None realised.

MAIN PROGRESSES

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

None realised.

DIFFICULTIES

None at this time.

ACTION PLAN

Task 9.1 will commence after month 12 as this is when the Concept Design phase will be complete and submitted. The Concept Design will inform the Safety Review.

TASK 9.2 – HAZARD ANALYSIS AND CRITICALITY STUDIES

MANPOWER

0.3pm

MAIN PROGRESSES

Task 9.2 – a "Preliminary hazard analysis": Information are expected from NNL to allow IRSN to start its safety review (characteristics of the spent fuel to be reprocessed, process diagram, main technical options adopted, flowsheet with mass flows of the main components and isotopes, key equipment items with operating conditions, etc.). As soon as Euro-GANEX plant design is confirmed and process design and flowsheet work make sufficient progress (WP8), NNL will share these data with IRSN.

Task 9.2 – "Criticality studies": Available data (chemical measurements) on solution densities, solvent activity and solvate concentration (Euro-GANEX process) have also to be collected for a range of solute concentrations to establish a density law for nuclear criticality safety evaluations.

IRSN drew up specifications which have been sent to NNL. NNL will ask the GENIORS community to identify who will be able to provide this information.

DIFFICULTIES

None at this time.

ACTION PLAN

Task 9.2-a: Start of the independent Euro-GANEX safety review focusing on criticality safety depending on available flowsheet and process data.

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

Task 9.2-b: Start of the writing up of the methodology to establish a density law for criticality safety evaluations (deliverable D9.3).

TASK 9.3 – QUANTIFICATION OF CORROSION RISKS IN EURO-GANEX AND EXAM PROCEESES

MANPOWER

6pm

MAIN PROGRESSES

In conjunction with electrochemical quartz crystal microbalance (QCM) experiments, linear sweep voltammetry (LSV) studies have been conducted on the corrosion of SS304L and SS316L in the presence of the 1st cycle GANEX ligand, DEHiBA.

The presence of DEHiBA in the organic phase is found to have no effect on the corrosion rate of SS304L and SS316L in Exxsol D80. The presence of DEHiBA in the organic phase when that phase is contacted with nitric acid is found to reduce the rate of SS316L in HNO3, but has no effect on SS304L.

DIFFICULTIES

None at this time.

ACTION PLAN

Electrochemical Impedance Spectroscopy (EIS) measurements to determine if an organic layer is the cause of surface corrosion inhibition.

Continue testing with TODGA.

LIST OF PUBLICATIONS

None

CONCLUSIONS

WP9 is currently delivering as planned with no immediate risks in the near future.

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

ANNEXES

TASK T9.3

The objectives of this task are:

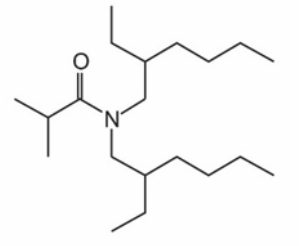
• To perform corrosion assessments for the process stainless steels SS304L & S316L in the presence of as many of the GANEX inventory permutations as resource permits.

• To perform long term corrosion studies in relevant inventory permutations.

• To conduct corrosion and plant material compatibility tests for extraction and stripping stream compositions proposed for the EXAm process.

N, N-di(2-ethyl hexyl) isobutyramide (DEHiBA) was the first ligand to be selected for study.

As the organic phase extraction ligand for uranium in 1st cycle extraction, it is present as 0.1 mol dm-3 DEHiBA in odourless kerosene in contact with 5 mol dm-3 HNO3 aqueous phase.



DEHiBA

Seven sets of linear sweep voltammetry (LSV) and electrochemical quartz crystal microbalance (QCM) experiments were conducted on each of the steels, SS316L and SS304L as follows.

• LSV and QCM experiments in 5 mol dm-3 HNO3

• LSV and QCM experiments in 5 mol dm-3 HNO3 contacted with Exxsol D80

• LSV and QCM experiments in 5 mol dm-3 HNO3 contacted with 0.1 mol dm-3 DEHiBA in Exxsol D80

• LSV and QCM experiments in Exxsol D80

• LSV and QCM experiments in 0.1 mol dm-3 DEHiBA in Exxsol D80

• LSV and QCM experiments in Exxsol D80 contacted with 5 mol dm-3 HNO3

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

• LSV and QCM experiments in 0.1 mol dm-3 DEHiBA in Exxsol D80 contacted with 5 mol dm-3 HNO3

Results were as follows.

For the organic phase experiments, both LSV and QCM measurements confirm corrosion in the organic phase (Octan-1-ol and Exxsol D80) is insignificant compared to aqueous phase (HNO3) corrosion.

DEHiBA appears to have little effect on corrosion in the organic phase, if anything acting as a slight corrosion inhibitor.

Contact of organic phase with a 5 mol dm-3 HNO3 aqueous phase produces no significant increase in corrosion in the presence or absence of DEHiBA.

For the aqueous phase experiments, LSV measurements show that SS304L and SS316L are significantly corroded in 5 mol dm-3 HNO3 over the potential range studied (0.3 to 1.2 V vs saturated silver chloride reference electrode).

SS316L is significantly more susceptible to transpassive dissolution than SS304L

Contact with the organic phase (Exxsol D80), inhibits transpassive corrosion of SS316L at potentials > 1V).

The open circuit corrosion rate as determined by calculation of the equilibrium corrosion current icorr almost halves after contact with an exxsol organic phase.

The presence of DEHiBA in the organic phase has little effect on SS304L corrosion rates, but further reduces corrosion of SS316L over Exxsol contact alone.

In summary: negligible corrosion occurs in the organic phase whilst DEHiBA is seen to protect SS316L against corrosion in an aqueous phase comprised of 5 mol dm-3 HNO3.

Further work: Thus, in the immediate term, electrochemical impedance spectroscopy (EIS) measurements will be conducted on SS304L and SS316L in 5 mol dm-3 HNO3 contacted with DEHiBA to determine if the formation of an adsorbed layer of DEHiBA at the steel surface is the cause of surface corrosion inhibition. This will be followed by a programme of experiments, similar to those described above, on the 2nd cycle GANEX ligand, TODGA.

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

DOMAIN 3 – WP10

DOMAIN	3	WP	WP10	WP Leader	SCK•CEN
Manpower dedicated to this WP on the period				0.12	
Contribution to Deliverables (number and title of each deliverable)					
D10.1 Clustering Events 1					
Contribution to Milestones (number and title of each milestone)					
MS11 List of EU projects and international initiatives to invite to the 1st workshop					
Main achievements - Progress					
At the 2nd Executive Committee meeting (ExCom2) held at Prague on 22nd of November 2017, it was decided to hold the first one around month 18 (i.e. around October 2018) to be organized by SCK•CEN and that it should be a three-day event. In the meantime, the agenda has been decided upon as follows: • Day 1: GENIORS Training and Education (T&E) event • Day 2: Clustering event, with expected participation from INSPYRE (still to be confirmed) and MYRRHA (confirmed) communities. Invitations will be sent to other researchers involved in the P&T field. • Day 3: Technical visit to the nuclear installations of the SCK•CEN The event will be organized in Antwerp (Belgium), date is still pending: weeks 41, 42 or 43. (During week 40, the international conference TOPFUEL will be held in Prague.)					
Main difficulties - Delays					
Responses from INSPYRE have not yet been received (as of mid-February 2018)					
Scientific publications, patents (beneficiary name and type of publication)					
Journal: Int. Conference Oral: Int. Conference Poster: Proceedings: Patent:					

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

Effective collaboration between Beneficiaries (put crosses in boxes)																								
	CEA	CHALMERS	CIEMAT	CNRS	CTU	ICHTJ	IIC	IRSN	JRC-ITU	JUELICH	KIT	LGI	NNL	POLIMI	SCK-CEN	TWENTE	UEDIN	UNIMAN	UNIPR	UNIVLEEDS	UREAD	ULANC	EDF	AREVA
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INTRODUCTION

The objective of WP10 "**Fuel Cycle Integration**" was to integrate the work done in GENIORS in a more global approach by creating synergies with other European and International initiatives and by involving stakeholders. This objective is to be achieved by organizing clustering events with the other European Projects sponsored under H2020 and involve other international research and development teams in these events.
Two tasks were defined in WP10:

- T10.1 Clustering with other European projects and international initiatives
- T10.2 Stakeholders/end-users Events

At the 2nd Executive Committee meeting (ExCom2) held at Prague on 22nd of November 2017, it was decided that of the four clustering events initially foreseen, only two would be withheld, one near the 18 month term and one near the end of the project.

At the same ExCom2 meeting, it was decided to organize the first clustering event in Antwerp in October 2018.

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

No activities were deployed for task 10.2 as the first activity for this task is foreseen only by month 24 (i.e. summer 2019).

TASK 10.1

MANPOWER

0.5 mm

MAIN PROGRESSES

Meeting places for the clustering event at month 18 have been selected, and need to be confirmed in February 2018.

DIFFICULTIES

Responses from INSPYRE have not yet been received (as of mid-February 2018).

ACTION PLAN

Confirm dates for clustering event and obtain response from INSPYRE.

TASK 10.2

No actions

LIST OF PUBLICATIONS

N/A

CONCLUSIONS

The action plan for Task 10.1 has been revised at ExCom2 meeting with the intention to organize two clustering events instead of four. Actions for the first clustering event are running.

No actions yet running for Task 10.2.

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

DOMAIN 4 – WP11

DOMAIN	4	WP	11	WP leader	S. Bourg
Manpower expected on the period		4pm	Manpower realized on the period		3pm
Contribution to Deliverables (number and title of each deliverable)					
Contribution to Milestones (number and title of each milestone)					
Main achievements - Progress					
Set up of the project: project quality plan, ECCP collaborative platform, diffusion list. Organisation of the Kick-off meeting in Aix en Provence, first Governing Board and ExCom, participation and post-processing. Consortium agreement preparation Initialisation of the HYPAR Organisation of the first GENIORS meeting in Prague, second excom					
Main difficulties - Delays					
Work to be done on the next semester					
WPASR1, continuous communication, monitoring of the progress Preparation of the second project meeting					
Scientific publications (journals, conference and status: submitted, approved, published) patents					
Effective collaboration with other Beneficiaries (give their name)					
CEA/LGI					

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

DOMAIN 4 – WP12

DOMAIN	4	WP	12	WP Leader	Bruce Hanson
Manpower dedicated to this WP on the period				0.25	

Contribution to Deliverables (number and title of each deliverable)

D421 Report on travel bursary and secondment grant attribution (ULEEDS)
D422 Organisation of the think-tank event (ULEEDS)
D423 Deliverable report on specification for on line packages (ULEEDS)
D424 Draft Purex package ready for testing (ULEEDS)
D425 iSanex/Ganex package ready for testing (ULEEDS)
D426 Packages tested and released (ULEEDS)
D427 Knowledge and Data Management Plan (CHALMERS)

Contribution to Milestones (number and title of each milestone)

MS14 Attribution of travel bursaries and/or secondment grants
MS17 Convene development group from project partners and agree roles for online training courses
MS18 Draft specification for on line packages for consultation
Write here the N° (Dx.y) of the milestones concerned by the work done in the semester. Mention when achieved.

Main achievements - Progress

T421 The process for applications and management of the grants has been established and a call for proposals has been sent out to all partners. To date 3 awards have been made.

T423 There has been an initial meeting to identify existing examples of online training packages. Delivery platform options have been identified.

Main difficulties - Delays

None to report.

Scientific publications, patents (beneficiary name and type of publication)

None planned.

Effective collaboration between Beneficiaries (put crosses in boxes)

	CEA	CHALMERS	CIEMAT	CNRS	CTU	ICHTJ	IIC	IRSN	JRC-ITU	JUELICH	KIT	LGI	NNL	POLIMI	SCK-CEN	TWENTE	UEDIN	UNIMAN	UNIPR	UNIVLEEDS	UREAD	ULANC	EDF	AREVA
CEA																								
CHALMERS																								
CIEMAT																								

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

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INTRODUCTION

This work package covers Training and Education and Knowledge Management. Work on the tasks has proceeded as planned.

TASK 421

MANPOWER

ULEEDS: 0.25 pm

MAIN PROGRESSES

The process for applications and management of the travel bursary and secondment scheme has been established and a call for proposals issues to all partners. Three awards – totalling – 6,780 Euro - have been made.

DIFFICULTIES

None

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

ACTION PLAN

Issue further calls for proposals.

TASK 423

MANPOWER

ULEEDS: 0.25 pm

MAIN PROGRESSES

An initial meeting with Leeds to identify existing examples has been carried out.
A number of options for the delivery platform have been identified:

- *Team with an external supplier; e.g. FutureLearn. ULEEDS has a partnership with FutureLearn who specialise in delivery of on-line courses.*
- *Team with Meet-CINCH and use the MOOC. The Meet-CINCH project is developing on-line material to support a Masters Programme. We could use the same platform as them.*
- *Use our own platform, such as the ULEEDS electronic blackboard.*
- *Create a standalone package.*

DIFFICULTIES

None

ACTION PLAN

Present an example syllabus at the 1st Annual meeting

Present options for the delivery platform at the 1st Annual meeting - outlining the advantages and disadvantages of each – prior to making the final decision.

LIST OF PUBLICATIONS

None planned

CONCLUSIONS

Progress of this work package is going to plan.

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

DOMAIN 4 – WP13

DOMAIN	4	WP	13	WP leader	C. Chavardès
Manpower expected on the period	1pm	Manpower realized on the period	1pm		

Contribution to Deliverables (number and title of each deliverable)

D13.1 Communication toolkit
D13.2 Communication and dissemination action plan
D13.3 Project website

Contribution to Milestones (number and title of each milestone)

Main achievements - Progress

- Communication toolkit (D13.1): designed the project logo and the project's PowerPoint presentation template.
- Developed and designed the official GENIORS public website: www.geniors.eu (D13.3).
- First series of online audience analysis through Google Analytics.
- Social media: daily management of the GENIORS Twitter account.
- Communication and dissemination action plan (D13.2): first version drafted and under review by project coordinator.

Main difficulties - Delays

Work to be done on the next semester

- Updates of the project website (deliverables for download, first results, news following project meeting in Prague…), and regular audience analysis (via Google Analytics).
- Daily management of the GENIORS Twitter account.
- Final version of the Communication and dissemination action plan (D13.2) to be submitted.
- Design of the communication materials for the different upcoming external and GENIORS events: flyer, poster, roll-up, and press releases.
- Editorial preparation of the first newsletter of the project, scheduled for June 2018.
-
-
-

Scientific publications (journals, conference and status: submitted, approved, published) patents

Effective collaboration with other Beneficiaries (give their name)

Stéphane Bourg (CEA) - LGI

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

INTRODUCTION

WP13 aims to engage with stakeholders and raise awareness on GENIORS and its achievements through specific communication actions. It will also ensure the dissemination of knowledge acquired towards the scientific community and other relevant stakeholders, including publications in scientific journals and participation in conferences.

TASK 13.1

MANPOWER

LGI was the only contributor to this task during this reporting period: 0.5 PM

MAIN PROGRESSES

The first elements of the communication toolkit were designed and distributed to all GENIORS partners to promote the project: this included the project logo and a PowerPoint presentation template.

DIFFICULTIES

- None -

ACTION PLAN

The logo will be used in project presentations, documents and reports, and communication material such as the project's flyer/brochure. A poster will be designed to be used at conferences, events and workshops in order to increase the project's visibility.

TASK 13.2

MANPOWER

LGI was the only contributor to this task during this reporting period: 0.5 PM

MAIN PROGRESSES

The communication and dissemination action plan deliverable D13.2 was drafted and submitted. It includes:

- Communication strategy: definition of audiences and key messages
- A detailed planning of all communication actions over the project duration according to the defined target audiences, including press releases
- An event and publications management plan

Task 13.2 will also disseminate the results of the GENIORS project to the scientific community and stakeholders by:

- Coordinating scientific publications, including open access journals, free (online) journals, and online repositories.

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227

- Coordinating the participation of partners in conferences to disseminate knowledge and results.

As we are early in the project, no results have yet been disseminated.

DIFFICULTIES

- None -

ACTION PLAN

The action plan will be implemented and monitored during the course of the project in order to identify areas of improvement and measure its success.

TASK 13.3

MANPOWER

This task has not yet started.

-

MAIN PROGRESSES

An electronic newsletter will be issued at the end of each year of the project to inform stakeholders of the project's progress.

DIFFICULTIES

-

ACTION PLAN

The first newsletter is planned to be distributed in June 2018. Exchanges with partners and information collection and drafting will start in April 2018.

TASK 13.4

MANPOWER

LGI was the only contributor to this task during this reporting period: 0.5 PM

MAIN PROGRESSES

A website was designed and released in September 2017.

DIFFICULTIES

Traffic to the website is limited. It is important to have news/updates from the various work packages shared with WP13 in order to update the website and make it a lively platform.

ACTION PLAN

The website will be maintained and continuously updated.

LIST OF PUBLICATIONS

-

CONCLUSIONS

WP13 is on track. However, the challenges that lie ahead are mainly the creation of content in order to populate the website and the GENIORS Twitter account. It is important for work package leaders to share updates in their WPs with the communications team in order to have information on which to communicate.

This project has received funding from the EURATOM research and training programme 2014-2018 under grant agreement No 730227