Establish objectives for advanced voloxidation with respect to the downstream effects

파이로 후속공정을 고려한 고도 휘발성산화공정의 목표 설정

2009. 9.
제 출 문

한국원자력연구원장 귀하

본 보고서를 사용후핵연료 고도 회발성 산화공정 기술개발 과제에서 수행한 “Establish objectives for advanced voloxidation with respect to the downstream effects”의 기술보고서로 제출 합니다.

제목 : Establish objectives for advanced voloxidation with respect to the downstream effects

2009 년 9 월

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SUMMARY

A voloxidation process is being considered at the Idaho National Laboratory (INL) and KAERI (Korea Atomic Energy Institute) as a head-end step for oxide reduction prior to electrorefining. The oxide reduction and electrorefining processes are the key steps to the pyroprocessing of spent oxide fuel. Three primary attributes of voloxidation are addressed in this report as they affect the pyroprocess. The attributes are reduced to particle size, fuel separation from the cladding or decladding, and volatile fission product removal. Therefore, this report described 4 possible cases and compared them in terms of electroreduction rate, product characteristics, salt carryover, etc., and gave grade to each case. A “1” indicates least effective or unfavorable while a “3” signifies most effective or beneficial. On the bases of evaluation, the best case was chosen. In order to successfully proceed the chosen process, a path forward was reviewed in terms of particle size control, fission product removal, off-gas treatment, etc.

The case (the low-temperature voloxidation taken to 1200°C under vacuum, so called advanced process) was chosen as the best option for head-end process of pyroprocessing. This justifies that the pursued direction (particle size control, fission product removal, selective nuclide trapping) of our project is right. INL and KAERI named our advanced voloxidation process as the PRESENT (Particle size control, fission product REmoval, , SElective Nuclide Trapping) process. Both parties hope that the PRESENT process will be a very good present for pyroprocessing.

Also, the required research areas and path forward of our project are discussed in the areas of particle size control, fission product removal, fission product trapping, respectively.
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1. Introduction

A voloxidation process is being considered at the Idaho National Laboratory (INL) and KAERI (Korea Atomic Energy Institute) as a head-end step for oxide reduction prior to electrorefining. The oxide reduction and electrorefining processes are the key steps to the pyroprocessing of spent oxide fuel. Three primary attributes of voloxidation are addressed in this report as they affect the pyroprocess. The attributes are reduced to particle size, fuel separation from the cladding or decladding, and volatile fission product removal. Therefore, this report described 4 possible cases and compared them in terms of electroreduction rate, product characteristics, salt carryover, etc. and gave grade to each case. A “1” indicates least effective or unfavorable while a “3” signifies most effective or beneficial. On the bases of evaluation, the best case was chosen.

In order to successfully proceed the chosen process, a path forward was reviewed in terms of particle size control, fission product removal, off-gas treatment, etc.

2. Evaluation of 4 cases

2.1 Considered Cases

Four cases of voloxidation are discussed: Case 1 assumes just chopping and no voloxidation, Case 2 is a low-temperature voloxidation performed at approximately 500°C following chopping, Case 3 is the low-temperature voloxidation taken to 1200°C under vacuum, and Case 4 is a hybrid of mechanical decladding followed by voloxidation at 500°C.

2.2. Evaluation Criteria

The evaluation following criteria are used in this report.

- Oxide reduction processing rate
- Product characteristics (e.g. particle size, density)
- Interference of cladding in following process steps
- Salt carryover to following process steps
- Degree and timing of fission product removal
- Material, control, and accountability verification
- Ease of product handling (containment and transfer)
• Waste reduction potential with regard to TRU in clad
• Ultimate disposition
• Flowsheet complexity in terms of time/money

2.3 Case 1

For case 1, the voloxidation step is not included in the pyroprocess and instead, oxide fuel is fed directly from a chopping step to the oxide reduction process. Since voloxidation reduces the particle size of spent oxide fuel drastically, a decreased reduction rate by at least a factor of three would be expected for non-voloxidized fuel based on tests performed with engineering scale oxide reduction equipment [1]. Tests to date at the lab-scale with non-voloxidized and voloxidized fuel have been inconclusive with respect to reduction rates considering the mechanical decladding operation for non-voloxidized fuel which renders similar particle size as voloxidized fuel [2]. A couple benefits of not including voloxidation in the process are ease of charge balancing UO$_2$ versus U$_3$O$_8$ during the oxide reduction step and increased bulk density for chopped fuel requiring less volume for processing.

Assuming no voloxidation step, fuel following the chopping step would be charged to the oxide reduction vessel with cladding. The cladding with reduced fuel is then transferred from the oxide reduction vessel to the electrorefining cell for final processing. Processing without cladding is advantageous to minimize potential side reactions of the cladding (zircalloy) with the salts for the two processes.

Several potentially volatile fission products are built-up in the fuel during reactor operations. For this report, the following fission products are key and considered only semi-volatile due to various research [3-7], including our own, that establishes incomplete release during the chopping and/or voloxidation operations: krypton, xenon, tritium, iodine, cesium, and technetium. A few other potentially volatile fission products should be mentioned but will not be discussed due to their relevance or similarity to discussed fission products, viz. rubidium, tellurium, ruthenium, and carbon-14. Considering Case 1, chopping will only result in negligible quantities of these semi-volatile fission products being released since most are either tied up chemically or physically within the fuel structure.
2.4 Case 2

The primary purpose of Case 2 is to declad the fuel into fine particles to increase the reduction rate during the oxide reduction step. Thus, only low temperature (~500°C) voloxidation is required to produce fines with sufficient decladding characteristics. One drawback to voloxidation is that the particle size produced may be too small, that is, containment of the particles both during oxide reduction and transfer operations is significantly more challenging. On the other hand, voloxidation products are well-homogenized and can be easily sampled for MC&A purposes whereas chopped fuel is prone to axial variations [2].

As a result of voloxidation, the inner surface of the zircalloy cladding is degraded by the force of fuel expansion and/or reaction with the oxidizing atmosphere. Based on cladding samples taken following voloxidation testing at the INL, the feasibility exists to reduce the TRU alpha-nuclide content to below that required for near surface disposal, i.e. less than 100 nCi/g. Potentially two options are possible for this, either pyroprocessing to etch the inner surface or more aggressive voloxidation conditions to completely remove the TRU content.

Another benefit or potential drawback for low temperature voloxidation is the removal of the semi-volatile fission products mentioned previously. The removal of krypton, technetium, and cesium would be expected to be between 10-30% [3, 6] while tritium release should be near 100% [4]. Xenon removal would be expected to be similar to krypton since only 5-10% of these gaseous molecules actually reside on the grain boundaries [8-9]. Iodine appears to track cesium removal (as CsI) but is much less prevalent than cesium so its removal would be expected to be less than 10% for low temperature voloxidation [10-11]. Although these fission products are removed from the process, a large fraction of them remain in the fuel and must be dealt with by later processes.

2.5 Case 3

The focus of Case 3 is to perform a low temperature voloxidation cycle for decladding and particle size reduction then to increase temperature under vacuum to completely remove the semi-volatile fission products mentioned previously. The same arguments for and against particle size during Case 2 apply to this case since particle
size is a function of oxidation temperature and fuel for both Cases 2 and 3 are oxidized at 500°C [7].

For Case 3, a two-step process or separation of the fuel and cladding after voloxidation at 500°C would be necessary to avoid oxidizing the cladding at the higher temperatures. Following the low temperature voloxidation, the cladding would have to be separated, either in-situ or by performing two process steps; one for decladding and one for fission product removal. This constraint would add complexity to the overall flowsheet when compared against Cases 1 and 2.

Since the complete removal of cesium, iodine, and technetium has been demonstrated at 1200°C under vacuum [7, 10], strontium remains the only problematic fission product of interest according to recent AFCI/GNEP flowsheets [12]. There are indications that strontium can be removed with very high temperatures (~1600°C) which would support the AFCI/GNEP flowsheets [13]. Ignoring strontium removal and given the relative concentrations of cesium and strontium in spent oxide fuel, the complete removal of cesium results in approximately 50% less radioactivity [14]. Another benefit of complete iodine removal by high temperature voloxidation may be during the oxide reduction operations. Recent oxide reduction testing has suggested problems with platinum contamination, possibly by iodine, although definitive results are pending [2]. Assuming complete removal of technetium, cesium, and iodine, these fission products could be directly incorporated into the pyroprocess waste forms (metal and ceramic) since these waste forms are their ultimate destination (Fig. 1).

Fig. 1. Fission Product Disposition following Voloxidation for Case 3
2.6 Case 4

For Case 4, a low temperature voloxidation at 500°C of declad fuel follows a mechanical decladding operation. The mechanical decladding operation would include slitting or crushing to promote the removal of fuel from the cladding. This case is very similar to that performed at the INL for the oxide reduction of non-voloxidized material and to that at KAERI in which fuel is declad by multiple axial slits prior to voloxidizing the resulting fuel pellets. A disadvantage to this option is that a small amount of fines would be produced as a result of the decladding operation which causes product handling problems. A potential advantage may be the ability to control the particle size by increasing the voloxidation temperature above 500°C [7].

Since the cladding is removed prior to voloxidation, contamination of the feed for the oxide reduction step is not as likely as for the other three cases. However, mechanical decladding would probably not be as efficient as voloxidation for fuel removal, particularly for the TRU content. Fission product removal without cladding at 500°C would be expected to be slightly better than Case 2 but not as effective as Case 3 based on previous research [7].

2.7 Additional comments on case 1

Electroreduction of rod-cut as it is can be considered as one of promising options for a starting materials of electroreduction process. In order to make rod-cuts, diamond saw and tube cutter are used for lab-scale experiment. On the while, shearing technology is used for commercial purpose. Therefore, Karell and Gourishankar [15] and Karell et al.[18] considered shearing characteristics of fuel elements for preparing their starting material of oxide reduction. They described that shearing a fuel bundle into 2.54 cm segments would dislodge 50% of the UO2 from the cladding. For testing the lithium reduction process, they used unirradiated stainless steel-clad fuel rods of 1-cm-long segments to represent the maximum expected particle size if fuel assemblies were sheared into 2.54 cm segments.

According to Davis et al.[22], after shearing irradiated fuel(spent LWR fuel), fines fraction under 40 μm is maximum 15.1 %. That of under 50 μm is maximum
18.1%. Normally used sieve for electro-reduction is No. 325 mesh. That is, its size of opening is 44 μm. In this case, fines under 45 μm can be 16.6%. This portion cannot be used in 325 mesh screen. So, it should be treated in other methods such as use for chemical analysis, treated in porous container, etc. In this regard, electroreduction of rod-cut as it is has one drawback.

Karell and Gourishankar [15] and Karell et al.[18] carried out oxide reduction experiments using clad UO2 fuel rods of 1 cm length and 0.95 cm OD. The reduction time was 36 hrs. On the other hand, crushed UO2 grain took ~12 hrs. They[18] described that the cladding effectively reduces the fuel surface area available for reaction. From these results, they noted that it is clear that the reaction rate of the relatively large, intact fragments inside the cladding hulls will determine the time for reducing a batch of chopped fuel.

According to Kleykamp[23], Zr is considered to be mainly dissolved in the fuel matrix. Calculation results by ORIGEN code show that its mass of spent PWR fuel(45,000 MWD/MTU, initial enrichment 4.5%, 5 yrs cooling) is ~ 5.0 kg/MTHM. It is larger than ~3.3 kg/MTHM of cesium. According to the data of Herrmann et al.[16, 19, 20] and Park[21], Zr was not detected by chemical analysis from the salts of electro-reducer. When 1 HMTU of spent PWR fuel is treated in electro-reducer, ~256 kg-clad contact with salt. Compare to 5 kg/MTHM, its value is very high (256/5= 51). So, in order to know the exact diffusion characteristics from cladding hulls, experiment on this will be needed. For reference, experiment on the case of stainless steel cladding was performed by Karell et al.[18]. Stainless cladding was intact after electro-reduction.

In summary, oxide reduction of rod cut takes too much time. And, fines fraction is too much to be used in wire screen container. In addition, behaviors of H-3, Tc and I must be taken into consideration. That is because I-129 and Tc-99 are long-lived nuclides and tritium is hazardous nuclide. The information on their behavior during electro-reduction seems to have not been published yet. That is, there may be still much uncertainty in applying this technology.
2.8 Comparison of case 2 and case 3

Considerable large fuel grains can be obtained from mechanical slitting. There may be no problem in particle size distribution. Fines portion under 45 µm may be neglected. However, the grain structure and off gas treatment efficiency have to be considered. At first, the grain density is ~93% of theoretical density. That is, porosity of grain is ~6%. On the other hand, the aggregates of the AVP are expected to have ~50% of theoretical density, and ~50% porosity. It means that reduction times for each case are quite different. The former case, the maximum grain size may be 1 cm. The latter case, the maximum grain size may be ~100 µm. According to Simpson and Herrmann’s model[17] and shrinking core model[24], the calculated reduction time ratio of 1 cm grain sphere and 100 µm grain is 10000. If 1 cm grain takes 18 hrs, 100 µm grain would take 0.1 min. In this case, 18 hrs was taken for 1 cm spherical grain because Karell and Gourishankar[15] reported 36 hrs for clad UO2 pellet and 12 hrs for crushed UO2 grains. Also, grain size of aggregate from AVP was considered conservatively as 100 µm. In fact, its grain size may be ~20 µm. Although there will be other factors for completing oxide reduction such as loading stating material, equipment setting, operation, equipment unsetting, reduction time can be definitely reduced. Afterwards, we need to carry out experiment to prove this theory.

Also, the behaviors of fission products should be known. It can be guessed that volatile fission products (Kr and Xe) can be released to off-gas line during electro-reduction process. However, tritium is considered to diffuse to salt phase. Further research on this is needed. And, behaviour of long-lived nuclides(I-129 and Tc-99) must be understand. Tc is considered to behave like other noble metals (Ru, Mo, Rh, Pd). And, iodine is considered to diffuse to salt phase and to form LiI. However, the information on the separation method of LiI from salt and disposal method seems to have not been published yet. Further research on this area is needed. On the other hand, in case of the ACV, as shown in Fig. A111, tritium is trapped on molecular sieve thru conversion from HT to HTO. And, technetium can be trapped by calcium based filter. Iodine is
trapped by Ag-X filter. In case of grains from mechanical slitting, cesium diffuses to salt phase. This salt containing cesium is mixed with zeolite and glass frits and be made to ceramic waste form of sodalite[25]. Recently, cesium is removed from salt by melt crystallization method[26] or zeolite adsorption method[27]. On the other hand, in case of the AVP, gaseous cesium is trapped by fly-ash filter at 800~1000℃. The trapped can be disposed of as the fly-ash filters in storage container. Or it can be made of fly-ash ingot.

2.9 Summary

The preceding effects, described to various degrees in the text, of voloxidation on the pyroprocess can be compared for the four cases and are shown in Table 1. A “1” indicates least effective or unfavorable while a “3” signifies most effective or beneficial. The total scores of case 3 is most high among 4 cases. This means that the goal of our project is right. Our INERI project has pursued particle size control, fission product removal, selective nuclide trapping, that is, PRESENT(Particle size control, fission product REMoval, SElective Nuclide Trapping) process. The PRESENT process will be very helpful for pyroprocessing.
Table 1.  Comparison of Voloxidation Effects on Pyroprocess

<table>
<thead>
<tr>
<th>Criteria</th>
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<th>Case 2</th>
<th>Case 3</th>
<th>Case 4</th>
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<td>Product Characteristics</td>
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<td>3</td>
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<td>3</td>
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<tr>
<td>Salt Carryover</td>
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<td>1</td>
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<tr>
<td>Fission Product Removal Rate/Timing</td>
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</table>
3. Path forward

3.1 Fission product removal

Experiment results of fission product removal from spent LWR fuel is promising shown in Table 2. Removal rate of Cs is 98%. Those of Kr, Xe, H-3, C-14, I-129 are 100%. And, those of Tc, Ru, Rh, Te, Mo and Rb are 92%, 98%, 83%, 53%, 62% and 96%, respectively. However, the temperature of 1200°C is still high in choosing proper material for voloxidizer. We introduced vacuum condition to prevent oxidizing inconel metal at 1200°C. So, we can manage to protect inside surface of voloxidizer. However, we did not protect the outer surface of voloxidizer. We need to operate voloxidizer under inert atmosphere.

Anyway, we need to lower the highest temperature by at least 100°C. Lowering 100°C is very important in maintaining the soundness of material over ~1000°C.

Table 2. Removal rates (%) of fission products by the PRESENT process.

<table>
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<th>H-3</th>
<th>C-14</th>
<th>Kr-85</th>
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</table>

3.2 Particle Size Control

The experimental results of particle size control of uranium oxide using SIMFUEL are also promising. Using 200gHM/batch rotary reactor, maximum 20 mm UOx particles were obtained. Pretest results of electroreduction of porous UO2 particles showed that uranium reduction rate was 99.6%. It is very good result.

However, operation of the rotary voloxidizer having one bearing system at high temperature was not good. So, we redesigned this voloxidizer to have two bearing system. The schematic diagram is shown in Fig. 2. This new voloxidizer will be operated within the end of 2009. Using this voloxidizer, optimum experimental condition for making good porous UOx particles will be obtained.

KAERI is going to make a large size rotary voloxidizer(25 kgHM/batch) to supply electoreduction process of PRIDE(PyRoprocessing Integrated DEmonstration) facility with porous UOx particles(SIMFUEL).
Verification using spent fuel should be needed. INL has an idea on the rotary voloxidizer. INL is going to modify the existed voloxidizer. So, if proper budget is given, verification using spent fuel would be performed.
3.3 Fission product trapping

Experimental results of the off-gas trapping in an INL hot-cell showed the feasibility on the selective trapping of Cs, Tc, I [28]. In order to increase trapping efficiency, new filters was developed and under performance testing. We need to confirm the trapping efficiency according to vacuum degree. Generally, if vacuum increases, removal rate of fission product increases and trapping efficiency of fission product decreases. So, we need to find out optimum vacuum degree, to compromise removal rate of fission product and trapping efficiency of fission product.

KAERI will also make large off-gas trapping unit (25 kgHM/batch) and test its performance in 2010-2011. Of course, large filter (~20 cm in diameter) will be manufactured and tested in the large off-gas trapping unit.
4. Conclusion

Case 3 (the low-temperature voloxidation taken to 1200°C under vacuum, so called advanced process) was chosen as the best option for head-end process of pyroprocessing. This justifies that the pursued direction (particle size control, fission product removal, selective nuclide trapping) of our project is right. INL and KAERI named our advanced voloxidation process as the PRESENT (Particle size control, fission product REmoval, SElective Nuclide Trapping) process. Both parties hope that the PRESENT process will be a very good present for pyroprocessing.

Also, the required research areas and path forward of our project are discussed in the areas of particle size control, fission product removal, fission product trapping, respectively.
References


2. S.D. Herrmann, personal communication (September 2008).


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**Title / Subtitle**

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**Publication Place**
Daejon

**Publisher**
KAERI

**Publication Date**
2009. 9

**Page**
24 p.

**Ill. & Tab.**
Yes ( ), No ( )

**Size**
A4.

**Note**

**Classified**
Open ( ), Restricted ( ), Name Class Document

**Report Type**
Technical Report

**Sponsoring Org.**

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**Subject Keywords**
(Voloxidation, Particle size control, Fission product removal, Fission product trapping, Selective trapping, Cs, Tc, I)
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전해환원 공정의 전처리공정으로서 휘발성산화공정이 KAERI 와 INL 에 의해 연구되고 있다. 이 보고서는 전처리공정의 4 가지 가능한 경우를 기술하고 전해환원속도, 생성물 특성, salt carryover 등의 항목으로 비교 평가하였다. 저온 산화 후 진공하 1200℃에서의 핵종제거 및 입도 제어하는 경우가 파이로 전처리공정으로 가장 좋은 것으로 평가되었다. 이것은 본 연구의 방향(입도제어, 핵종제거, 선택적 핵종 포집)이 옳다는 것을 나타낸다. 각 연구 분야별 공정 목표치가 검토 및 제시되었다. 주제명키워드 (10 단어내외) 휘발성산화공정, 입도제어, 핵분열생성물 제거, 핵분열 생성물 포집, 선택적 포집, Cs, Tc, I