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**ARTICLE**

# Influence of the Experimental Conditions on the Radiolytic Stability of *N, N, N', N'*-Tetraoctyldiglycolamide (TODGA)

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The stability of the extraction systems employed for the recovery of minor actinide involved in the advanced nuclear cycles is a limiting point for the development of several extraction processes. This work summarizes the efforts developed in recent years at the High-Level Waste Unit at CIEMAT to validate, from the point of view of its radiolytic stability, the use of the TODGA-based systems under development for the recovery of actinides through processes such as *i*-SANEX, EURO-GANEX and AmSel. Therefore, during years, several authors have studied TODGA resistance, and the implications of different experimental conditions on the radiolytic stability of TODGA have been explored. Factors such as the presence of oxygen, phase contact and metal complexation during the irradiation process have been recently published. However, other representative experimental conditions, such as the presence of a phase modifier and the irradiation procedure, have not been studied with a focus on TODGA stability. For that reason, this study aims to evaluate the effects of the presence of phase modifiers in the organic solvent as well as the irradiation procedure, to clarify the impact of each factor not only on TODGA degradation but also on the formation of its degradation compounds.

***KEYWORDS: advanced nuclear fuel reprocessing, minor actinides recovery, solvent extraction, TODGA stability studies, Radiolytic stability***

## I. Introduction

Research in nuclear fission technologies is advancing today towards more sustainable cycles, the advanced nuclear cycle, which involves scenarios where not only the recycling of uranium and plutonium is proposed, reusing more than 80% of natural uranium, but also the minor actinides (MAs) are considered. This strategy would significantly reduce the burden of High-Level Waste (HLW) long-term management and optimize the utilization of a final deep geological repository; but it also involves the development and industrialization of partitioning processes able to isolate different families of radionuclides.[1] The methodologies for separating radionuclides used for this purpose can be either pyrometallurgical (based on electrochemical separations in a saline medium at high-temperature) or hydrometallurgical (based on liquid-liquid extractions using selective extractants). The latter is currently considered the reference technology for demonstrating these advanced cycles due to the successful industrial application of the PUREX process.[2] Nevertheless, its industrial application implies to solve important challenges such as the degradation of the extraction systems due to the negative effects of the highly radioactive field and nitric acid concentration where nuclear fuel must be dissolved. In fact, to reach a high Technological Readiness Level (TRL) for such processes depends not only on the good extraction abilities but also on the demonstration of their robustness and the capability to predict their behavior in the long-term and the development of regeneration since they will run in continuous operation.[3]

In this context, in order to validate the use of these systems from the point of view of their industrialization, nowadays the advancement on simulation strategies of the radiation damage is crucial in assessing, comprehending, and forecasting the radiolytic effects that influence the performance of nuclear fuel reprocessing. The efforts of the HLW Unit at CIEMAT in this research are focus on the long-term prediction of the viability of designed extraction systems through the performance of stability studies. In recent years, an entire methodology for studying radiation resistance has been developed based on the correlation between observed behaviour changes and the system's composition after radiation. For this purpose, samples simulating relevant conditions of the process under study are typically exposed to external gamma radiation. After irradiation, among the parameters studied, such as changes in distribution coefficients (through alpha and gamma spectrometry, and Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) techniques), viscosity, pH, or the formation of third phases. In addition, special attention is given to the analysis composition of the samples through different techniques such as High-Performance Liquid Chromatography coupled with Mass Spectrometry (HPLC-MS) and Raman spectroscopy. This analysis focuses on the decrease in extractant concentration as a function of the received dose and the identification and quantification of the formed degradation compounds (DCs). These studies not only allow the characterization and optimization of the robustness of the systems but also help to identify their operational limits, potential causes of long-term mal-operation performance, and possible solvent regeneration

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strategies that enable efficient and safe continuous operation.

This work summarizes the efforts developed in the latest years in the HLW Unit at CIEMAT to address the degradation of a promising ligand for the actinides recovery such *N,N,N',N'*-tetraoctyldiglycolamide (TODGA),[4] which is involved in different relevant processes included in the advanced nuclear cycle like *i*-SANEX,[5] AmSel[6] and EURO-GANEX[7] processes, from fundamental stability studies to simple but reliable dynamic irradiation experiments.

The outcomes particularly focus on the stability of the main extractants (**Fig. 1**) used in the aforementioned processes, such as TODGA, without forgetting its interaction, depending on the process, with other important extractants like DMDOHEMA (*N,N'*-dimethyl-*N,N'*-dioctylhexylethoxymalonamide), $SO_3$-Ph-BTP (2,6-bis(5,6-di-(sulfophenyl)-1,2,4-triazin -3-yl)-pyridine), AHA (acetohydroxamic acid) or $SO_3$-Ph-BTBP (6,6'-bis(5,6-di(sulfophenyl)-1,2,4-triazin -3-yl)-2,2'-bipyridine).

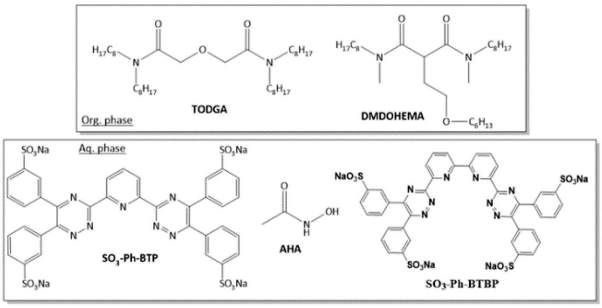

**Fig. 1** Structures of the main extractants involved in *i*-SANEX, AmSel or EURO-GANEX processes

Over the years, TODGA has been the subject of investigation by numerous researchers concluding that TODGA has demonstrated a satisfactory stability for several processes. However, its resistance has been found to depend on the experimental conditions.[8-15] Therefore, simulating real conditions in stability studies becomes imperative to accurately assess the resistance of solvents and determine their viability for industrial processes. Therefore, with time, the implications of different experimental conditions on the radiolytic stability of TODGA have been explored. Factors such as oxygen presence,[11] the phases contact,[12] metal complexation[15] and presence of a phase modifier such as octanol,[16] during the irradiation process have been recently published. However, other representative experimental conditions, such as the presence of a phase modifier such as DMDOHEMA ligand and the irradiation procedure, have not been studied focus on TODGA stability. For that reason, this study aims to evaluate the effects of presence of phase modifiers in the organic solvent as well as the irradiation procedure to clarify the impact of each factor.

## II. Experimental

### 1. Materials

The extractants used in the organic phase were *N,N,N',N'*-tetraoctyldiglycolamide (TODGA) and *N,N'*-dimethyl-*N,N'*-dioctylhexylethoxymalonamide (DMDOHEMA). TODGA was synthesized at CIEMAT modifying an existing literature procedure under air and without drying solvent and glassware, obtaining the product with a purity above 97% as confirmed by HPLC-MS.[17] DMDOHEMA with a purity of 99% by HPLC-MS was acquired from Technocomm Ltd. The diluent employed in the organic phase was odourless kerosene (OK) with a purity of 98%, obtained from Alfa Aesar. In the aqueous phase, in all studied cases, $HNO_3$ was used, and it was acquired from VWR chemical as nitric acid 65% with the grade AnalaR NORMAPUR for analysis, and it was additionally purified twice by a Quartz sub-boiling distillation system (MLS-Milestone) and used in a short period of time.

### 2. Irradiation Procedure

Irradiation experiments of the different samples were performed in the Náyade irradiation facility (CIEMAT).[18] This facility consists in a 1.2 $m^2$ by 4.5 m pool with 60 sources of $^{60}Co$. The irradiation container used provides homogeneous irradiation flux. The irradiations have been performed in two modes: in static conditions, where glass vessels are employed using a dose rate of 3.95 kGy/h, or in dynamic conditions using an irradiation loop device developed at CIEMAT[19] with a dose rate of 2.89 kGy/h.

### 3. HPLC-MS Analysis

The chemical composition of the irradiated organic samples was characterized by HPLC–MS. These measurements were performed using an HPLC–MS Bruker EVOQ™ (Triple Quadrupole detector) with an ACE 3 C18-PFP column (50 mm × 2.1 mm) at 40°C, employing a gradient of mobile phase [(A: 0.1% HCOOH in $H_2O$), (B: 0.1% HCOOH in $CH_3CN$)]. $APCI^+$ ionization mode was used for the quantification of TODGA, and $ESI^+$ ionization mode was used for DCs. Samples for HPLC–MS studies were analyzed without pre-evaporation and diluted 1:30,000 in HPLC-grade MeOH. Calibration curves were generated for TODGA, DMDOHEMA and each TODGA DC from 10 to 1000 ppb, with correlation coefficients in the range of 0.994–0.999 in all cases. All measurements were performed in duplicate to allow for uncertainty analysis, with results showing a maximum error of ± 3%.

## III. Results and Discussion

After irradiation, all samples were characterized by HPLC–MS to assess the TODGA resistance to gamma radiation under these conditions.

### 1. Effect of Addition of Phase Modifier on Stability of TODGA

To study the stability of TODGA, experiments were conducted using two types of solvents: one containing only TODGA (referred to as the TODGA solvent: 0.2 mol/L TODGA dissolved in OK) and another with a modifier, DMDOHEMA (referred to as the EURO-GANEX solvent: 0.2 mol/L TODGA + 0.5 mol/L DMDOHEMA in OK). Both solvents were irradiated up to 500 kGy in contact with an aqueous phase containing 0.5 mol/L $HNO_3$. **Figure 2** shows the concentration of TODGA as a function of the absorbed dose for the two mentioned solvents. At 200 kGy, the results indicate a higher concentration of TODGA in the TODGA solvent than in the EURO-GANEX solvent. However, at 500 kGy, which is an absorbed dose more representative of the stability studies, the degradation of TODGA was greater in the case of TODGA solvent (52% reduction in TODGA concentration in the TODGA solvent while a 43% reduction in the EURO-GANEX solvent). Therefore, these results suggest that the addition of phase modifiers plays a beneficial role in enhancing TODGA stability under irradiation conditions, and this is in agreement with published works found in the literature that have employed different phase modifiers.$^{20)}$

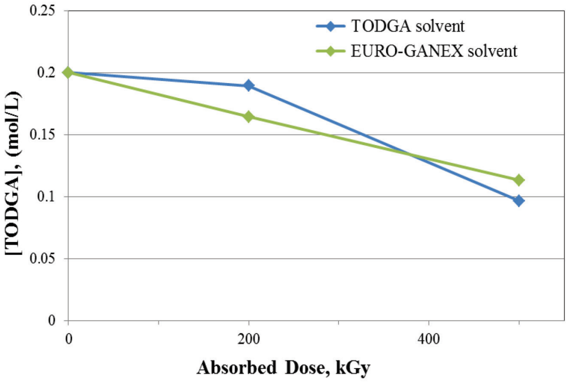

**Fig. 2** Concentration of TODGA as a function of absorbed dose for the TODGA solvent (0.2 mol/L TODGA in OK) and EURO-GANEX solvent (0.2 mol/L TODGA + 0.5 mol/L DMDOHEMA in OK) after irradiation in contact with 0.5 mol/L HNO3

To fully characterize the organic phases, the identification and quantification of possible DCs formed due to radiation were assessed. First, a qualitative study was carried out as a function of absorbed dose, revealing signals for 8 typical TODGA DCs, whose structures are shown in **Fig. 3**, and align with those reported in literature.$^{8\text{-}10)}$ In the EURO-GANEX solvent, in addition, 6 DMDOHEMA DCs were also identified, but the addition of DMDOHEMA does not appear to provoke changes in the TODGA degradation pathways during irradiation, as no new signals associated with TODGA DCs were observed.

Since the aim is to study the stability of TODGA solvent, this work is focused only on the quantification of TODGA DCs in both solvents. Thus, in each solvent studied, the main six DCs (DC I-VI) were quantified, and the results are illustrated in **Fig. 4**. In both cases, the most abundant DCs were IV, V and VI, which is expected due to the radiation effect on the weakest bonds in TODGA (C-O$_{\text{ether}}$ and N-C) and it is consistent with the literature.$^{7\text{-}11)}$

No significant changes in the TODGA degradation pathway were observed when DMDOHEMA was included in the solvent. This suggests that the addition of DMDOHEMA as a phase modifier does not alter the predominant degradation pathway of TODGA under irradiation conditions.

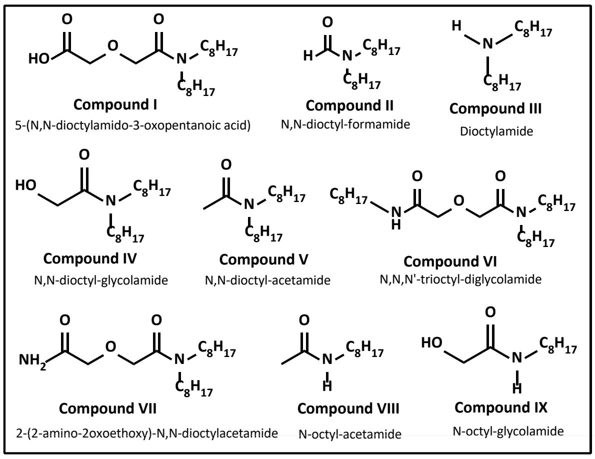

**Fig. 3** Structures of the main TODGA DCs

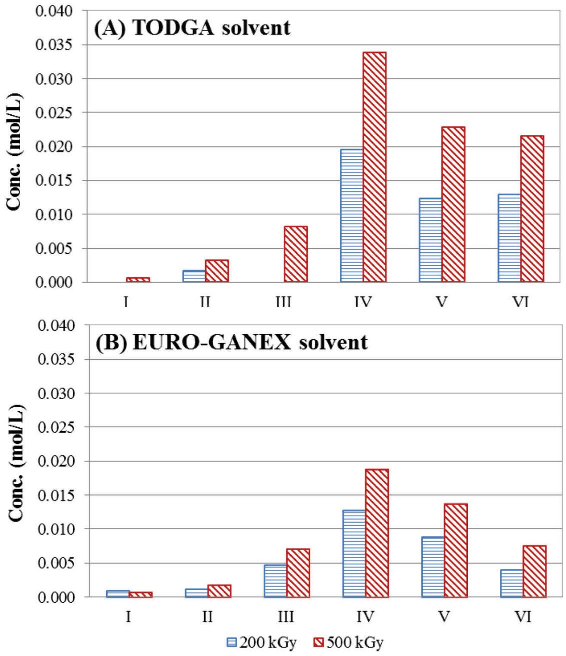

**Fig. 4** Quantification of different TODGA DCs for (A) TODGA solvent (0.2 mol/L TODGA in OK) and (B) EURO-GANEX solvent (0.2 mol/L TODGA + 0.5 mol/L DMDOHEMA in OK) after irradiation at 200 and 500 kGy in contact with 0.5 mol/L HNO3

## 2. Effect of Irradiation Procedure on Stability of Solvent

To study the potential implications of the irradiation procedure, the EURO-GANEX solvent (0.2 mol/L TODGA + 0.5 mol/L DMDOHEMA in OK) was irradiated up to 500 kGy in different modes, and the stability of solvents was compared. The first irradiation mode involved static irradiation conditions, which are typically used to gain a fundamental understanding of degradation pathways and provide an easy mode to compare the robustness of different ligands. The second mode was a dynamic irradiation, performed using an irradiation loop device that simulates different aspects included in the industrial extraction process, such as the continuous movement of phases as well as their contact during irradiation. In both cases, the organic solvent was irradiated in contact with an aqueous phase of nitric acid, with concentrations of 0.5 mol/L and 5 mol/L $HNO_3$ in the static and dynamic experiments, respectively.

The results of the TODGA and DMDOHEMA quantification in the studied cases are shown in **Fig. 5**. As expected, the concentrations of the extractants decrease as a function of dose. At 200 kGy, no significant differences were observed between the irradiation modes. Nevertheless, at 500 kGy, notable differences were detected in both irradiation modes, indicating less degradation of the solvents under dynamic conditions (43% for TODGA and 31% for DMDOHEMA in static conditions (at 0.5 mol/L $HNO_3$), compared to 25% for both compounds in dynamic mode (at 5 mol/L $HNO_3$)). However, it is essential to acknowledge the difference in nitric acid concentration, which may contribute to the observed differences. The protective effect of nitric acid on TODGA stability, as suggested by literature,[9,13)] may contribute in this higher stability observed, although controversial results can be found in literature about the effect of nitric acid in the stability of diglycolamides. Some studies report that nitric acid acts as a sensitizer, promoting the degradation of DGA.[21,22)] Conversely, other works suggest it has little to no impact,[8,10,23,24)] while some indicate a stabilizing, protective effect.[9,13,16)] In a recent study, Horne et al. examined the degradation rate of TODGA in the absence of an aqueous phase and in the presence of 3 mol/L $HNO_3$.[16)] Their findings showed that nitric acid provides a protective effect only when 1-octanol is present in the TODGA–dodecane organic phase. Therefore, based on these studies, this nitric acid effect is more pronounced when the organic diluent contains octanol, as it enhances the transfer of nitric acid from the aqueous phase into the organic phase, leading—according to the literature—to a stronger protective effect.[9,13,16)] In this case, since the organic diluent is solely kerosene (i.e., without octanol), significant nitric acid extraction into the organic phase is not expected. Therefore, the higher stability observed is likely related primarily to the irradiation mode, although nitric acid may also contribute.

Regarding the analysis of DCs, first in the qualitative study of the irradiated samples, significant changes were observed in the chromatograms obtained in the samples irradiated in both static and dynamic modes. In the samples irradiated in static conditions, the typical 8 TODGA DCs and 6 DMDOHEMA DCs were identified, meanwhile in the dynamic mode, three additional new signals have also been identified as potential undescribed DCs. The formation of these possible new DCs suggests that factors present in dynamic irradiation conditions, such as better contact between phases and higher oxygen content, may influence the degradation pathways of TODGA, which is in agreement with our previous works, where further details about these unkown DCs are described.[11,19)] This finding emphasizes the importance of experimental conditions on the stability of the solvent, as three new degradation compounds (DCs) were observed in this case. These compounds may exhibit different extraction properties compared to TODGA and could influence the extraction properties of the irradiated TODGA solvent.

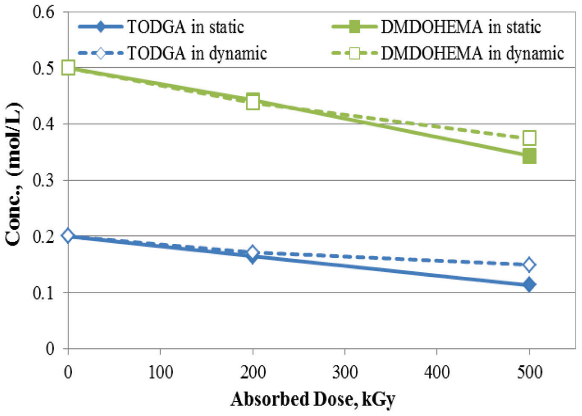

**Fig. 5** Concentration of TODGA and DMDOHEMA as a function of absorbed dose irradiated in static and dynamic mode, in contact with 0.5 mol/L $HNO_3$ (in the case of EURO-GANEX solvent in static mode) and 5 mol/L $HNO_3$ (in the case of EURO-GANEX solvent in dynamic mode)

**Figure 6** shows the quantification of the main TODGA DCs (DC I-VI in Fig. 3) obtained for the systems irradiated in static and dynamic conditions. As mentioned before, DCs IV, V and VI are typically the most abundant due to the effect of radiation on the weakest bonds in TODGA (C-$O_{ether}$ and N-C). In samples irradiated under static conditions, these DCs are predominant. However, in the case of irradiating under dynamic conditions, the results differ notably: although DC IV remains the most abundant, DC I and DC III increase

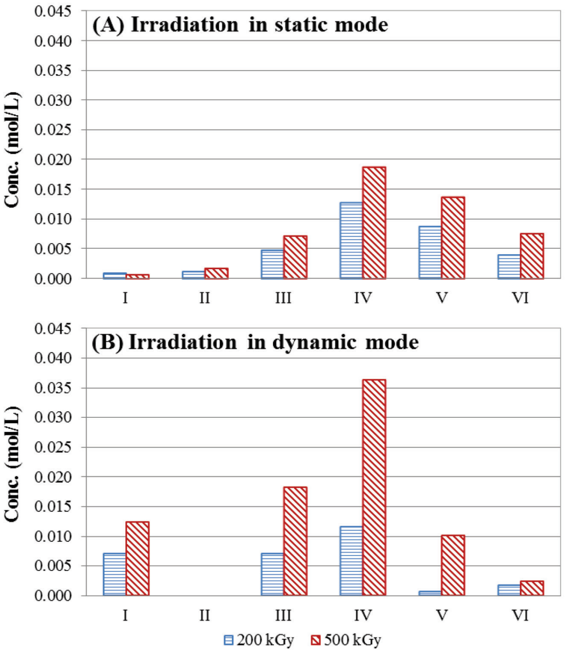

**Fig. 6** Quantification of different TODGA DCs for EURO-GANEX solvent when it has been irradiated (A) in static mode (in contact with 0.5 mol/L $HNO_3$) and (B) in dynamic mode (in contact with 5 mol/L $HNO_3$)

considerably in concentration, while DC IV and DC VI concentration decrease in this condition. This highlights the change in the degradation pathway producing the rupture of the $C_{carbonyl}$-N bond in more quantity using dynamic conditions than static conditions. These results are in concordance with our previous work,[11,19)] in which an air-sparging flow was used, suggesting that a higher oxygen content or improved phase mixing promotes a shift in the degradation pathway, favoring the production of DC I and III over DC V and VI. A higher proportion of these DCs (I and III) could be explained by hydrolysis degradation due to the differences in $HNO_3$ concentrations employed, being more abundant in the dynamic mode (5 mol/L $HNO_3$).

## IV. Conclusion

The evaluation of the experimental conditions on the radiolytic stability of TODGA was conducted to elucidate the impact of different effects, such as the addition of phase modifiers like DMDOHEMA in the organic solvent and the irradiation procedure (static or dynamic conditions). The focus was not only on the degradation of TODGA but also on its main degradation compounds (DCs).

The addition of phase modifiers, such as DMDOHEMA, in the organic solvent demonstrated a clear effect on TODGA stability, resulting in reduced degradation without altering degradation pathways since always the DCs are the same in both studied cases, those which came from the weakest bonds rupture in TODGA ($C$-$O_{ether}$ and N-C).

Regarding the irradiation procedure, dynamic experiments showed less TODGA and DMDOHEMA degradation in the EURO-GANEX solvent compared to static conditions. Additionally, the irradiation procedure affects TODGA degradation pathways, promoting also the rupture of the $C_{carbonyl}$-N bond.

Therefore, in this work, the impact of two significant effects for the TODGA stability have been assessed taking into account that in the industrial process, a mixture of different parameters is involved. Indeed, taking into account the combination of all these factors is crucial for understanding how they collectively impact the stability of solvents in real processes. Simulating real conditions in stability studies becomes imperative to accurately assess the resistance of solvents and determine their viability for industrial processes. This approach ensures that solvent systems developed in the laboratory are robust enough to withstand the challenges encountered in practical applications, ultimately contributing to the advancement and optimization of industrial processes.

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