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Technical Note

The effect of irradiation on hydrodynamic properties of extraction mixtures based on diamides of N-heterocyclic dicarboxylic acids in heavy fluorinated diluents

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ABSTRACT

Hydrodynamic properties have been investigated for promising extraction systems: 0.05 mol L^{-1} solutions of di(N-ethyl-4-ethylanilide) of 2,2′-bipyridine-6,6′-dicarboxylic acid, di(N-ethyl-4-fluoroanilide) of 2,6-pyridinedicarboxylic acid and di(N-ethyl-4-hexylanilide) of 2,2′-bipyridine-6,6′-dicarboxylic acid in meta-nitrobenzotrifluoride (F-3) or trifluoromethylphenyl sulfone (FS-13) diluents. To evaluate the perspectives for their use as extraction mixtures at the final stage of the nuclear fuel cycle, the change in density, viscosity, surface tension, and phase separation rate under irradiation with accelerated electrons was studied. The concentrations of extractants in the irradiated mixtures have been determined and the radiation-chemical yields have been calculated. Irradiation significantly decreases the phase separation rate at the stages of extraction and back extraction for all the studied systems. The viscosity of the DYP-7 solution in FS-13 increase above the values suitable for its use in extraction processes.

1. Introduction

Extraction processes is a basis of modern technological schemes of the spent nuclear fuel (SNF) reprocessing. One of the promising technologies of the SNF reprocessing is the UNEX process [1] based on the extraction mixture of chlorinated cobalt dicarbollide (CCD), carbamoyl phosphine oxide (CMPO) and polyethylene glycol (PEG) in a polar diluent trifluoromethylphenyl sulfone [2]. The advantage of this process is the possibility of collective extraction of long-lived radionuclides (cesium, strontium and minor actinides) from highly acidic (up to 6 mol L^{-1} HNO_3) solutions – liquid high-level waste (HLW). The disadvantages of the classical UNEX process are associated with the presence of phosphorus atoms in the composition of the CMPO. As an alternative to CMPO, diamides of N-heterocyclic dicarboxylic acids have been proposed [3–6]: N,N′-diethyl-N,N′-di(4-hexyl-phenyl)-diamide of 2,2′-dipyridyl-6,6′-dicarboxylic acid (DYP-7), N,N′-diethyl-N,N′-di(4-ethyl-phenyl)-diamide of 2,2′-dipyridyl-6,6′-dicarboxylic acid (DYP-9), N,N′-diethyl-N,N′-di(4-fluorophenyl)-dipicolylamide (Et(pFPh)DPA). They can be used in extraction systems with CCD. This fact together with their high chemical stability makes it possible to consider them as promising extractants in the HLW treatment in a modified UNEX-process.

Among many fluorinated ethers and esters, ketones and sulfones, trifluoromethylphenylsulfone (FS-13) and meta-nitrobenzotrifluoride (F-3) have been chosen as promising diluents because the first one is the main diluent in the UNEX process [7], and the second underlies one of the first processes of co-extraction of ^{137}Cs and ^{90}Sr, the so-called DCC process [8].

During technological operations, the extraction mixture is irradiated, and this results in the formation and accumulation of products of radiation-chemical decomposition of the extractant and diluent, which leads to worsening of hydrodynamic properties and selectivity of the extraction system. Although the circulating extractant is constantly regenerated in the extraction cycle by washing it with soda, some of the degradation products are not removed and are gradually accumulated [9–12].

In our previous works [13–16], we have studied the radiation-thermal stability of both pure diluents and solutions of new extractants based on diamides of dicarboxylic acids in heavy fluorine-containing diluents. Their high thermal stability was determined as acceptable for practical use in the extraction processes of HLW fractionation.

The main products of FS-13 radiolysis are: hexafluoroethane

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(G_p = 2.2 molecules/100 eV), phenylsulfonic acid (Gp = 2.1 molecules/100 eV) and hydrogen (G_p = 0.6 molecules/100 eV), which contains up to 5% by volume of methane and ethane [13]. When FS-13 was irradiated in a mixture with 7 mol L^{-1} of nitric acid, trifluoronitromethane (G_p = 1.2 molecules/100 eV) and nitrobenzene (0.2 molecules/100 eV) were detected in the radiolysis products, as well as gases NO, N_2O and CO, in addition to hexafluoroethane and phenylsulfonic acid.

Analysis of unirradiated DYP-7 diamide after heating to 202 °C by the methods of mass spectrometry, chromatography and UV spectroscopy, showed that it contains three compounds [14]: the original DYP-7; the product of its thermal diethylation – di-N,N,-(4-hexylphenyl) amide 2,2′-bipyridyl-6,6′-dicarboxylic acid; product of further dearylation/reduction – 6,-formyl-N-(4-hexylphenyl)-2,2′-bipyridyl-6-carboxamide. The IR spectra of the original DYP-7 treated with nitric acid did not contain features of NO_3^- ions and the nitro groups of the organic compounds that indicates the absence of a noticeable interaction of DYP-7 and nitric acid.

The formation of aldehyde functional group during the thermolysis of amides was studied by the example of the decomposition of polyamide fibers [17,18]. When they are thermally degraded, first of all, there is a loss of ethyl groups of the amide fragment. The observed N-dealkylation is an analogue of free-radical oxidative dealkylation of tertiary amides under the action of peroxydisulfates [19]. Electron donor atoms in amide groups increase the electron density on the nitrogen atom that results in increase of the stability of the corresponding radical. On the contrary, presence of acceptor atoms decreases the stability of the radical [20]. This explains the difference in thermal processes, accompanying the decomposition of diamides, carrying electron-donor (DYP7 and DYP-9) and electron-acceptor (Et(pFPh)DPA) substituents.

Previous studies [21,22] showed that the main process of radiation decomposition of DYP-7 and DYP-9 in an FS-13 diluent is radiation-stimulated hydrolysis with the formation of the corresponding monoamides. The radiolytic decomposition products of diamide Et(pFPh)DPA were not detected, probably because of their neutral nature. Apparently, in contrast to DYP-7 and DYP-9 diamides, the decomposition of diamide Et(pFPh)DPA under irradiation goes primarily in the direction of the formation of nitriles, not free carboxylic acids.

The quality criteria of the circulating extractant in technological operations are the magnitude of changes in: the capacity of the extraction solution, its selectivity and hydrodynamic properties in various operations of processing the extractant with acidic and alkaline aqueous solutions. The decrease in the capacity of the extraction system is caused by decrease in the concentration of the extractant due to its chemical and radiation decomposition, as well as physical losses in aqueous solutions and gas streams. The radiation loss of the extractant depends on the dose of radiation and can be expressed as the radiation-chemical yield of the extractant decomposition. For the considered extraction mixtures such information is missing in the literature. Therefore, for the final conclusion about the possibility of the safe use of new extractants based on diamides of dicarboxylic acids, it is necessary to study their radiation-chemical resistance and changes in their main hydrodynamic properties under irradiation, namely: the density and viscosity of the extractant, the surface tension and the phase separation rate.

The objective of the present work is to determine the radiation stability of extraction mixtures based on the aforementioned diamides solutions in heavy fluorinated polar diluents: meta-nitrobenzotrifluoride (F-3) and trifluoromethylphenyl sulfone (FS-13). The structural formulas of the studied extractants and diluents are presented in Figs. 1 and 2.

2. Experimental

During technological operations, the extraction mixture is affected by ionizing radiation resulting in the change of its characteristics. Particular emphasis should be placed on the hydrodynamic parameters of the irradiated mixtures, such as density and viscosity of the extractant, phase separation rate. In present work we have used a special technique that has been developed for the determination of these parameters [23,24]. The experimental relative error of the parameters does not exceed 5%. The diamides of 2,2′-bipyridine-6,6′-dicarboxylic acid have been synthesized at the Department of Chemistry of Lomonosov Moscow State University [25], other reagents were obtained from commercial sources and were used without further purification.

Since β-radiation is the main contributor to the energy released by fission products during prolonged exposure [9, p.9], the ionizing radiation was simulated by irradiating the studied mixtures with accelerated electrons. For this purpose we have used the linear accelerator UELV-10-10-S-70 (energy of electrons 8 MeV, pulse duration 6 μs, pulse repetition frequency 300 Hz, average beam current 700 μA, vertical frequency 1 Hz, scan width 245 mm). The irradiation experiments were performed in a cylindrical glass reactor equipped with a water seal and rotated on a carousel-like

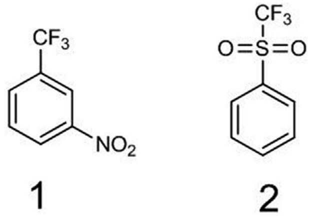

Fig. 2. Diluents: 1 – meta-nitrobenzotrifluoride (F-3), 2 – trifluoromethylphenyl sulfone (FS-13).

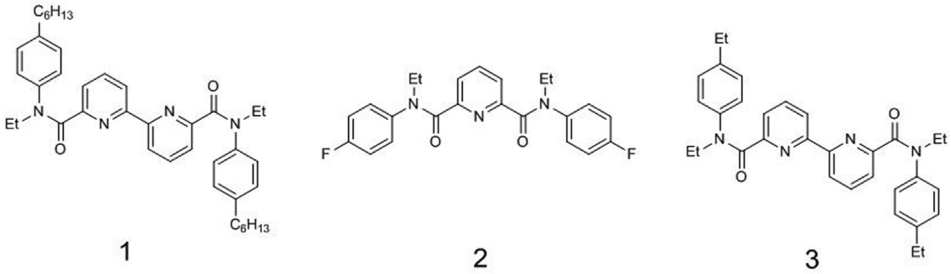

Fig. 1. Extractants: 1 – di(N-ethyl-4-hexylanilide) of 2,2′-bipyridine-6,6′-dicarboxylic acid (DYP-7), 2 – di(N-ethyl-4-fluoroanilide) of 2,6-pyridinedicarboxylic acid (Et(pFPh)DPA), 3 – di(N-ethyl-4-ethylanilide) of 2,2′-bipyridine-6,6′-dicarboxylic acid (DYP-9).

table with a velocity of 16 cm s^{-1}. The electron beam was moving along the vertical axis of the vessel with an extractant solution in the diluent. Extraction mixtures were saturated three times within 20 min with equal volumes of 3 mol L^{-1} HNO_3 before irradiation. One pass of the electron beam creates a dose of 10 kGy in a sample. Samples with absorbed doses of 100, 250 and 500 kGy were prepared by variation of the number of passes.

Plates of a copolymer with a phenazine dye produced by FSUE "VNIIFTRI" were used for dosimetry of accelerated electrons. Doses were determined from the difference in optical density of irradiated and non-irradiated plates measured with a spectrophotometer at wavelength $\lambda = 512$ nm using a calibration table.

After irradiation, samples of extractant solutions in the diluent were contacted with 3 mol L^{-1} HNO_3 solution imitating an extraction stage of elements. Then the organic phase was shaken twice (each time for 10 min) with equal volumes of 0.02 mol L^{-1} HNO_3 solution imitating a back-extraction stage. After back-extraction, the organic solution was regenerated by shaking it three times (each time for 10 min) with equal volumes of 0.5 mol L^{-1} Na_2CO_3 solution. Every time a new solution of nitric acid or sodium carbonate was used. After the regeneration stage, samples of 2 mL of both the organic and aqueous phases were taken for chromatographic analysis. The time of separation of the aqueous-organic emulsions was also measured. Separation rates were calculated for every contact of the phases — v1, v2, and v3, respectively. At all stages of the regeneration of the organic phase, its density, viscosity and interfacial tension were determined at 20 °C.

The phase separation rate was calculated as the ratio of the height of the organic phase layer (at equal phase volumes) to the time of complete phase separation (mm s^{-1}). The surface tension at the boundary “organic phase-air” was determined with a KSV Minitrough™ (Finland) feedback device, equipped with automated Langmuir weights and a Wilhelmi platinum plate with a perimeter of 49 mm. The dynamic viscosity of the samples was calculated by the Poiseuil equation:

$$\eta = \eta_0 \frac{\rho \cdot t}{\rho_0 \cdot t_0}$$

where η_0, ρ_0, t_0 are characteristics of distilled water.

The degradation products of the extraction mixtures were analyzed with liquid chromatography (Agilent 1260) with a UV detector and a mass-spectrometric detector Bruker maxis impact with possible modes of chemical ionization, electrospray, and photoionization. For the analysis, samples of 10 μL were taken and diluted up to 1 mL with ethanol. The diluted mixtures were treated in an ultrasonic bath for 15 min. Then samples of 10 μL were again taken and diluted up to 1 mL with ethanol. The volume of the injected sample was 1 μL. Analytical procedure: start — 95% water and 5% acetonitrile, gradient up to 30 min to 5% water and 95% acetonitrile, then isocratic — 20 min.

3. Results and discussion

All the studied diamides are soluble in the F-3 and FS-13 diluents at a concentration of 0.05 mol L^{-1}, the final solutions are homogeneous colored liquids. Density values changed slightly with irradiation for all the extractants in both diluents (F-3, FS-13), that indicates their radiation stability (Fig. 3).

The surface tension and viscosity of the extraction mixtures are more relevant parameters for assessing the hydrodynamic characteristics as compared to the phase separation rates. The latter depends on the diameter of the mixing vessel, mixing rate, phase ratio and other parameters. The viscosity of extractant solutions in F-3 diluent increases after irradiation to 250 kGy (Fig. 4), then decreases. This may be explained by the accumulation of intermediate degradation products and their subsequent destruction. In the FS-13 diluent, a smooth increase in viscosity values was observed with increasing the irradiation dose, and they are significantly higher the viscosity values of the solutions in diluent F-3 under similar conditions, especially for the extraction mixture with DYP-7. This eliminates the possibility of using these mixtures under intensive irradiation conditions that are relevant for real reprocessing of liquid HLW.

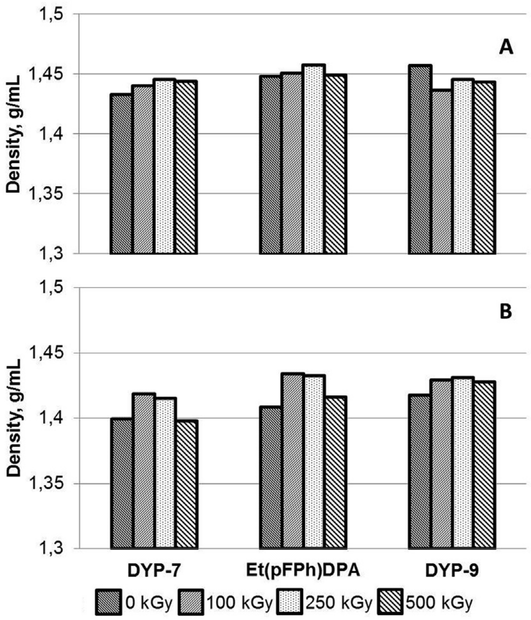

Fig. 3. Density values of the extraction mixtures in the F-3 (A) and FS-13 (B) diluents after irradiation to absorbed doses of 100, 250 and 500 kGy.

The rate of diffusion processes during extraction is reciprocal to the magnitude of the surface tension. The surface tension of the

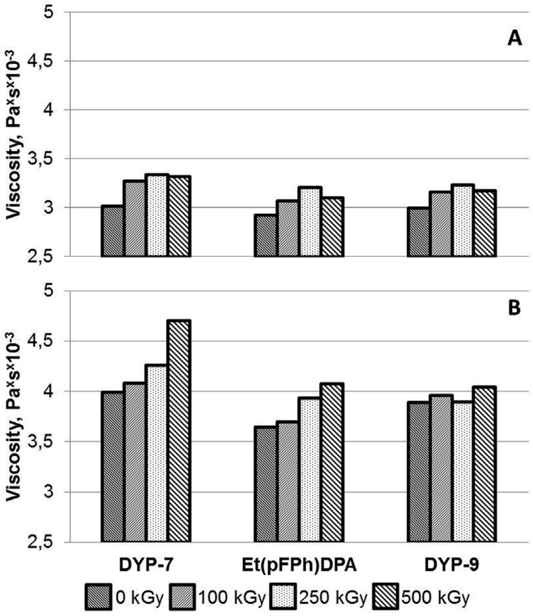

Fig. 4. Viscosity values of the extraction mixtures in the F-3 (A) and FS-13 (B) diluents after irradiation to absorbed doses of 100, 250 and 500 kGy.

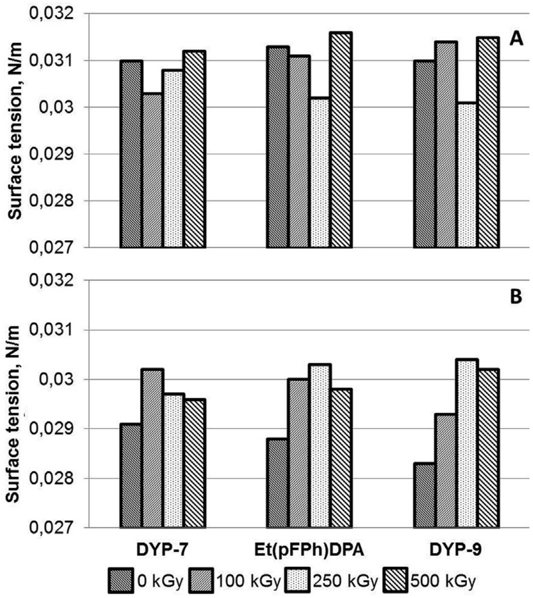

Fig. 5. Surface tension values of the extraction mixtures in the F-3 (A) and FS-13 (B) diluents after irradiation to absorbed doses of 100, 250 and 500 kGy.

solutions of the diamides of dicarboxylic acids in the F-3 diluent varies slightly with irradiation dose, and its average value is about 5% higher than that in the FS-13 diluent (Fig. 5).

The dependences of the phase separation rates of the mixtures for all three stages (extraction, back-extraction, regeneration) on the irradiation dose are shown in Figs. 6–8. The phase separation rate at the extraction stage decreases with increasing of the irradiation dose for all the mixtures (Fig. 6), the effect is particularly significant for DYP-7 diamide in the FS-13 diluent. The average decrease in the phase separation rate at a dose of 500 kGy is about 50% relative to unirradiated systems.

For diamide DYP-9, the separation rates have an exponential dependence. Unirradiated DYP-7 diamide in the FS-13 diluent has the phase separation rate 2 times higher than for other investigated diamides. A sharp decrease of the phase separation rate of this mixture during irradiation (Fig. 6B) is probably due to its degradation and significant chemical changes.

For systems with the F-3 diluent, the separation rates at the extraction stage are higher compared with similar systems in the FS-13 diluent, which is explained by higher surface tension values of the mixtures with the F-3 diluent. At the back-extraction stage, the phase separation rates for all the investigated diamides in both the F-3 and FS-13 diluents have lower values compared to the same systems at the extraction stage (Fig. 7). The values of the separation rates decrease with increasing dose of irradiation due to the accumulation of degradation products which presence increases the viscosity of the corresponding mixtures. In average, the phase separation rates at the absorbed dose of 500 kGy were 40% lower compared to the unirradiated samples. Like at the extraction stage, unirradiated DYP-7 diamide in the FS-13 diluent has the phase separation rate 2 times higher than for other investigated diamides.

At the stage of regeneration of the irradiated samples, especially at the absorbed dose of 500 kGy, the organic and aqueous phases were barely separated due to the presence of large amounts of precipitation. It was possible to isolate the pure organic phase only in several hours. In average, the phase separation rates at this stage for all the investigated systems were 5 times lower than at the extraction stage.

The phase separation rates varied significantly for different treatment procedures of the solutions, especially for the washing with soda solutions. This can be explained by the formation of acidic degradation products. Formation of emulsions was observed

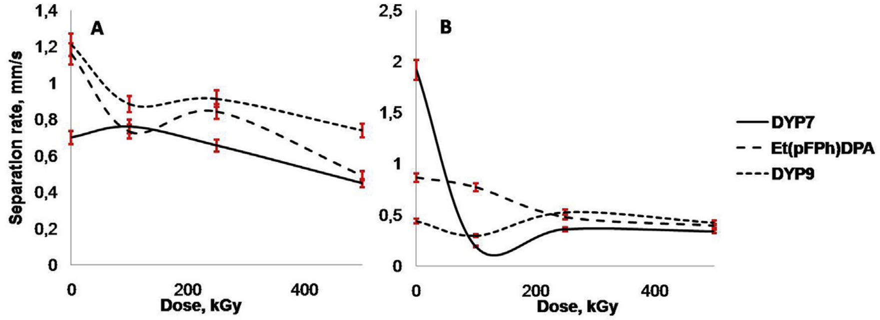

Fig. 6. The effect of irradiation dose on the phase separation rate at the extraction stage: A – in the F-3 diluent, B – in the FS-13 diluent.

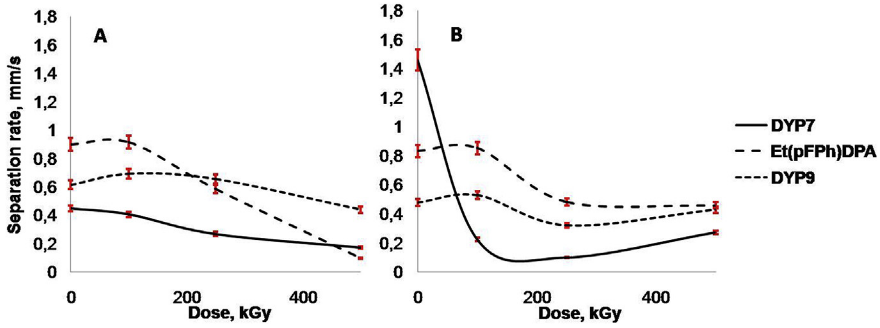

Fig. 7. The effect of irradiation dose on the phase separation rate at the back-extraction stage with 0.02 mol L^{-1} HNO_3 solution: A – in the F-3 diluent, B – in the FS-13 diluent.

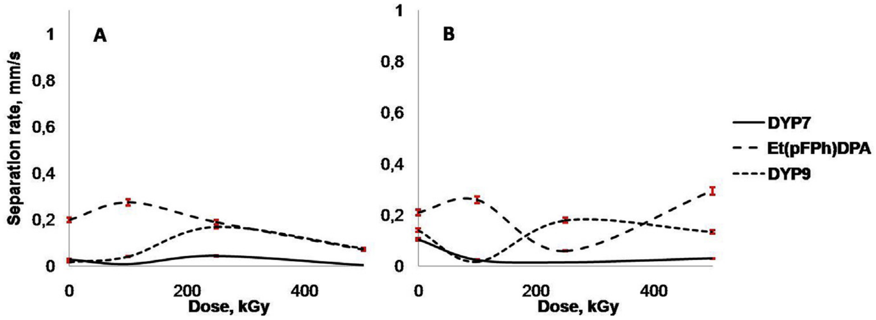

Fig. 8. The effect of irradiation dose on the phase separation rate at the regeneration stage with 0.5 mol L^{-1} Na_2CO_3 solution: A – in the F-3 diluent, B – in the FS-13 diluent.

in the case of DYP-7 and DYP-9 diamides containing alkyl substituents in the aromatic ring. This can be associated with structural changes of alkyl substituents under irradiation and leads consequently to an increase in dynamic viscosity of these systems.

Degradation products of the F-3 and FS-13 diluents accumulated in the extraction systems with the increase of the absorbed dose. Composition of the main degradation products was established earlier by liquid chromatography [21]. Some of these compounds are washed out during different treatments of the solutions, but cyclic F-containing and dimeric aromatic products are able to accumulate in the system, which results in a quality decrease of the extraction mixtures. Systematic monitoring of the concentration of the extractant in the irradiated samples after soda treatment is mandatory during operation of radiochemical plants (RCP). This data is necessary to confirm the performance of the processes, and also serves as a criterion for evaluating the effectiveness of operations for the regeneration of the extraction solution. The extractants degradation products have been not detected by liquid chromatography in the irradiated samples after treatment with soda solutions. Data on the extractants concentration depending on the absorbed dose are presented in Table 1. Based on these results, the radiation-chemical yields of decomposition of the studied extractants have been calculated (Table 2).

Thus, DYP-9, due to its chemical nature, demonstrates the lowest radiation stability in both the FS-13 and F-3 diluents. Diamide Et(pFPh)DPA decomposed significantly in the dose range of 250–500 kGy in the FS-13 diluent. This may be due to the accumulation of degradation products, which can form reactive radicals under irradiation that, in turn, leads to the destruction of this extractant. It should be noted that diamide Et(pFPh)DPA demonstrates high radiation stability in the F-3 diluent.

Table 1
The degree of degradation of the diamides of dicarboxylic acids depending on the absorbed dose.

Diamide	Degradation degree, %					
	100 kGy		250 kGy		500 kGy	
	F-3	FS-13	F-3	FS-13	F-3	FS-13
DYP-7	10.4	24.4	21.5	27.6	51.7	46.6
Et(pFPh)DPA	13.3	14.8	22.1	25.6	41.1	74.6
DYP-9	14.5	56.4	25.2	74.1	55.7	85.6

Table 2
Radiation-chemical yields of decomposition of the diamides of dicarboxylic acids depending on the absorbed dose.

Diamide	G, molecule per 100 eV					
	100 kGy		250 kGy		500 kGy	
	F-3	FS-13	F-3	FS-13	F-3	FS-13
DYP-7	0.32	1.03	0.26	0.46	0.32	0.36
Et(pFPh)DPA	0.47	0.23	0.31	0.24	0.29	0.45
DYP-9	0.46	2.04	0.32	1.02	0.35	0.61

4. Conclusions

The effect of irradiation on the properties of extraction systems based on three diamides of dicarboxylic acids in the heavy polar fluorinated diluents F-3 and FS-13 has been investigated. In the case of the FS-13 diluent, irradiation of the extractants containing alkyl substituents in the aromatic ring leads to the formation of emulsions during treatment of irradiated mixutres with soda solutions. Dynamic viscosity of the emulsions can reach values close to $4.0 \cdot 10^{-3}$ Pa s. The maximum viscosity values have been measured for the extraction mixture with DYP-7 containing hexyl substituents. The surface tension values at the phase boundaries are comparable for all three investigated diamides after irradiation and do not depend on the type of diluent. Increase of the absorbed dose results in an increase of the density and viscosity of the extraction mixtures, which may indicate the formation of dimerization products and recombination of fragments of diluent and extractant molecules, as well as the formation of radicals of degradation products. The increase of the density and viscosity of the extraction mixtures results in a sharp slowdown of the phase separation, especially at the regeneration stage.

The concentrations of extractants and radiation-chemical yields of their degradation depending on the absorbed dose have been determined. Diamide DYP-9 in the FS-13 diluent demonstrates the lowest radiation stability due to the nature of this extractant, while diamide Et(pFPh)DPA in the F-3 diluent decomposed by only 41% at the absorbed dose of 500 kGy. These results can be served as a basis for recommending the possible use of these extraction mixtures in real radiochemical production.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.net.2019.02.009.

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