252

S. D. Dimović, *et al.*: Leaching Kinetics of Co(II) and Sr(II) Contaminated Soil ...
Nuclear Technology & Radiation Protection: Year 2018, Vol. 33, No. 3, pp. 252-259

LEACHING KINETICS OF Co(II) AND Sr(II) CONTAMINATED SOIL VIA CHEMICAL EXTRACTION METHOD

by

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Artificially contaminated soil, with mixture of Co(II) and Sr(II) ions in different proportions, was tested. The main aim was to investigate the possibility of ethylenediaminetetraacetic acid (EDTA) utilization in the chemical extraction process as a method for soil remediation in the case of mixed contamination. The efficiency of this process when EDTA of $5 \cdot 10^{-4}$ molL^{-1} was used was low, while the application of the EDTA solution of the initial concentration $5 \cdot 10^{-3}$ molL^{-1} or higher showed almost the same efficiency. This suggested that the application of the solution EDTA ($5 \cdot 10^{-3}$ molL^{-1}) is optimal from the aspect of cost and degradation of soil properties. Furthermore, process kinetic was fitted using one parameter shrinking core models. Kinetic data modeling showed that the process is complex, demanding multi resistance kinetic model.

Key words: chemical extraction, kinetics, contamination, modeling

INTRODUCTION

Terrestrial radionuclides are naturally presented in soils as minerals and rocks constituents. They are long-lived isotopes where the most important and most abundant are isotopes formed by ^{238}U or ^{232}Th radioactive decay. As a consequence, natural radioactivity of soil originates mainly from ^{226}Ra, ^{232}Th, and ^{40}K, with the world averaged values of 32 Bqkg, 45 Bqkg, and 420 Bqkg, respectively [1]. In addition to the natural radionuclides, various artificial radioactive isotopes may be transferred to the environment. Thus, soil contamination with radionuclides can occur due to the accidental release after nuclear accidents, nuclear weapon testing, under leakage of radionuclides during waste processing, transport, or permanent disposal. These activities provoke significant concentrations of other contaminants like Co, Sr, Cs, Ni, Am, and other isotopes. In USA, ^{237}Cs, ^{226}Ra, ^{238}U, ^{60}Co, ^{90}Sr, ^{232}Th, and $^{238-242}$Pu are denoted as the most important radionuclides in the contaminated soils [2].

Generally, the remediation of contaminated soils using different techniques like phytoremediation, soil washing, soil stabilization and, chemical extraction, is applied [3]. The only efficient way for remediation of radioactive ions contamination is removal of isotopes from soil matrices. Chemical extraction is applied using chemical reagents which dissolve, desorb, contaminants form chelates and complex compounds. Likewise, different chemicals like inorganic and organic acids, complexing agents, inorganic salts, oxidizing agents, *etc.* [4], even commercial products like Coca Cola can be utilized for this method [5]. The comparison between efficiencies of inorganic acid and salt, as well as, complexing agent for pollutant removal [6], showed that inorganic chemicals were less effective. Furthermore, it has to be noticed that application of aggressive inorganic acids provokes soil components dissolution and leads to decrease of soil quality and productivity. Among chelating agents, the most extensive research was done with the utilization of ethylenediaminetetraacetic acid (EDTA) [7], but also with nitriloacetic acid (NTA) [8, 9] and, diethylenetriamine pentaacetic acid (DTPA) [5]. The efficiency of this process is highly influenced by soil properties, contaminant properties, and operating conditions. Soil and contaminant types define the total amount of contaminant content and distribution, where these parameters are specified and fixed for a certain scenario. On the other hand, operating conditions like reagent type, concentration, pH value and volume, treatment duration, *etc.* are adjustable and variable, submissive to optimization. The investiga-

S. D. Dimović, *et al.*: Leaching Kinetics of Co(II) and Sr(II) Contaminated Soil ...
Nuclear Technology & Radiation Protection: Year 2018, Vol. 33, No. 3, pp. 252-259

253

tion of operating variables was given for the soil contaminated with heavy metals [7, 10, 11] and radionuclides [12-14]. The effectiveness of extraction with differently concentrated solutions like HCl, $CaCl_2$, EDTA (in the form of Na salt), tartaric and citric acid, for decontamination of soils containing Co^{2+} and Sr^{2+} ions in the excessive amounts, showed that the most effective were EDTA solutions for both cations. On the contrary, $CaCl_2$ solution exhibited highest efficiency considering solely Sr(II) contamination [14]. However, these experiments were performed for contact time of 1 hour and process kinetics was not investigated. Consequently, the major goal of this study was to investigate kinetics of Co and Sr leaching process from artificially contaminated soil. The information about process kinetics has both scientific and practical significance and is beneficial for system design or process intensification.

EXPERIMENTAL PART

Soil contamination

Representative soil sample, from the location of Vinča Institute of Nuclear Sciences, was used in this investigation. This location was chosen because of soil radioactive contamination possibility at this site. The sampling procedure and characterization of this soil sample can be found elsewhere [15]. It was shown that the sample was weakly alkaline with low phosphorus content and low cation exchange capacity, mainly consisted of quartz, kyanite, muscovite, albite, calcium, magnesium carbonate, *etc*.

The selected soil was artificially contaminated using aqueous solutions of Co(II) and Sr(II) nitrate salts. Due to the fact that chemical behavior of stable and radioactive isotopes is the same, in this study elements in stable form were used. Soil contamination was completed with two different solutions (denoted as S1 and S2) containing cations in different proportions with total constant concentration of 0.0045 $molL^{-1}$:

– solution S1 contained Co(II) and Sr(II) ions in molar ratio 1:2, and
– solution S2 contained these cations in molar ratio Co:Sr = 2:1.

One hundred grams of uncontaminated soil was mixed with the prepared solutions, in the solid/liquid ratio 1:10. After a period of 10 days, the suspensions were centrifuged in order to separate solid and liquid phases. Solid phases were dried in the air atmosphere, at room temperature, while liquid phases were used for determination of residual metals' concentrations. The differences between exact initial concentrations of Co(II) and Sr(II) ions in stock solutions and their concentration in the filtrates after soil contamination, can be utilized to determine the sorbed amounts of investigated cations onto soil.

Extraction kinetics

Dried solid residues were utilized in further experiments for investigation of chemical extraction procedure. Differently concentrated solutions of EDTA (5 10^{-4}, 5 10^{-3}, 5 10^{-2}, and 0.1 $molL^{-1}$) were prepared in distilled water. The initial pH values of EDTA solutions were around pH = 3. Chemical extraction process *i. e.* leaching of contaminants from the polluted soil, was prepared by mixing 0.5 g of contaminated soil with 20 mL of leaching solution in PVC flasks. The batches were shaken on the rotary shaker (10 rpm agitation), by varying contact times between 15 minutes and 48 hours, in order to obtain kinetic curves. The suspensions were centrifuged (9000 rpm, 10 minutes) and metals' concentrations in the liquid phases were measured.

Also, pH values of the filtrates were determined using WTW InoLab pH-meter.

Measurements of metals' concentrations in liquid phases (stock solutions for contamination, the filtrates after soil contamination and filtrates after extraction) were performed by flame atomic absorption spectrometer Perkin Elmer 3100. Concentration of Co(II) was determined at wavelength of 241.2 nm, while Sr(II) was determined at 460.7 nm.

Leaching experiments were exhibited in duplicate and the results are presented as mean values between replicates.

RESULTS AND DISCUSSION

Soil contamination

The interaction between contaminants was established through different mechanisms like ion-exchange, electrostatic interactions, specific sorption, surface precipitation and structural incorporation. The processes occurred are very complex since interactions can be established between metal contaminant and water, minerals, organics, microorganisms and gasses as constituents of soil matrix [16].

Results of contamination test showed that the majority of applied contaminants remained bonded *i. e.* sorbed to the soil matrix. Thus, the application of S1 solution with twice higher molar proportion of Sr(II) than Co(II) showed that 73 % of Sr (II) and 90 % of Co(II) was sorbed. This contaminated soil was denoted as SS1. Otherwise, 73 % of initially contained Co(II) and 49 % of Sr(II) in the stock solution S2 was sorbed after mixing with soil, making the contaminated soil SS2. These results indicated relative high affinity and sorption capacity of the investigated soil sample towards the chosen pollutants. Recalculating, it can be concluded that sorption capacity of 1 g of dried soil sample was 1.71 mg Co(II) and 3.01 mg Sr(II) after contact with solution S1 and 3.07 mg

254

S. D. Dimović, *et al.*: Leaching Kinetics of Co(II) and Sr(II) Contaminated Soil ...
Nuclear Technology & Radiation Protection: Year 2018, Vol. 33, No. 3, pp. 252-259

Co(II) and 1.38 mg Sr(II) after contact with S2. The obtained capacities were lower than the maximum sorption capacity of this soil type, obtained for single solute sorption, which were found to be 6.62 mg Sr(II) per gram and 5.43 mg Co(II) per gram [14]. Measured pH values of the filtrates after contamination were found to be 7.2 0.1, regardless of the applied solution. Since the measured pH values of uncontaminated soil in water suspension was around 7.9 [15], the lower pH values after contamination can be attributed to the inner-sphere complexes formation between surface groups of soils and metal cations. This bonding mechanism was already approved and found to be significant for Co(II) cations sorption [14, 17].

Leaching kinetics

Testing the leaching kinetics under described conditions gave different results, dependent on applied EDTA solution (fig. 1). Generally, leached amounts of contaminants increased with time. Moreover, increase of EDTA concentration provoked more intensive leaching from contaminated soil. The obtained curves represent two-step processes: the first, rapid stage at the beginning and the second, subsequent gradual release of the contaminants from the solid to the liquid phase.

Process efficiency expressed as percentages of removed cations is presented in fig. 2.

Desorption of Co(II) from SS1, using the most diluted solution, achieved equilibrium after 6h of contact time and maximum efficiency of 10 %. On the contrary, the application of more concentrated solutions provoked more efficient desorption (up to 43 %) and similar removal percentages, regardless to the applied solution, with the absence of equilibrium achievement. Considering the same soil, Sr (II) elimination was more efficient, up to 20 % in 5 10^{-4} molL^{-1} EDTA with the equilibrium achievement after 3 hours, while in the more concentrated solutions, desorption was significantly higher (65 %) with attained equilibrium after 24 hours.

Soil SS2 contained higher amounts of bonded Co(II) and thus, higher amounts of leached Co(II) were measured, from 20 % to 75 %, for the contact
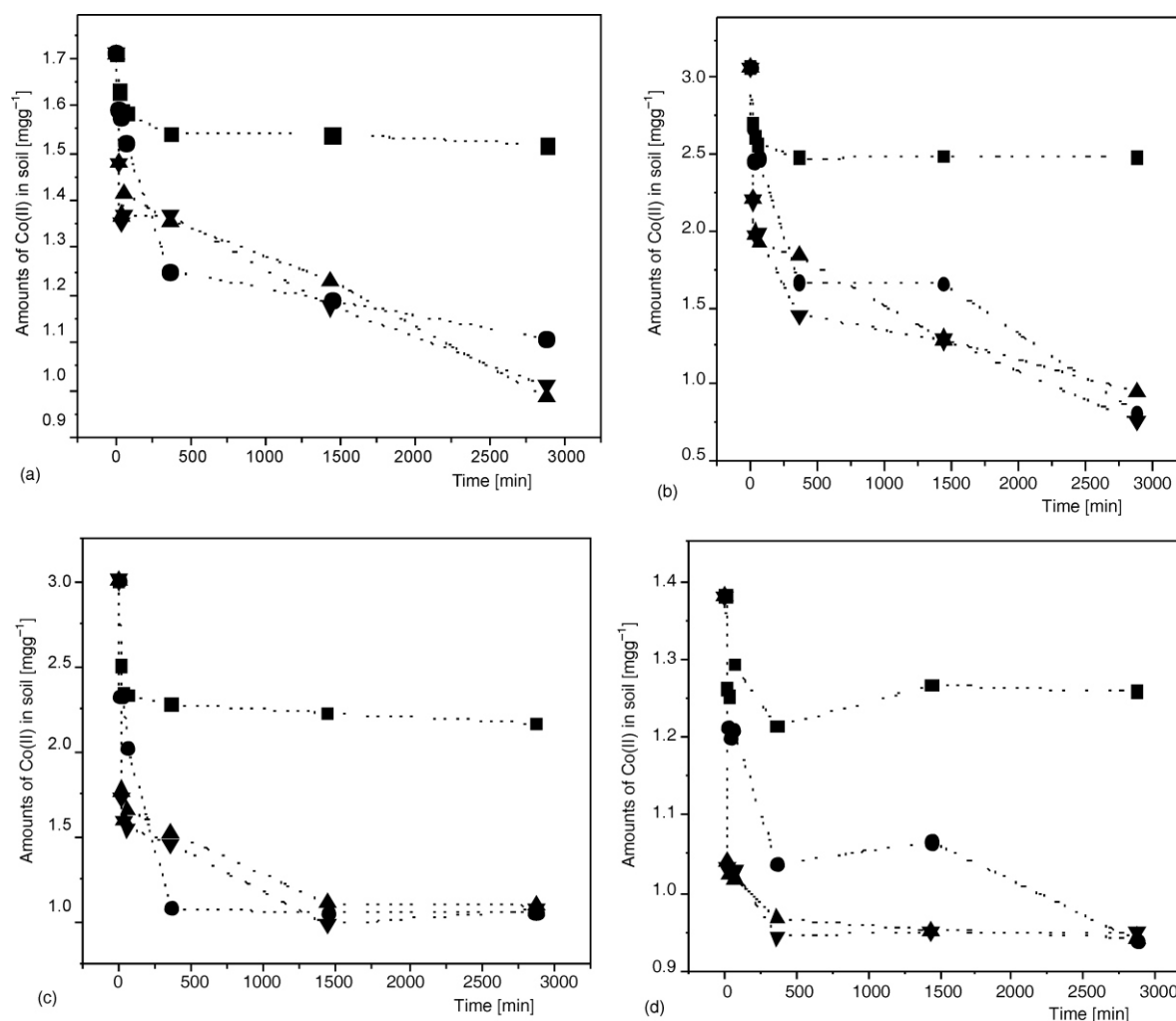

Figure 1. Amounts of Co(II) in (a) SS1 and (b) SS2 and amounts of Sr(II) in (c) SS1 and (d) SS2 during leaching process; symbols denote applied EDTA concentration: (■) 5 10^{-4} molL^{-1}, (●) 5 10^{-3} molL^{-1}, (▲) 5 10^{-2} molL^{-1} and (▼) 0.1 molL^{-1}

S. D. Dimović, *et al.*: Leaching Kinetics of Co(II) and Sr(II) Contaminated Soil ...
Nuclear Technology & Radiation Protection: Year 2018, Vol. 33, No. 3, pp. 252-259
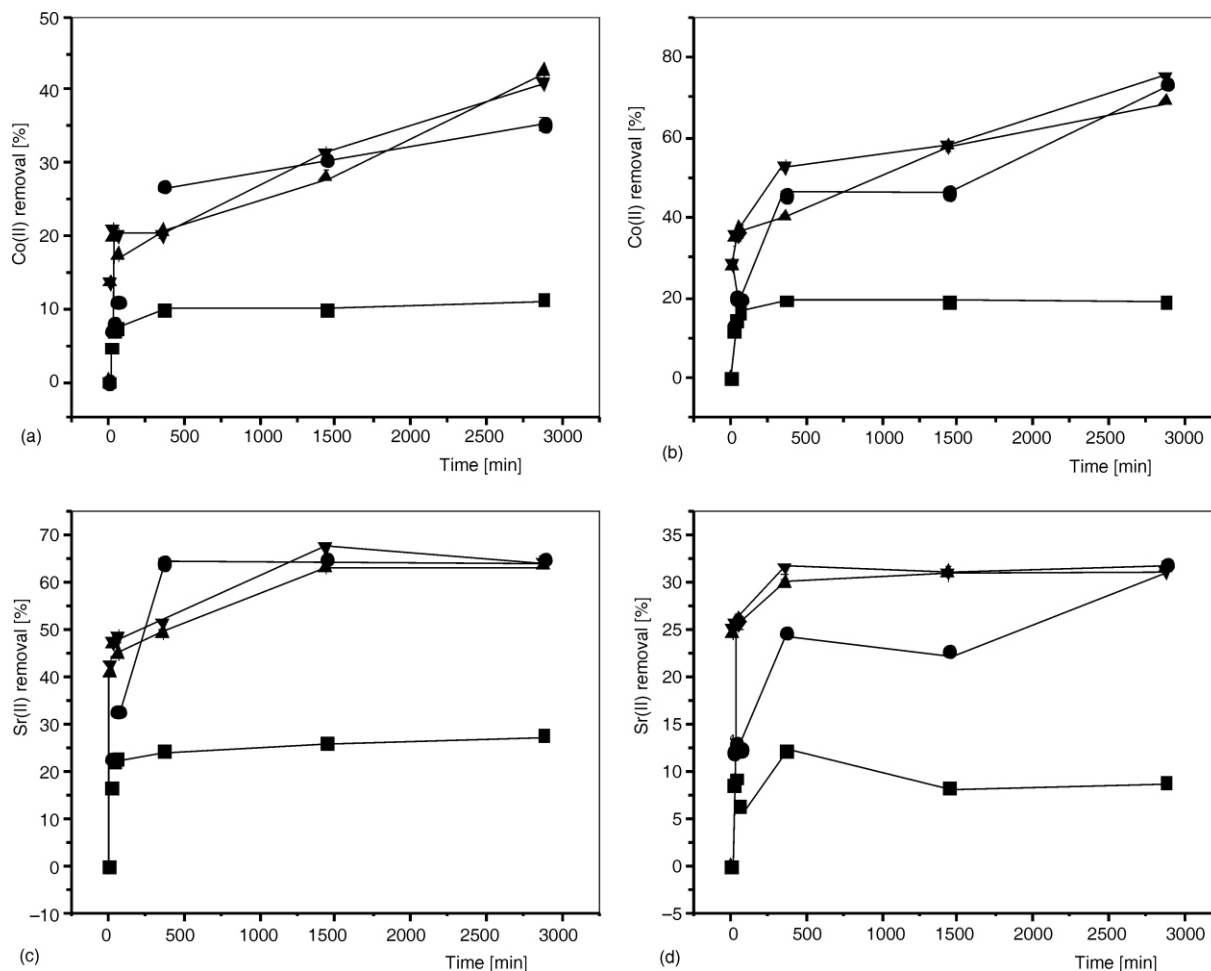
255

Figure 2. Process efficiency for Co(II) leaching from (a) SS1 and (b) SS2 and Sr(II) leaching from (c) SS1 and (d) SS2; symbols denote applied EDTA concentration: (■) 5 10^{-4} molL^{-1}, (●) 5 10^{-3} molL^{-1}, (▲) 5 10^{-2} molL^{-1}, and (▼) 0.1 molL^{-1}
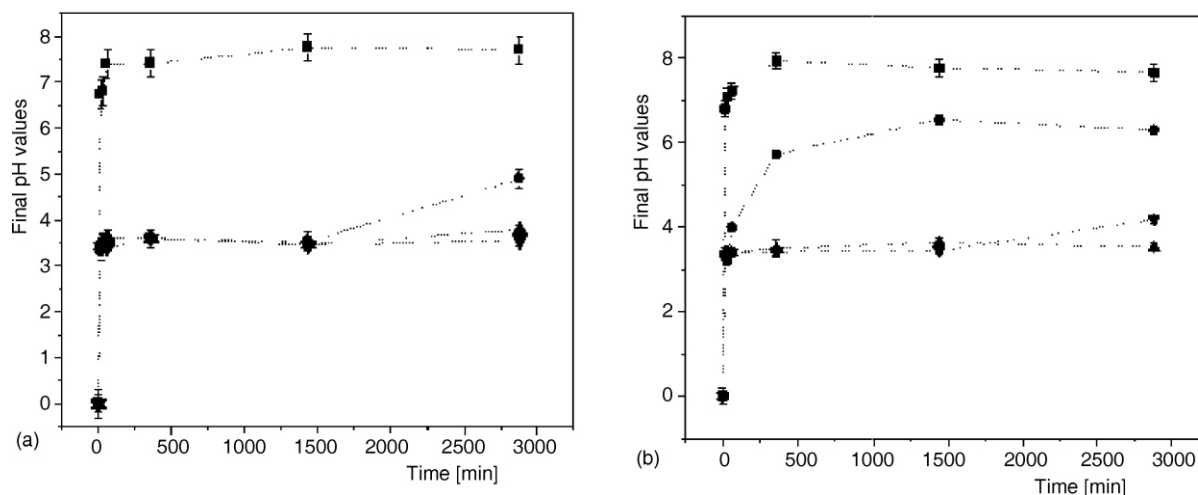

Figure 3. Measured pH values during chemical extraction process with soils (a) SS1 and (b) SS2. Symbols denote applied EDTA concentration: (■) 5 10^{-4} molL^{-1}, (●) 5 10^{-3} molL^{-1}, (▲) 5 10^{-2} molL^{-1}, and (▼) 0.1 molL^{-1}

time of 48 hours. Equilibrium was achieved only in the system containing the most diluted leaching solution. Furthermore, Sr(II) leaching attained equilibrium after 6 hours, regardless to the applied extraction solution, with the highest efficiency of 32 %.

Treatment of differently contaminated soils using EDTA solution showed that pH values of the leach-

ate varied as a function of applied leaching solution (fig. 3). Generally, measured pH values increased at the beginning of the process and then remained constant, which is caused by the soil components buffering properties [18, 19]. The highest values were measured in the solutions with the initial EDTA concentration of 5 10^{-4} mol/L (around 7.7). On the

256

S. D. Dimović, *et al.*: Leaching Kinetics of Co(II) and Sr(II) Contaminated Soil ...
Nuclear Technology & Radiation Protection: Year 2018, Vol. 33, No. 3, pp. 252-259

contrary, the application of EDTA solutions with higher concentrations induced lower pH values, measured in the filtrates, after leaching process, which is in accordance with lower initial pH values of these solutions, but also with higher leached amounts.

According to the obtained results, EDTA solutions in the acidic form are highly efficient for the cleaning of soils with mixed contamination in high amounts, which presented the proof for wide application in the soil remediation processes. EDTA has the ability for pollutant bonding since it is a polyhydroxy compound with the strong chelating potential. Precisely, the mechanism of soil washing using EDTA is quite complex and involves exchange reactions, soil components dissolution and metal remobilization [20]. Consequently, the removal of contaminants from soil is not restricted only to mobile, easy bioavailable fraction, but also onto strongly bonded, which supports high process efficiency.

Process modeling

Since leaching mechanism is complex involving various chemical reactions [20] in the heterogeneous system, it probably can be described using non catalytic reaction models [21]. In that sense, shrinking core model, which assumes that the reactions between liquid and solid matrices occurred at the outer surface of solid, can be used for these processes description. The overall leaching process can be governed by diffusion through the film, diffusion through the product layer and surface chemical reaction [22].

When the controlling step is the diffusion through the film, the process can be expressed by

$$\chi = k_{df} t \qquad (1)$$

Otherwise, when the rate limiting step is diffusion through the product layer, it can be described with

$$1 - 3(1-\chi)^{2/3} + 2(1-\chi) = k_d t \qquad (2)$$

while, when chemical reaction governs the process, the following equation can be applied

$$1 - (1-\chi)^{1/3} = k_r t \qquad (3)$$

In eqs. 1-3, the conversion fraction of solid particle is denoted with χ, t is the reaction time, while with k are denoted apparent rate constants for diffusion through the fluid film (subscript df), for diffusion through the product layer (subscript d) and for chemical reaction (subscript r).

In general, the eqs. 1-3 represents the straight lines which passes through the origin, with the slope equal to apparent rate constant k, in the graphs representing reaction time t on the abscise and the function given on the expression left side on the ordinate.

The abovementioned expressions are applied onto experimental results of Co(II) and Sr(II) leaching from contaminated soils. The results of calculated apparent constants, as well as model validation parameters like R^2 (coefficient of determination), F (obtained from Fisher test) and p-values (probability test), are obtained. The application of film diffusion model (eq. 1) gave the results presented in tab. 1. Generally, diffusion of investigated metals through the liquid film increased with the applied solution concentration increase. This is a result of higher amounts of released contaminants from solid surface, which induce more intensive driving force. Relative high R^2 and F values and $p < 0.05$, pointed out good agreement between model and experimental results obtained for leaching of Co(II) ions. Otherwise, this model is not applicable for description of Sr(II) leaching.

Mathematical model, assuming significant resistance only for diffusion through the product layer (tab. 2), showed relative good description of experimental data, except for Sr(II) leaching from SS2 using the most diluted EDTA solution ($p > 0.05$).

Furthermore, leaching of Co(II) and Sr(II) ions from SS1 in $5 \cdot 10^{-4}$ molL^{-1} EDTA cannot be explained by reaction model (eq. 3). On the contrary, this model can be applied satisfactorily onto other datasets. Since the tested one-resistance models can describe experimental results with satisfactorily accuracy, it can be concluded that these resistances plays important role in the investigated processes. Thus, higher accuracy in process description can be achieved using multiple resistance model.

CONCLUSIONS

Artificially contaminated soil was treated by EDTA reagents in different concentrations in order to define the efficiency of this treatment. Primarily, the main aim was to test the possibility of EDTA utilization in the chemical extraction process as a method for soil remediation for mixed contamination. Process efficiency with EDTA lowest concentration ($5 \cdot 10^{-4}$ molL^{-1}) used was also low. Otherwise, the application of the EDTA solution with the initial concentration of $5 \cdot 10^{-3}$ molL^{-1} or higher showed almost the same efficiency. This suggested that the application of the solution EDTA ($5 \cdot 10^{-3}$ molL^{-1}) is optimal from the aspect of cost and soil degradation properties. Furthermore, process kinetic was fitted using one parameter shrinking core models. As the rate limiting steps of the investigated process, film diffusion, chemical reaction and the diffusion through product layer, were tested. The results revealed that each of the tested steps are involved in the overall mechanism influencing process rate. Consequently, the investigated leaching kinetic curves can be satisfactorily described with the proposed mathematical models. Nevertheless, for highly accurate data description, it is necessary to define the model which involvesd all rate resistances.

S. D. Dimović, *et al.*: Leaching Kinetics of Co(II) and Sr(II) Contaminated Soil ...
Nuclear Technology & Radiation Protection: Year 2018, Vol. 33, No. 3, pp. 252-259

257

Table 1. Parameters obtained for kinetics'data fitting using film difusion model

	$k_{df} \, 10^3 \, [min^{-1}]$	R^2	F	p
Co-SS1				
$5 \, 10^{-4}$	1.52	0.880	21.98	0.018
$5 \, 10^{-3}$	2.11	0.912	30.92	0.011
$5 \, 10^{-2}$	3.90	0.813	13.01	0.036
0.1	4.30	0.855	17.755	0.024
Co-SS2				
$5 \, 10^{-4}$	3.38	0.866	19.31	0.022
$5 \, 10^{-3}$	4.17	0.863	18.83	0.022
$5 \, 10^{-2}$	7.89	0.855	17.73	0.024
0.1	7.70	0.832	14.88	0.031
Sr-SS1				
$5 \, 10^{-4}$	0.818	0.479	3.68	0.128*
$5 \, 10^{-3}$	1.94	0.814	17.50	0.014
$5 \, 10^{-2}$	1.67	0.450	3.27	0.145*
0.1	1.75	0.455	3.34	0.141*
Sr-SS2				
$5 \, 10^{-4}$	0.382	0.568	5.28	0.083*
$5 \, 10^{-3}$	0.382	0.568	5.28	0.083*
$5 \, 10^{-2}$	1.00	0.473	3.60	0.131*
0.1	1.05	0.500	4.01	0.116*

(*) – without statistical significance

Table 2. Parameters obtained for kinetics'data fitting using model involving diffusion through the product layer

	$k_d \, 10^4 \, [min^{-1}]$	R^2	F	p
Co-SS1				
$5 \, 10^{-4}$	0.105	0.7960	15.57	0.017
$5 \, 10^{-3}$	0.195	0.882	44.74	0.0005
$5 \, 10^{-2}$	0.255	0.943	99.78	$5.82 \, 10^{-5}$
0.1	0.249	0.872	80.32	$1.08 \, 10^{-4}$
Co-SS2				
$5 \, 10^{-4}$	1.86	0.931	40.60	0.008
$5 \, 10^{-3}$	0.955	0.944	101.6	$5.53 \, 10^{-5}$
$5 \, 10^{-2}$	0.917	0.919	68.25	$1.70 \, 10^{-5}$
0.1	1.12	0.914	63.57	$2.07 \, 10^{-4}$
Sr-SS1				
$5 \, 10^{-4}$	6.30	0.999	1561	$6.40 \, 10^{-4}$
$5 \, 10^{-3}$	7.74	0.900	18	0.051*
$5 \, 10^{-2}$	34.9	0.972	68.51	0.014
0.1	35.9	0.959	46.89	0.021
Sr-SS2				
$5 \, 10^{-4}$	0.444	0.538	3.50	0.16*
$5 \, 10^{-3}$	1.24	0.780	10.60	0.047
$5 \, 10^{-2}$	5.64	0.816	13.32	0.035
0.1	5.45	0.786	11.00	0.045

(*) – without statistical significance

AUTHORS' CONTRIBUTIONS

The experiments were done by M. Z. Šljivić-Ivanović, M. D. Jović, S. D. Dimović, and I. D. Smičiklas. Data analysis, discussion of the obtained results as well as writing of the manuscript was done by all of the listed authors.

258

S. D. Dimović, *et al*.: Leaching Kinetics of Co(II) and Sr(II) Contaminated Soil ...
Nuclear Technology & Radiation Protection: Year 2018, Vol. 33, No. 3, pp. 252-259

Table 3. Parameters obtained for kinetics' data fitting using model involving chemical reaction

	$k_r\ 10^4\ [min^{-1}]$	R^2	F	p
Co-SS1				
$5\ 10^{-4}$	1.10	0.622	6.571	0.062*
$5\ 10^{-3}$	0.563	0.762	19.24	0.005
$5\ 10^{-2}$	0.636	0.761	19.08	0.005
0.1	0.634	0.730	16.23	0.007
Co-SS2				
$5\ 10^{-4}$	11.90	0.870	20.00	0.021
$5\ 10^{-3}$	1.30	0.858	36.26	0.001
$5\ 10^{-2}$	1.30	0.730	16.25	0.007
0.1	1.45	0.742	17.30	0.006
Sr-SS1				
$5\ 10^{-4}$	29.5	0.971	68.75	0.014
$5\ 10^{-3}$	33.1	0.972	18.3	0.051*
$5\ 10^{-2}$	72.6	0.972	33.65	0.028
0.1	73.8	0.972	28.73	0.033
Sr-SS2				
$5\ 10^{-4}$	5.69	0.651	5.612	0.099*
$5\ 10^{-3}$	9.72	0.780	10.63	0.047
$5\ 10^{-2}$	21.1	0.798	11.87	0.041
0.1	20.8	0.782	10.76	0.046

(*) – without statistical significance

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КИНЕТИКА ИЗЛУЖИВАЊА ЈОНА Co(II) И Sr(II) ИЗ КОНТАМИНИРАНОГ ЗЕМЉИШТА МЕТОДОМ ХЕМИЈСКЕ ЕКСТРАКЦИЈЕ

У овом раду тестирано је земљиште вештачки контаминирано смешом јона Co(II) и Sr(II). Циљ је био да се истражи могућност примене етилендиаминтетра сирћетне киселине (EDTA) у процесу хемијске екстракције као методе за ремедијацију земљишта у случају контаминације. Ефикасност процеса када се користи EDTA концентрације $5 \cdot 10^{-4}$ molL^{-1} је мала, док примена EDTA почетне концентрације је $5 \cdot 10^{-3}$ molL^{-1}, или веће, приближно једнако побољшава ефикасност. Стога, коришћење раствора EDTA концентрације $5 \cdot 10^{-3}$ molL^{-1} оптимално је са аспекта цене и потенцијалне деградације својстава земљишта. Кинетика процеса фитована је моделима непрореаговалог језгра који укључују један отпор преносу масе. Резултати моделовања указују на то да је процес веома комплексан што упућује на неопходну примену модела са више отпора.

Кључне речи: хемијска екстракција, кинетика, контаминација, моделовање