



The Efficiency of Using Na EDTA and DTPA to Extract Different Fractions of Soil Strontium



Ihab M. Farid^a, Magdy A. Rizk^b, Mohamed H. H. Abbas^{a*}, Amal A. A. ElShazly^{a,b}

^aBenha University, Faculty of Agriculture, Soils and Water Department, Egypt

^bNuclear Research Center, Egyptian Atomic Energy Authority EAEA, Inshas, Egypt

STRONTIUM takes the same uptake and distribution pathways of Ca within plants; hence disturbs many secondary metabolites. In humans, it can replace Ca in bones and teeth, making them brittle. Thus, remediating Sr-polluted soils has become an obligation to attain a hazard-free environment. In this context, Sr is one of the potentially toxic elements that have received little attention. Thus, the current study aims at investigating the efficiency of using different chelating agents i.e. Na EDTA and DTPA for the chemical extraction of Sr from contaminated soils. To attain this aim, three soil types i.e. a clay loam one and two sandy loam soils varying in their CaCO₃ content were artificially polluted with Sr at three levels (50, 100 and 200 mg Sr kg⁻¹); then left to equilibrate for 3 months. Thereafter, Sr was determined within the different soil fractions. In the clayey soil, most Sr was bounded with sesquioxide; followed by the exchangeable fraction, while in the other two soils (sandy loam ones), most of soil-Sr dominated within the exchangeable form, followed by the carbonate bound fraction. The contaminated soils were then subjected to soil leaching via either Na EDTA or DTPA and the extractable amount by both methods were comparable, with slight superiority for DTPA. Generally, the extraction efficiencies of Sr by these chelating agents were low in the sandy loam soils which contained 50 mg Sr kg⁻¹ while increased considerably with increasing the level of soil contamination recording approximately 76-88%. Afterward, fractionation of Sr was accomplished again where the carbonate and organic bound fractions were the dominant ones; yet the exchangeable fraction still represents a substantial percentage. Generally, all fractions prior to soil leaching were correlated with each other. The two chelating agents were capable of extracting Sr among the different soil fractions except for the sesquioxide bound which was not extractable by Na-EDTA ($r^2=0.371$) yet it can be extracted by DTPA ($r^2=0.413$). This result provides more evidence for the feasibility of using both EDTA-2Na and DTPA in the chemical extraction of Sr from contaminated soils.

Keywords: strontium; soil polluting; chemical extraction, Na-EDTA; DTPA.

1. Introduction

Strontium is one of the alkaline earth metals (Nedobukh and Semenishchev, 2020) that shares several chemical characteristics with Ca (Jovanović *et al.*, 2021). Thus, Sr is considered a calcium analog (Chatterjee *et al.*, 2020; Koshy and Pathak, 2020). It takes the same Ca uptake and distribution pathways within plants (Jovanović *et al.*, 2021); nevertheless, it does not perform nutritional functions (Höllriegl,

2019). It is mobile within plants (Burger and Lichtscheidl, 2019), translocate in high concentrations to the aboveground plant parts, and disturb many secondary metabolites (Dresler *et al.*, 2018). This in turn decreases plant growth and productivity (El-Shazly *et al.*, 2016). There are two main routes for this element to enter the human system i.e. via plant ingestion and/or breathing vapors or dust containing Sr that dissolve with moisture inside the lung and enter the bloodstream

*Corresponding author e-mail: mohamed.abbas@fagr.bu.edu.eg

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quickly (ATSDR, 2004). It replaces Ca in bones and teeth (Voronina *et al.*, 2020), making them brittle (Koshy and Pathak, 2020) and causes Sr rickets, especially in infants (Scott *et al.*, 2020)

Strontium average concentration in soils is approximately 0.035% (Nedobukh and Semenishchev, 2020); yet the anthropogenic activities particularly milling, burning of coal, and applying phosphate fertilizers, increase its levels substantially in the surroundings (Höllriegl, 2019). This potentially toxic element has received little attention (Rinklebe *et al.*, 2019). Unlike organic contaminants, PTEs, in general, do not undergo biodegradation and persist in soil (Hashim *et al.*, 2017; Abbas and Bassouny, 2018; Mohamed *et al.*, 2018; ElShazly *et al.*, 2019 and b; Farid *et al.*, 2019; Abbas *et al.*, 2020; Bassouny and Abbas, 2020; Bassouny *et al.*, 2020; Negim and Sweed 2020; El-Ramady *et al.*, 2021; Hussein *et al.*, 2022; Ali *et al.*, 2023; El-Shwarby *et al.*, 2023; Mekawi *et al.*, 2023). In particular, Sr has high electronegativity and can form complexes with organic and inorganic ligand (Nedobukh and Semenishchev, 2020). These complexes are not highly stable (Nedobukh and Semenishchev, 2020); and may be leached out of soils via dissolving in acid solutions (Zhang *et al.*, 2010) or chelating agents such as EDTA (Guillén *et al.*, 2018; Begum *et al.*, 2020) and DTPA (Margon *et al.*, 2013; Hassan *et al.*, 2020). The current study aims at investigating the efficiency of using different chelating agents i.e., EDTA and DTPA for the chemical extraction process of Sr from artificially contaminated soils of different characteristics at three levels (50, 100 and 200 mg Sr kg⁻¹); then left to

equilibrate for 3 months. Thereafter, Sr was determined within the different soil fractions. Specifically, we anticipate that Sr remains longer in the soil in mobile fractions before being transferred to more stable ones (Hypothesis 1). The residual fraction of Sr in this short period (90 days after pollution) is unstable and therefore the HF/HClO₄ method is not appropriate for determining this fraction within this short time (Hypothesis 2). The chelating agents particularly EDTA and DTPA extracts efficiently Sr from soil (hypothesis 3) and the extracted amounts were not only from the soluble and exchangeable forms but from different Sr fractions in soil (hypothesis 4).

2. Materials and Methods

2.1 Soil sampling and analytical methods

Three surface soil samples (0–30 cm) varying in their CaCO₃ contents were collected from (S₁) Mashtul El Souq (30° 21' 27" N and 31° 23' 02" E), Al-Sharqiyah Governorate, (S₂) Anshas (30° 23' 19" N and 31° 26' 52" E), Al-Sharqiyah Governorate and (S₃) Ameria industrial area (33° 17' 54" N and 44° 17' 59" E), Alexandria Governorate. These samples were air dried, crushed and finely ground, then sieved to pass through a 2 mm sieve. Physicochemical characteristics of the investigated soils were determined according to Klute (1986) and Sparks *et al.* (1996) and the obtained results are presented in Table 1.

Table 1. Physicochemical characteristics of the investigated soils.

Property	Mashtoul El Souq (S ₁)	Anshass (S ₂)	Aamria (S ₃)
Coarse Sand %	19.00	30.90	31.50
Fine Sand %	22.20	38.80	33.60
Clay %	28.8	9.30	12.60
Silt %	30.00	21.00	22.30
Textural Class	Clay Loam	Sandy loam	Sandy loam
Bulk density (Mg m ⁻³)	1.40	1.52	1.41
pH (1:2.5)	7.88	7.79	7.83
EC (1:5) dS m ⁻¹	2.49	5.89	3.29
Organic matter content (g kg ⁻¹)	18.00	8.60	8.6.
CaCO ₃ (g kg ⁻¹)	99.00	165.00	231.00
CEC (cmol Kg ⁻¹)	24.32	4.05	10.97
Sr concentration (mg L⁻¹)			
Total	ND	ND	ND
Soluble	ND	ND	ND

ND: Not Detected

These soils were then contaminated with strontium chloride (extra pure, M.W. 266.62 obtained from Alpha Chemika) at three different rates 50 mg (Sr50), 100 mg (Sr100) and 200 mg Sr kg⁻¹ (Sr), then left to equilibrate for 90 days while maintaining soil moisture at the field capacity via the gravimetric method. Thereafter, Sr was determined within different soil fractions.

2.2. Sequential extraction procedure

A sequential extraction procedure was followed (pre- and post-soil leaching with synthetic chelating agents) to determine the distribution of Sr among the different soil fractions as outlined by **Tessier et al. (1979)** as follows: (1) **water soluble**: extracted by 15 mL of deionized water for 2 hours, (2) **exchangeable**: the residue from step “1” was treated by 8 mL of 1 M MgCl₂ (pH = 7.0) for an hour, (3) **carbonate bound**: the residue from step “2” was treated with 8 mL sodium acetate (CH₃COONa) 1 M then adjusted to pH = 5.0 and left to equilibrate for 5 hours, (4) **sesquioxide bound**: the residue from step “3” was extracted by 20mL of 0.04 M hydroxylamine hydrochloride (NH₂OH.HCl) in 25% (v/v) at 96°C with agitation for 6 hours, (5) **organic bound**: the residue from step “4” was extracted by both 3 mL of 0.02M HNO₃ and 8 ml of 30% H₂O₂ (adjusted to pH = 2.0 with HNO₃), then heated to 85°C for 5 hours. After cooling, 5 mL of 3.2 M NH₄OAc was added and the mixture was diluted to 20 mL then agitated for 30 minutes and (6) **the residual fraction**: the residue from step “5” was placed in platinum crucible and digested using a mixture of hydrofluoric and perchloric acid.

Prior to the beginning of each extraction stage, residues were shaken with 8mL deionized water for 30 minutes, centrifuged, and the leaching solutions were discarded. All soil extractants were analyzed for Sr using Atomic Absorption Spectroscopy (Buck 210VGA).

2.3. Soil leaching with Na-EDTA and DTPA

Other soil portions were treated with either 0.05 M Na-EDTA disodium salt (M.W. 372.24, purity 99.0%, obtained from SuvChem Laboratory Chemicals) or DTPA (FW 393.3, Sigma Chemical company) solutions (adjusted at pH= 7) at a rate of 1:10 (w/v) ratio then left to equilibrate for 24 h at room temperature while being shaken. Thereafter, the extracts by decantation passed through a 0.45 µm filter and their content of Sr was determined.

2.4. Statistical analysis

Data were subjected to one-way ANOVA and Duncan's text using PASW statistical software 18. Figures were plotted via Sigma Plot 10

3. Results and Discussion

3.1. Distribution of Sr among the different soil fractions prior to soil leaching with synthetic chelating agents

This protocol of soil fractionation was conducted prior to soil leaching with Na-EDTA or DTPA and the obtained results are presented in Fig 2. In the clayey soil, most soil-Sr was bounded with sesquioxides; followed by the exchangeable fraction. Probably, Sr was initially sorbed on clay particles (Smičiklas *et al.*, 2015; Siroux *et al.*, 2021); then formed short-range complexes with sesquioxide, especially with Al- and Fe-oxides (Chiang *et al.*, 2010). Nevertheless, the available content of Sr (soluble + exchangeable fractions) in the clayey soil was still high, comprising from 22-27% of the total content in the soil.

In the other two soils (sandy loam ones), most of the soil-Sr dominated within the exchangeable form, followed by the carbonate bounded fraction. In this context, the carbonate bounded fraction was noticeably higher in the soil with higher content of CaCO₃, forming crystals as found by Saito *et al.* (2020). The above results justify partially the first hypothesis.

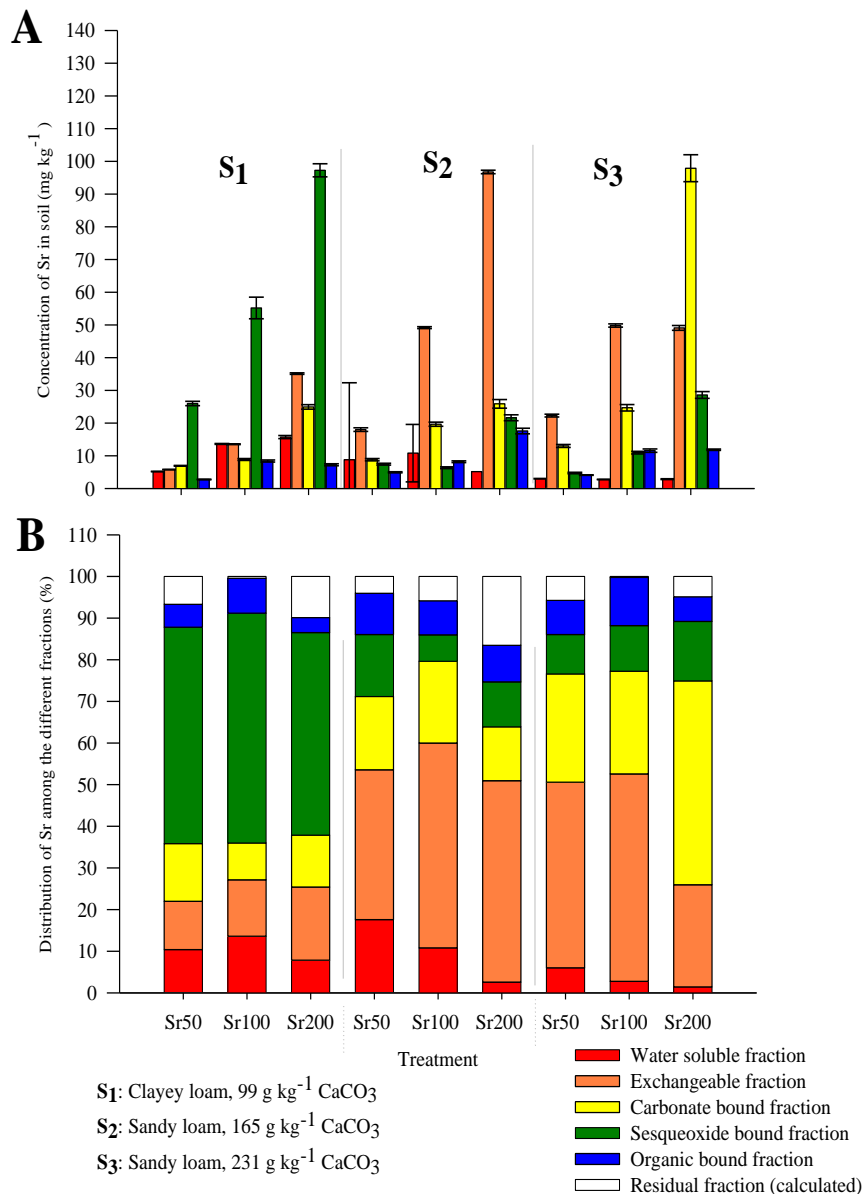


Fig. 1. Distribution of Sr (mean \pm standard deviation) among the different soil fractions prior to soil leaching with either EDTA-2Na or DTPA: (A) means+ standard deviation and (B) the percentage from the total content in soil.

3.2. The residual fraction of soil Sr

This fraction was determined via HF/HClO_4 and the obtained results are presented in Fig 2A. This fraction was relatively high in highly contaminated sandy loam soils (Sr200) while displaying lesser values in soils of lower contamination levels i.e., Sr50 and Sr 100. In this regard, the order of residual Sr in soil that contained relatively low content of CaCO_3 (165 mg kg^{-1}) was $\text{Sr200} > \text{Sr100} > \text{Sr50}$, while in the other soil that contained higher CaCO_3 content (231 mg kg^{-1}) the following sequence was detected: $\text{Sr200} > \text{Sr50} > \text{Sr100}$. There might exist dynamic equilibria between the residual fraction and the other soil fractions, causing substitutable exchange among

Sr fractions, especially within Sr50 and Sr100. More evidence to support the rapid transformations of Sr among the different fractions after a short time period of contamination with Sr was noticed by **Lee et al. (2022)** who found that Sr was partially co-precipitated in soil. Even in the long term, Sr, which was dominated within the residual form on the topsoil, decreased considerably corresponding to all other fractions in deeper soil layers (**Lujaniene et al., 2002**). Based on the above results, the second approach becomes acceptable.

In the clay loam soil, sesquioxide fractions might be in dynamic equilibrium with the residual Sr; and this might clarify partially why Sr200 exhibited lower

residual Sr versus Sr100. Overall, the results of the residual fraction of Sr were confusing; therefore, the residual Sr was estimated via another method i.e. subtracting the different Sr fractions from the total content in soil then a regression relation was conducted between the measured and the estimated residual Sr values (Fig 2B). The calculated “ r^2 ” value was low indicating an insignificant relationship

between the estimated and the measured quantities of residual soil-Sr. Probably, this methodology is not suitable for determining the residual Sr fraction in soil within the short period of contamination because of the rapid exchange of Sr among the different Sr fractions.

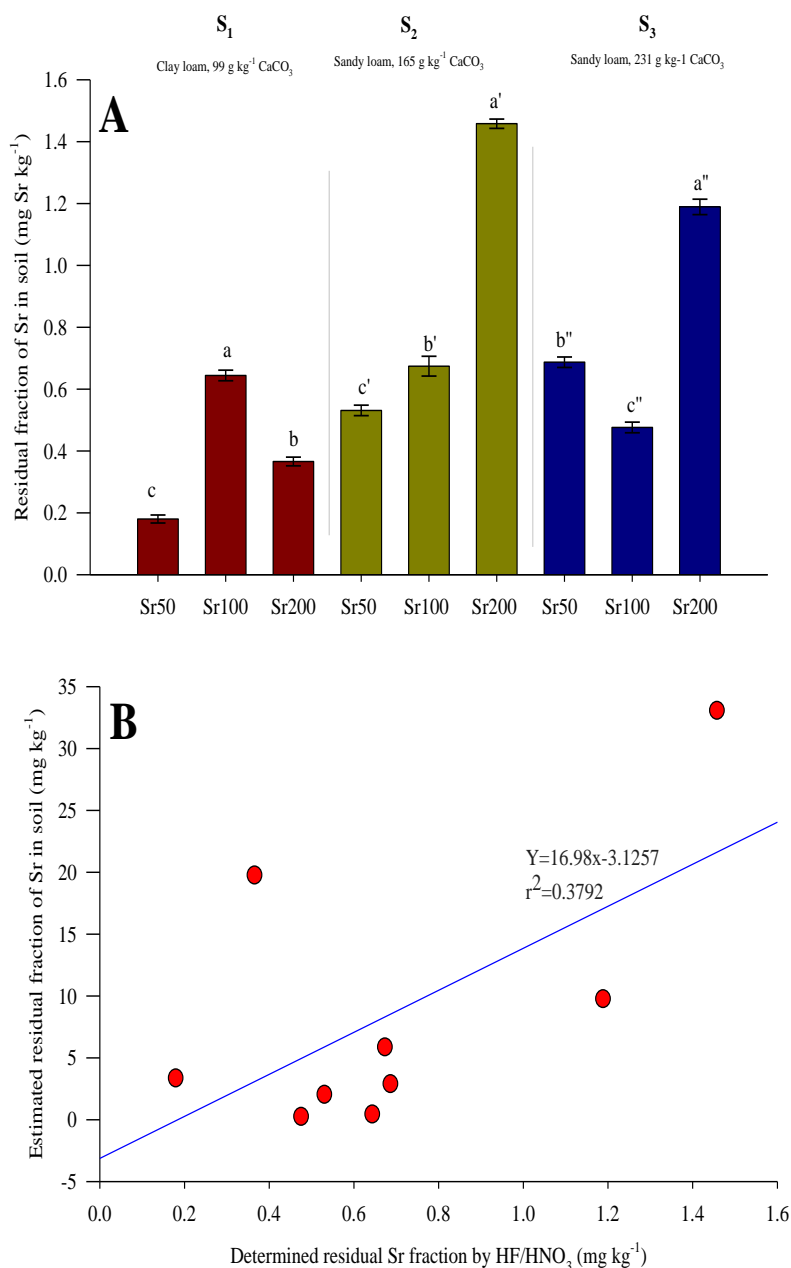


Fig. 2. The residual fraction of Sr in soil (mean± standard deviation): (A) measurable amount by HF digestion (means± stand dev.) (B) in relation with the estimated residual fraction in soil (subtracting the different Sr fractions from the total content in soil). Similar letters indicate no significant variations among treatments.

3.3. Chemical extraction of Sr from the contaminated soils via EDTA versus DTPA

The extracted amounts of Sr from soil via either EDTA or DTPA seemed to be comparable, with a little superiority for DTPA. Probably, DTPA increased the dissociation rate of Sr-bearing minerals comparable to EDTA (de Pasquali *et al.*, 2019). Based on these results, the extraction efficiency (EF) values were calculated as a percent of the total content in soil according to Xu *et al.* (2009). Generally, the EF values were higher in sandy loam soils versus the clayey ones. Probably, Sr underwent surface complexation (Berns *et al.*, 2018) on clay minerals (Mulyutin *et al.*, 2012) and this process was fast (Smičiklas *et al.*, 2015). A point to note is that the extraction efficiency of Sr with these

chelating agents was low in Sr 50. Probably, these chelating agents have relatively low capabilities to bind with soil Sr while preferring to chelate with other soil cations (de Pasquali *et al.*, 2019). Nevertheless, the EF values increased considerably in the sandy loam soils with increasing the level of soil contamination recording approximately 76-88% EF in both Sr100 and Sr200. In another trial to separate Sr from contaminated resins, these two chelating agents recorded up to 100% Sr removal efficiency by Surrao *et al.* (2019). Overall, these results confirm partially the third hypothesis, especially in light textured soils.

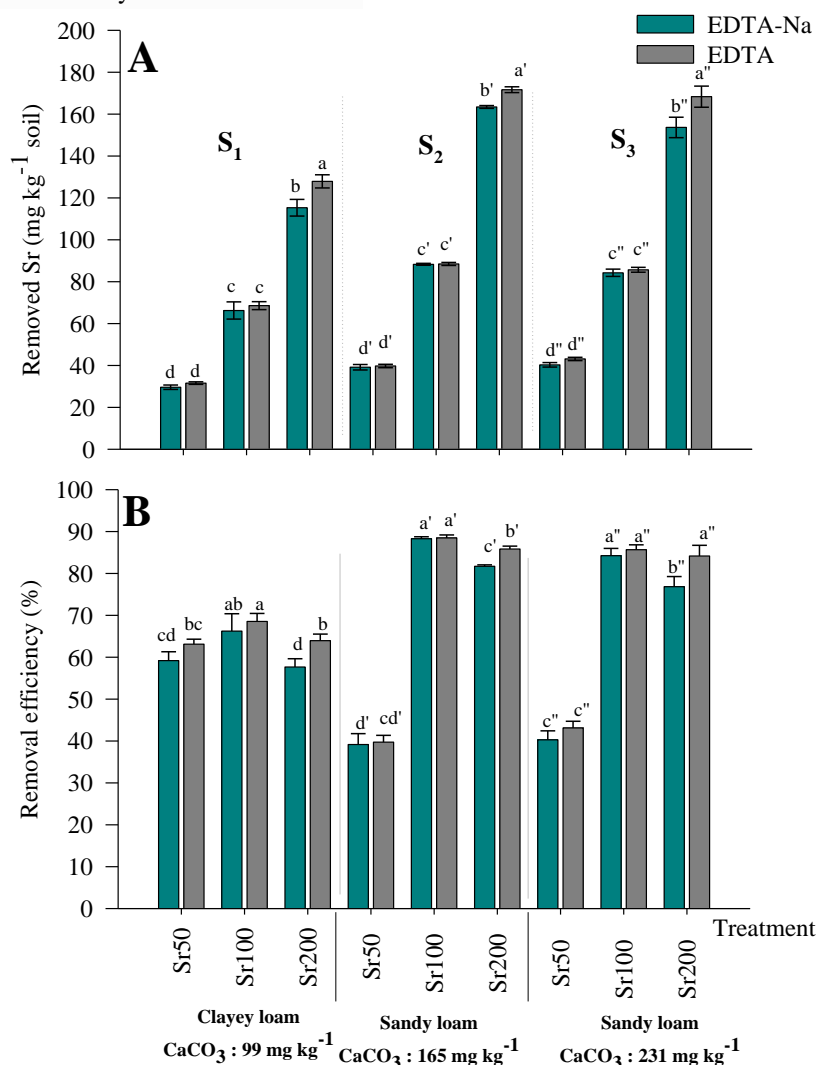


Fig. 3. Chemical extraction of Sr from artificially contaminated soils (mean \pm standard deviation) via either EDTA or DTPA (means \pm st dev, A) and the calculated removal efficiency of this procedures (B). Similar letters indicate no significant variations among treatments.

3.4. Correlation coefficients between different Sr fractions and their chemically extractable amounts via Na-EDTA and DTPA

Soluble Sr in soil was correlated significantly with the corresponding exchangeable form and these two fractions were bounded with the other fractions (Table 2). Mostly, Sr ions were sorbed onto low-affinity sorption sites (Bouzidi et al., 2019) and this process dominated slowly before reaching equilibrium (Shi et al., 2021). The only insignificant

correlation among the different Sr fractions was found between the sesquioxide bound Sr and the organic bound fraction. On the other hand, the two chelating agents were capable of extracting Sr among the different soil fractions except for the sesquioxide bounded fraction which was not extractable by Na-EDTA ($r^2=0.371$) yet it can be extracted by DTPA ($r^2=0.413$). This result provides more evidence for the feasibility of using DTPA in soil leaching of Sr from contaminated soils and therefore authenticates the fourth hypothesis.

Table 2. Coefficients of determination (r^2) calculated for the equilibrium among different Sr fractions in artificially contaminated soils (prior to soil leaching with synthetic chelating agents) in relation with the extractable amounts of Sr via soil leaching with either EDTA or DTPA.

	Na-EDTA						
	Extractable Sr	DTPA Extractable Sr	Soluble Sr	Exch- Sr	Carbonate bounded Sr	Sesquioxide bounded Sr	Organic bounded Sr
	Na-EDTA extractable Sr						
EDTA extractable Sr	0.997**						
Soluble Sr	0.764**	0.773**					
Exchangeable Sr	0.936**	0.953**	0.778**				
Carbonate bounded Sr	0.877**	0.890**	0.609**	0.945**			
Sesquioxide bounded Sr	0.371	0.413*	0.369	0.648**	0.719**		
Organic bounded Sr	0.883**	0.890**	0.645**	0.810**	0.754**	0.205	

** Correlation is significant at the 0.01 level (2-tailed).

* Correlation is significant at the 0.05 level (2-tailed).

3.5. Distribution of Sr among the different soil fractions after soil leaching with synthetic chelating agents

After soil leaching with the investigated synthetic chelating agents (Na EDTA and DTPA), considerable decline occurred in all Sr fractions; thereafter, Sr was re-distributed again among the different soil fractions as presented in Fig 5. In this concern, the sesquioxide, carbonate and organic bound fractions were the dominant ones after soil leaching; yet the exchangeable fraction still represents a substantial percentage. Although, high concentrations of this contaminant are bound to relatively mobile fractions (Smičiklas et al., 2015); organics and sesquioxides especially Al- and Fe-oxides play significant roles in Sr sorption (Chiang et al., 2010).

Conclusions

Different Sr fractions were equilibrated with each other during the first 90 days after pollution. The synthetic chelating agents, particularly DTPA were capable of extracting Sr from the different soil fractions (mainly from the soluble and exchangeable fractions), recording fair efficiencies (76-88%) in highly contaminated soil while in the low contaminated soils of light texture; these two chelating agents might not be enough to attain successful remediation via soil leaching of the contaminant.

4. Conflicts of interest

There are no conflicts to declare.

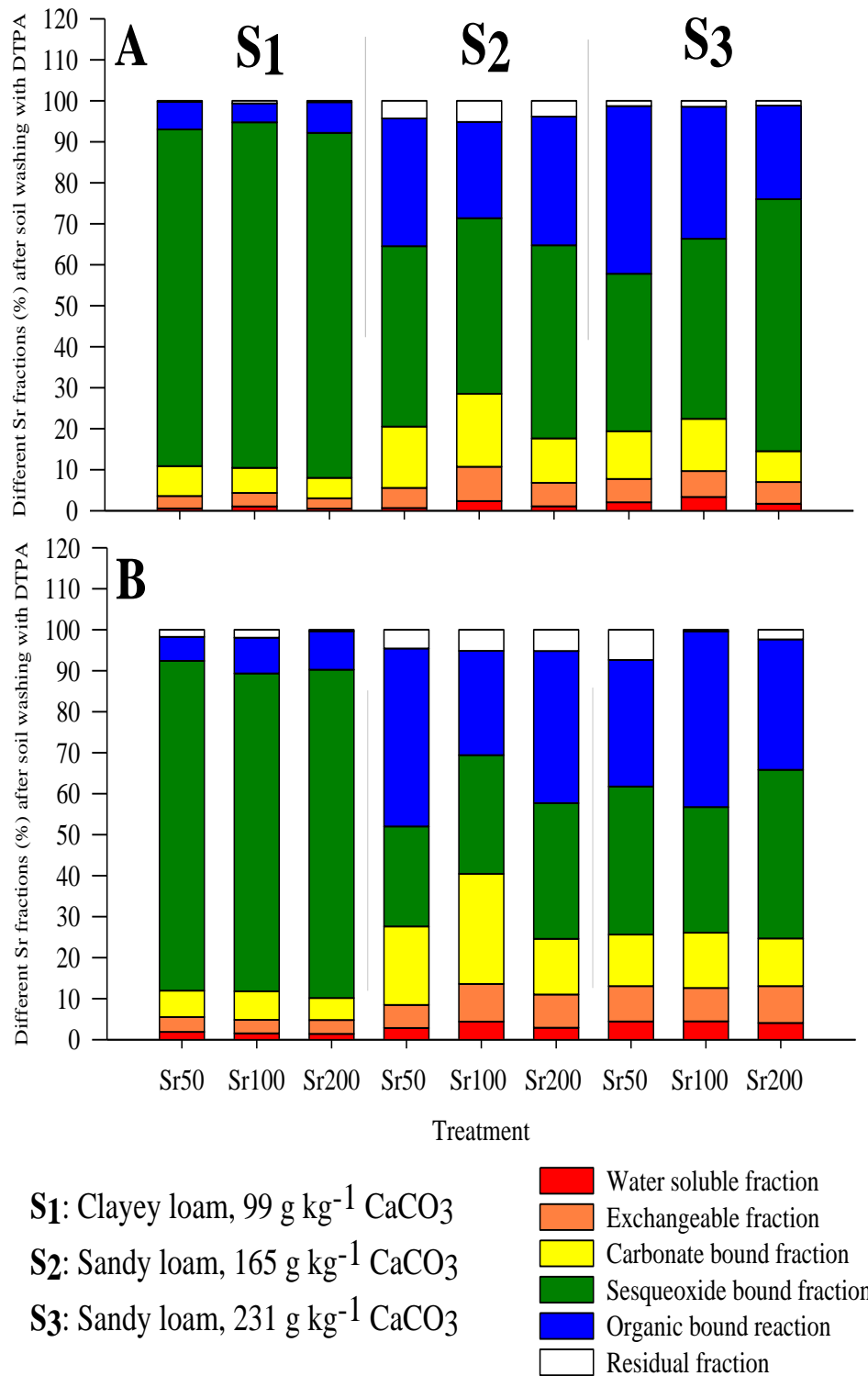


Fig. 4. Distribution of Sr among the different soil fractions after soil leaching with either EDTA-2Na (A) or DTPA (B).

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