Final report

Project title: Comparison of soil redistribution due to water erosion under conventional and conservation tillage using rare earth oxides as tracers

Project number: PD OTKA 112729

Introduction

Agricultural practices, like conservation tillage based on the sustainable use of natural resources and the protection of biodiversity and landscape values, have a key role in the mitigation of the already unsustainable but still increasing rates of soil erosion. Studies comparing conventional and different kinds of conservation agricultural practices proved that applying adequate sustainable agricultural systems can guarantee that the rate of soil loss remains under that of soil formation (Bádonyi et al., 2008, Madarász et al. 2021).

Although laboratory and field studies of soil erosion on arable lands have several decades of tradition, our knowledge on the exact place of origin of eroded sediment is still limited resulting in gaps in controlling soil loss and its associated nutrient and pollutant transport, as well as in developing appropriate watershed management tools. As traditional modelling and monitoring techniques for soil erosion do not supply information on the rates and spatial patterns of soil redistribution within the studied area, most existing soil loss data are spatially averaged. The need for spatially distributed data for validation of process-based soil erosion models directed scientific interest early to the development of tracing methods. These approaches are used to determine the rate of soil loss or sediment generation, identify and quantify sediment sources, and track soil redistribution through the landscape. To fulfill this requirement various types of tracers have been developed and used.

The suitability of rear earth oxides (REOs) for sediment tracing was already confirmed by several previous studies in both laboratory and field scale, due to the advantageous characteristics of REOs as tracers (Zhang et al 2001; Polyakov et al., 2004; Kimoto et al. 2006; Deasy and Quinton, 2010; Michaelides et al., 2010). However, these experiments did not gain information on soil redistribution along the studied slope profile resulting in a lack of knowledge in this field.

One of the main goals of this project was to fill this gap: to investigate and track sediment redistribution along the slope, thus testing the applicability of the REO tracer method in field scale, in a multi-year period. Carrying out this study on the experimental site of the Geographical Institute of the Research Centre for Astronomy and Earth Sciences (RCAES), where 2x2 arable plots of conventional and conservation tillage practices are applied, added information of great value to the results.

Though the role of agricultural cultivation in soil transportation may not be as obvious as those of water or wind erosion, it is proven that the extent and severity of its effects are in most cases at least in the same order of magnitude (Van Oost et al, 2006). The rate of the effects of tillage erosion depends on several factors. Slope gradient, tillage direction, and depth are the most determining ones, but tillage speed, soil condition, and characteristics of the device used are also important (Van Muysen et al, 2002). The effect of tillage erosion on soil quality has also been investigated (Kosmas et al, 2001; Heckrath et al, 2005) verifying its impact on the variability of crop yield within a plot.

Difficulties during the project

Originally, analysis of REE concentrations of the samples was planned to be carried out using the X-ray fluorescence spectrometer of the Geological and Geochemical Institute. As this instrument has been out of order before starting these analyses, the samples had to be sent to ACME Analytical Laboratories Ltd. for ICP-MS analyses instead. Due to the significant increase of analysis cost, replanning the budget and the sampling was necessary as the number of analysed samples had to be limited. I have asked for permission for this replanning which was approved by NKFIH. After evaluating the first results of the ICP-MS analysis I decided to take samples from the sediment after each rainfall event and to take them from the plot itself two times a year: once after the rainy period in spring-early summer and once in autumn according to the weather conditions, also harmonised with the cultivation activities.

Laboratory experiment to study incorporation of rare earth element oxides in soil aggregates of different size fractions has been carried out according to the working plan. The experiment described by Zhang et al. (2001) was slightly modified and extended by a wetting drying experiment to obtain more information on the binding characteristics of the REOs. Analysis of the samples of this experiment were nevertheless not carried out because of the followings:

The opportunity of investigating soil redistribution using REOs as tracers by rainfall simulator on the field and in laboratory emerged during the project and promised much more interesting results than those of the here mentioned planned laboratory experiment. As both my financial and human resources had to be considered particularly due to the replanning caused by the unexpectedly increased analytical costs I have decided to leave over the analysis of the samples from this laboratory experiment. This decision was made considering the results which could be expected from both (the planned and the rainfall simulation) investigations. The use of rainfall simulators with the given conditions (described among the results) has promised novel outcomes while the originally proposed experiment is practically a repetition of a one described in the literature.

As I have been hoping all along the project that I still can make the analysis of this samples and thus fulfil the working plan I have not asked for modification of the project plan.

As my baby was born in July 2017, I have asked for permission to intermit the project. Because some parts of the work could not be stopped even in this case, the pause started only in November. Despite of this, of course progresses in the work could not be made in this situation like planned, but tasks which could not be delayed were carried out in time also during the pause.

Due to the above mentioned problems aggravated with the difficulties of the quarantine situation during the extension time of my project and the fact that as a consequence of them the final database was set up very late, publication of the final results is still missing. It is however under preparation and I am planning to publish it in a leading journal of the topic.

Materials and methods

The experimental site

The experimental station of the Geographical Institute at Szentgyörgyvár was established in 2003 as part of the SOWAP (Soil and Surface Water Protection Using Conservation Tillage in Northern and Central Europe) project to study conventional (ploughing) and conservation tillage in a comparative manner (Field et al., 2007; Kertész et al., 2007; Lane, 2007; Bádonyi et al., 2008a, 2008b; Madarász et al., 2011; Madarász et al., 2021). The study area is located in southwest Hungary, in a hilly region 7.5 km west of Lake Balaton, near the village of Szentgyörgyvár (46.748°N, 17.147°E, 150 m a.s.l.). The climate is warm-summer humid continental, the long-term mean annual temperature is 11°C, and mean annual precipitation is 700 mm. The slope of the field-scale experimental site is 10%. The parent material is loess and the soil is eroded silty loam Luvisol with low soil organic matter content (WRB, 2014). For the erosion experiment of conventional and conservation tillage, 2×2 identical plots of 24×50 m (width × length, 1200 m^2) were established. Plots were isolated from the rest of the slope using metal bunds. Agricultural tillage is still possible on plots of this size, but they are small enough for the proper collection and measurement of runoff and soil loss. The only difference between the plot pairs was the tillage type; they received the same treatment in every other aspect.

Ordinarily, contour tillage is applied on the plots. Conventional tillage consisted of mouldboard ploughing to 25–30 cm depth, harrowing, and seed-bed preparation every year. Conservation tillage was a plough-less, non-inversion tillage operation, which was characterised by a reduction in the number of tillage operations and by leaving crop residues on the soil surface covering ~30% of it. A cultivator was applied here in 8–10 cm depth.

Experimental design

In this study, two neighbouring plots were used: one under ploughing tillage (PT) and one under conservation tillage (CT).

The plots were divided using downslope symmetry and the two sides into three equal stripes along contour lines. Thus, six equal subplots were generated on both cultivation types (Fig. 1). As the first step of the research, soil sampling was carried out in the spring of 2015. Twelve samples (two from each subplot) were taken both from the PT and CT plots to determine the background rare earth elements’ (REE) concentrations. Each sample composed of five subsamples taken from about 0.2 m apart from each other. Samples were taken from the upper soil layer.

Basic soil characteristics were determined as well in the laboratory of the Geographical Institute RCAES. After receiving the results with the rare earth element background concentrations exact planning of the soil erosion tracking become possible.

Taking background concentrations, experimental considerations, and actual rare earth oxide prices into account tagging of a one-meter-wide stripe running through the middle of each subplot were resolved using the oxides of Pr, Yb, Er, Ho, Sm, and Gd (Fig. 1).

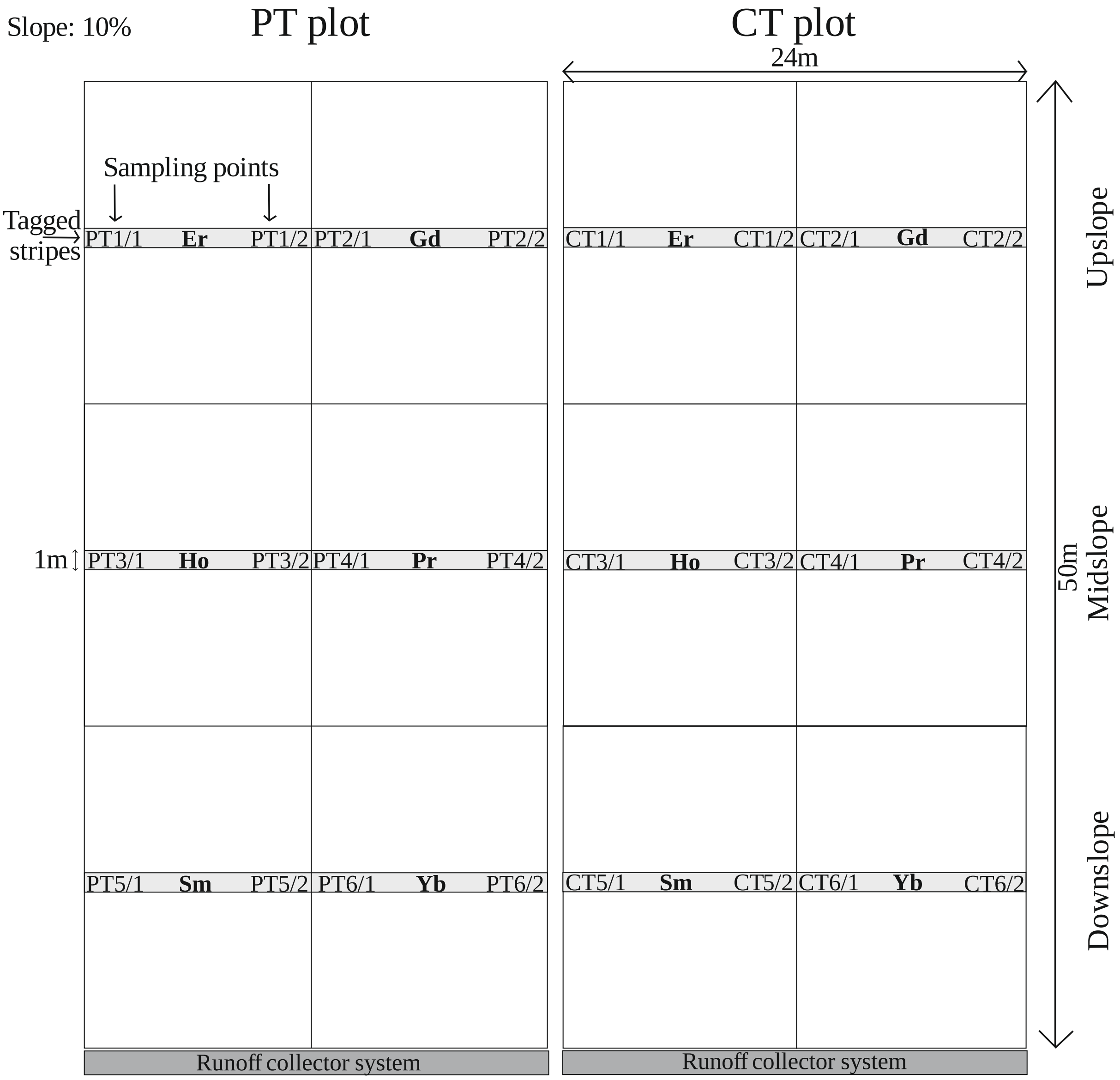


Fig. 1: *Scheme of the experimental site at Szentgyörgyvár with the tagging pattern.*

Tagging was designed according to the following: 100 mg/kg REE target concentration was calculated in view of the background concentrations. This value was assigned to be high enough for detecting by X-Ray fluorescent sepctrometry in the case of each selected REE. Amounts to disperse were calculated differently for the PT and CT plots because of the difference in tillage depths. The aim was to have the same concentration in the cultivated layers in both cases. Tillage depth is 30 and 8 cm on the PT and the CT plots, respectively so REO amounts were calculated according to these layer thicknesses.

Tagging the soil stripes with REOs on the field after autumn tillage had to be designed. Tracers were applied in October 2015 on a smooth and even soil surface. REOs were applied to the slope in suspension in deionised water, using a calibrated airbrush. The soil surface was wetted before spraying to ensure better adherence. Even distribution of the given REO in the whole length of the stripes was ensured by spraying it by square metres using a 1x1 m wooden frame (Fig. 2).

Fig. 2: *Tagging 1 m wide stripes with REOs*.

At the beginning of the second year of the project, spring cultivation processes finished the mixing of the REE oxides into the cultivated soil layer. The upper soil layer of the plots was mixed by cultivation in an approximate depth of 30 cm on the PT and 8 cm on the CT plot. Initial samples were collected afterward to determine the result of the tagging and mixing. Results of the analysis of these samples verified that tagging of the stripes and mixing of the tagged surface layers with the cultivated layers were successful on the PT plot. On the CT plot Sm and Yb, the elements used for tagging on the downslope tagged stripe were not found in the samples.(Fig.5, 6)

Sampling was carried out according to the followings: As costs of sample analysis increased significantly compared to the original plans for the reasons described above, the number of analysed samples had to be limited. Therefore, after studying the first results of ICP-MS analysis I decided to take samples from the plots not after all rainfall events but at least two times a year: once after the rainy period in spring-early summer and once in autumn according to the weather conditions, also harmonised with the cultivation activities. Samples were taken thus from the plots once before tagging (03.03.2015) and 8 times after tagging (14.04.2016, 23.06.2016, 15.09.2016, 20.06.2017, 28.09.2017, 18.04.2018, 20.06.2018, 04.10.2018). Two samples (composed of five sub-samples) of each tagged stripe were taken during each sampling time. Each rill found at the sampling times was also sampled. Moreover, sediment leaving the study plot was collected and sampled after each rainfall event.

Usually, contour tillage is applied on the experimental plots which is much more favourable resulting in significantly less net downslope soil displacement, runoff, and sediment yield, reducing it in different rate compared to upslope and downslope tillage depending on topography, soil properties, climate, and management practices (Van Muysen et al, 2002; Jia et al, 2020; Zhang et al, 2004). However, its role in soil translocation in both downslope and contour direction has to be taken into consideration also in the case of cultivation along the contours.

REE analyses

Concentrations of the REEs in the soil and sediment samples were analysed using an ELAN 9000 ICP-MS at the Acme Labs, Bureau Veritas Laboratory, Canada. Before the analyses, the samples were ground to a fine powder using a ball mill. Then, 5 g of each sample were digested by lithium-borate fusion which is a highly aggressive dissolution method being effective for most refractory and resistive mineral phases and supporting real total element concentration values. Besides lanthanides (except Pm), some refractory elements and actinides (Ba, Be, Co, Cs, Ga, Nb, Rb, Sn, Sr, Ta, Th, U, V, W, Y, Zr) were also analysed. For data evaluation, several lanthanides not used for sediment tracking were also used for comparison as reference elements (La, Ce, Nd, Eu, Tb, Dy, Tm, Lu).

Table 1. *The results of quality control and quality of analyses.*

Differences between the analysed and referred concentration values (%)								
REE used for tracking	Pr	Sm	Gd	Ho	Er	Yb		
	2.0 ± 9.6	-8.5 ± 11.6	0.0 ± 13.2	0.1 ± 10.4	6.9 ± 11.8	1.3 ± 7.2		
Reference REE	La	Ce	Nd	Eu	Tb	Dy	Tm	Lu
	9.6 ± 9.7	0.6 ± 5.2	-5.8 ± 12.5	-12.5 ± 12.6	-6.1 ± 8.7	7.6 ± 19.0	-7.6 ± 11.0	2.8 ± 8.5
Relative standard deviations of the parallel analyses (%)								
REE used for tracking	Pr	Sm	Gd	Ho	Er	Yb		
	2.5 ± 2.4	2.7 ± 2.4	2.4 ± 1.7	3.5 ± 4.5	3.8 ± 2.3	3.2 ± 2.6		
Reference REE	La	Ce	Nd	Eu	Tb	Dy	Tm	Lu
	2.8 ± 2.5	2.8 ± 2.4	2.6 ± 2.4	3.4 ± 2.4	2.7 ± 2.3	2.4 ± 2.1	3.7 ± 3.4	3.4 ± 2.7

Altogether, 386 soil and sediment samples were analysed according to the following sample types: background soil samples before tracking (29), soil samples after tracking (226), samples from the rills formed on the plots (26), and runoff sediment samples (105).

For the quality control of the analyses, nine additional standard soil and rock reference materials were also analysed in parallel. The results of quality control and quality of analyses are summarized in Table 1. The control analyses showed very small differences compared to the reference values. For Pr, Yb, La, Ce, and Lu, all analysed concentration values were within the reference ranges of the standard materials. For Sm, Gd, Ho, Er, Nd, and Dy, the analysed concentration values were out of the reference ranges of the standard materials at one single case, and this was found at two cases for Eu, Tb, and Tm. Additionally, 17% of the samples were analysed in parallel (66 samples). The relative standard deviation of the parallel analyses was mostly less than 5%. The results of the quality control showed the high reliability of the analytical data.

Statistical analyses

Descriptive statistics of the data set were carried out first. The following calculations were carried out to show if the REE concentrations of the given sample groups are significantly different. The values of skewness and kurtosis between -1 and 1 were verified to provide the assumption of normality when the D'Agostino-Pearson test was significant. As the data sets showed normal distribution, t-tests were used when two groups were compared, and one-way analyses of variance test were used when more than two groups were compared. In the latter case, the Tukey test was used as a post-hoc analysis. The significance level of each test was set to $\alpha = 0.05$. The statistical analyses were carried out using the StatistiXL add-in of the MS Excel software.

Reference REEs

Eight REEs not used for tagging (La, Ce, Nd, Eu, Tb, Dy, Tm, Lu) were used as reference elements while evaluating concentration values of the tracer REEs in the soil and eroded sediment samples. Sometimes significant differences between these values and those of soil background point to the fact that tracer concentration values above background concentration do not automatically refer to origin from tagging. Different behaviour observed among these 8 elements showed the importance of using carefully selected reference elements. This issue will be discussed in detail in the following chapters.

Results

Plot experiment

Background REE concentrations

Background REE concentrations are 8.31 ± 0.49 mg/kg for Pr, 5.80 ± 0.38 mg/kg for Sm, 5.15 ± 0.25 mg/kg for Gd, 0.98 ± 0.06 mg/kg for Ho, 2.84 ± 0.16 mg/kg for Er, and 2.89 ± 0.15 mg/kg for Yb. The t-test showed that there is no significant difference between the background REE concentrations of the PT and that of the CT plots (Fig. 3). One-sample t-test also showed that there is no significant difference between the REE concentrations of the different sampling points with only one exception where significantly lower REE concentrations were found as compared to all other sampling points. In this case, between 86 and 95% of the background REE concentrations were found, which is still very close to the background values and does not require the modification of the tracking procedure. These features are also characteristic of the reference elements. Based on the background soil REE concentrations, the study area can be regarded as geochemically homogeneous, which is ideal for REE tracking studies.

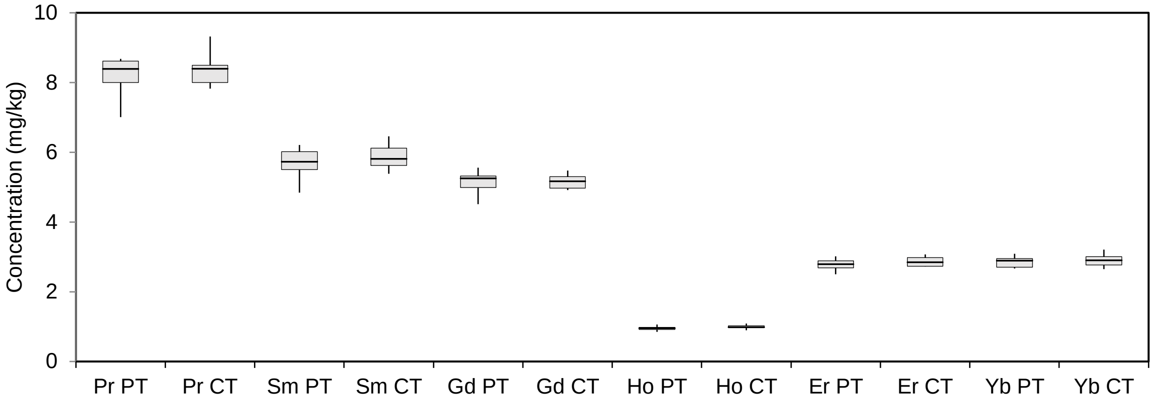


Fig. 3: *Box-plot of the background soil REE concentrations in the PT and CT plots (mg/kg).*

Sediment redistribution within the plots due to water erosion and tillage

According to the results of the ICP-MS analysis of the samples, tagging of the PT plot was successful and well designed as REEs used could be detected in significantly higher amounts than the background concentrations even after three years after tagging from the samples of the last date. However, raising background concentrations by two orders of magnitude, instead of one, as it was carried out in this experiment, would make data evaluation easier but would however also increase the costs of tagging of course.

On the CT plot, tracers can be clearly detected in the samples of the first four sampling occasions, except the case of Sm which was not found in convincingly elevated concentration in the samples. This implies that the mixing effect of the cultivation on this plot was not efficient, though the cultivator used here was expected to mix the upper 8 cm sufficiently. Similarly to those found by Steven and Quinton (2008) during their field experiment, the majority of the dispersed REOs remained in the top 0,5-1 cm of the soil without mixing and were thus easily washed away by the runoffs, which happened the fastest in the downslope area in the cases of Sm and Yb in our case.

The conception of not sufficient mixing is also confirmed by the fact that Ho could be detected in high concentration (>background concentration + 20%) in the samples for the longest time, still two and half years after tagging. This REE had the lowest background concentration among the selected elements so its concentration increase was the highest due to the tagging.

In the later (5^{th}-8^{th}) sampling campaigns, significantly higher value could not be found even in the tagged stripes (except Ho). This also can be caused by the imperfection of mixing described above.

Measured REE concentration of samples from the PT plot was in all cases significantly higher (2-23 times higher) on the tagged spots compared with the “untagged” ones (“untagged” means here the spots not tagged with the REE in question). The rate of the difference varies for the different elements increasing in the following order: Sm, Pr, Gd, Yb, Er, Ho. Similarly, a significant difference could be observed on this plot between the REE concentrations of the tagged spots and the background concentrations of the soil with similar proportions and tendencies among the elements. REE concentrations of the tagged spots of the PT plot do not change with time confirming that tracer amounts applied on this plot were high enough to persist for three years. Measured concentration values showed, however, large deviation which can be explained by the uneven homogenizing effect of agricultural cultivation. Moreover, dilution effect of soil translocation from untagged areas also influenced the concentrations.

10% higher REE concentration values compared to the maximum of the background concentrations were measured in the cases of 6.3% of the untagged spots (Fig. 5). These points were in most cases in the neighbouring tagged area referring to sideward soil replacement probably due to contour tillage operations. In these sampling points 1.3-1.9 times background concentrations were measured, which values were usually significantly higher than background concentrations.

Similar statements can be done for the CT plot, however, differences in concentrations are smaller in each case. REE concentrations of the tagged spots are also here significantly higher (1.1-3.4 times) compared with the untagged spots (Fig. 4). Proportions concerning the individual elements are also like those of the PT plot. REE concentrations of the tagged spots showed large variation and do not show a trend with time, either here.
In the case of the untagged spots, 3.8% of the samples have at least 10% higher REE concentrations compared to background concentration, in most cases near the respective tagged stripes (Fig. 6). At these sites, REE concentrations are 1.1-1.5 times background concentrations which was found to be a significant difference.

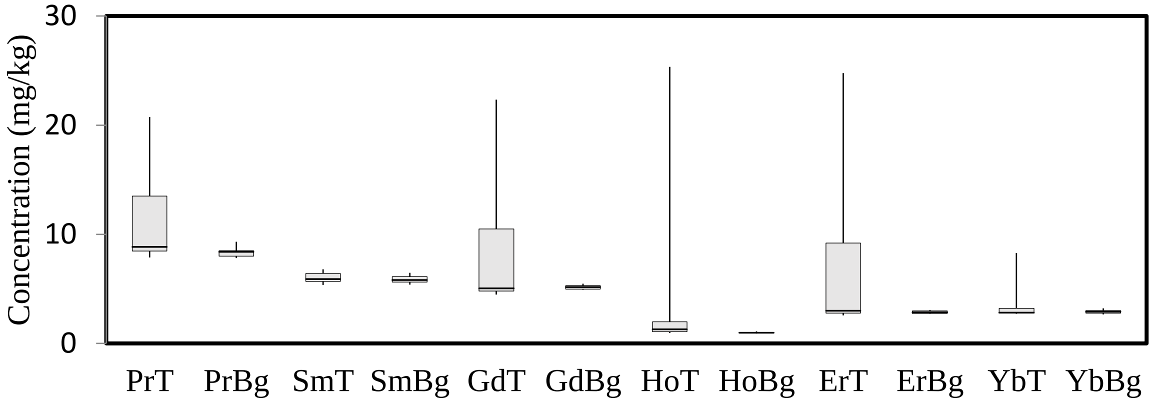


Fig. 4: *REE concentrations of the tagged stripes and REE background concentrations on the CT plot. The difference is always significant.*

Differences between reference REE concentrations of the samples and their background concentrations are not significant on either plot being in most cases lower than 3%. However, in very few cases (in 0,26% and 0,68% of the cases on the PT and CT plots, respectively) it could be observed that measured concentration exceeded background maximums by more than 10%. This implies that the difference is not a resultof an analysis error. Although the difference is not significant.

Fig. 5: *REE tracers found in soil samples at the different samplings in the tagged stripes and the rills of the PT plot in a higher amount than background maximum +10% at the different sampling occasions. Tagged stripes are the coloured rectangles with element symbols (PT1/1 - PT1/2, PT2/1 – PT2/2…. are the parallel sampling points of the individual tagged spots). Quadrats with element symbols denote the REEs found in the sample from the given sampling point. Size of the quadrates symbolise the quantity of REE found in the sample:* Pr *- above background maximum + 20%;* Pr *- above background maximum + 10%. Quadrates without the tagged stripes represent the samples of the rills marked with black lines starting downwards from the tagged stripes. (Representation of the tagged stripes are not to scale on this figure.)*

Fig. 6: *REE tracers found in soil samples at the different samplings in the tagged stripes of the CT plot at the different sampling occasions. See legend at Fig. 5.*

Considering that reference REEs were detectable in the case of some elements (Lu, Tm) in the majority of the samples above background average and a remarkable proportion of the samples from the plots above background maximum (Table 2), tracer REEs found in the samples were recorded as deriving from dispersion with values above background maximum + 10% (Fig. 5, 6).

Without this consideration, handling values above background maximum as deriving from tracing would produce significantly different results. The difference is clearly visible when comparing Figure 7 and the relating parts of Figure 5 and 6.

Table 2: *Proportion of the samples from the plots with reference REE values above background concentrations.*

	% of all samples	**La**	**Ce**	**Nd**	**Eu**	**Tb**	**Dy**	**Tm**	**Lu**
PT plot	≥ background average	18,6	17,9	78,1	61,5	51,0	61,5	88,5	97,9
	≥ background average + 10%	0,0	0,0	11,5	1,0	0,0	11,5	1,0	2,1
	≥ background maximum	2,1	2,1	9,4	7,3	26,0	14,6	13,5	15,6
	≥ background maximum + 10%	0,0	0,0	0,0	0,0	0,0	0,0	1,0	1,0
CT plot	≥ background average	7,3	8,3	37,5	55,2	46,9	39,6	80,2	92,7
	≥ background average + 10%	0,0	0,0	1,0	6,3	2,1	1,0	1,0	1,0
	≥ background maximum	0,0	0,0	1,0	1,0	5,2	0,0	1,0	5,2
	≥ background maximum + 10%	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0

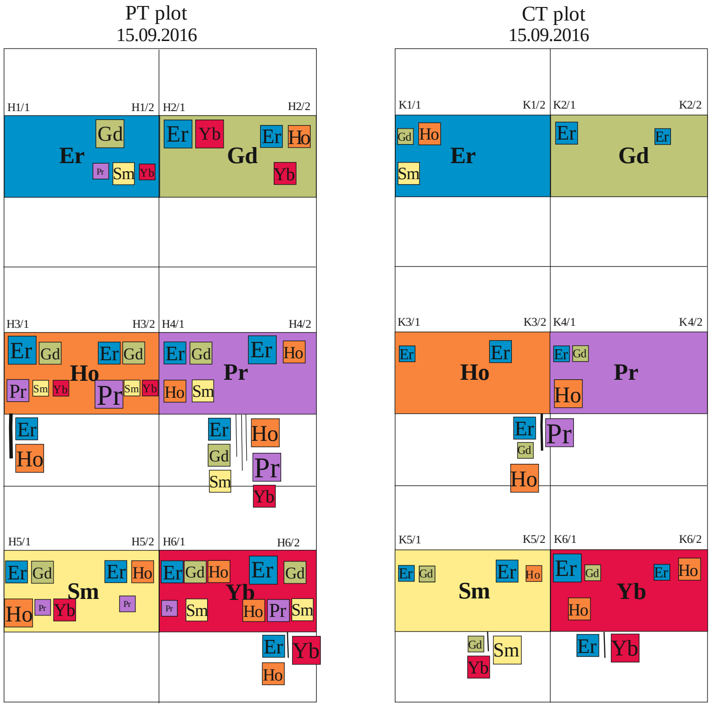


Fig. 7: *REE tracers found in soil samples at the different samplings in the tagged stripes and in the rills of the PT and CT plot in higher quantity than background average + 10%.*
Quadrats with element symbols denote the REEs found in the sample from the given sampling point. Size of the quadrates symbolise the quantity of REE found in the sample: Pr *- above background maximum + 10%;* Pr *- above background maximum;* Pr*- above background average +10%. See legend at Fig. 5.*

As the specific effect of tillage in soil displacement was not examined in this experiment, its effect in downslope soil movement can not be evaluated here, but it can be recognised very well in the horizontal translocation demonstrated by the presence of dispersed REEs across the plot.

REE concentrations in the sediment of the rills

Rills formed rarely within the plots during the study period (only in two of the sampling cases) which is in harmony with the observations during the 17 years of the experimental site. As these rills did not cut below the tracing depth, they do not affect experimental results. Their formation was affected not only by the amount and intensity of precipitation but also by the presence and state of the plant cover and the soil disturbance by the actual agricultural activities. In the summer and autumn of 2016, by maize cover, several rills were developed in a similar pattern on both plots. Although the number and pattern of the rills formed on the two plots were similar, conspicuous differences could be observed in their morphology. The length, width, and deepness of these linear erosion forms were found to be more robust on the PT plot. Altogether, 19 and 6 sediment samples were collected from the surface 1 cm of these channels for REE analyses in the June (I.) and September (II.) of 2016, respectively. Rills were sampled preferentially outside of the tracked soil stripes and longer ones on two or three points.

Measured concentrations of the REEs used for tracking above the given rills were always higher in the rill samples than the background soil values, although the difference was not always significant due to their very high variation among the different sampling points. Enrichment factors (the ratio of the concentration of the given REE in the rill sediment and the background soil) of the elements used for tracking above the rills show the strongest enrichment in the case of Ho. On average, these concentration values are 3.0-times higher than the average background concentration in the case of Pr, and these ratios are up to 2.1 for Sm, 3.7 for Gd, 21.0 for Ho, 6.6 for Er and 5.0 for Yb. Enrichment factors of La, Eu, Lu as reference REEs show that the increased values of the previous ones are subsequent upon the raised concentrations by tracking (Table 3., Fig. 8).

Table 3. *Enrichment factors of the samples from the rills of the REEs used for tracking directly above the rills and highest values in the rill samples of three reference REEs (I.: sampling in June 2016; II.: sampling in September 2016).*

			Er	**Gd**	**Ho**	**Pr**	**Sm**	**Yb**
Enrichment factor	PT plot	I.	7,81	1,26	22,57	2,80	3,33	2,82
			1,92		2,28	1,44	2,26	1,17
								1,18
		II.			33,50	2,81		8,79
	CT plot	I.	10,00	6,10	32,83	5,10	1,52	9,36
		II.			14,00		2,39	6,89

			La	**Eu**	**Lu**
Enrichment factor	PT plot	I.	1,10	1,15	1,25
		II.	1,00	1,09	1,18
	CT plot	I.	1,05	1,15	1,23
		II.	0,98	1,13	1,2

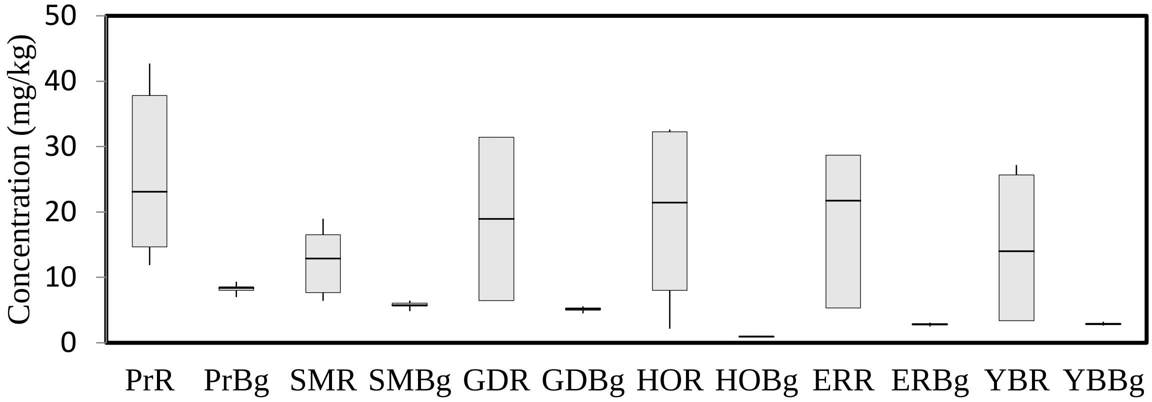


Fig. 8. *Concentration of REEs was significantly higher in the sediment of the rills directly below the tracked stripes compared to the background soil concentrations. R = rill, Bg = background.*

In some cases, concentration enrichment was found for other REE than those used for tracking the stripe lying directly above the sampling point (Fig. 5, 6). Between 52 and 92% of the concentration values exceeded the 95% confidence interval of the background REE concentrations, and between 20 and 76% of the concentration values were higher than the maximum background REE concentrations. The highest ratios were found for Er and Ho, which exhibited the lowest background concentrations among the REEs used for tracking. In the cases of these two elements, REE enrichment was observed not only in the closest neighboring tracked stripes but also in samples of farther rills (both sideways and downwards). As expected, almost every REE showed elevated concentrations at the downslope area, whereas only cross-contamination could be observed at the highest parts. Additionally, these concentration values were only 10-30% higher than the maximum background values, except for Ho, where up to 6.5-times higher values were also observed.

No difference was found between the measured concentration values at the two sampling dates. For the reference REEs, very low differences (below 5%) were found between the concentrations of rill samples and background soil. Similarly to the REEs used for tracking, no difference was found between the measured concentration values at the two sampling dates.

Analysis of the REE content of the sediment leaving the plots

REE concentrations measured from the sediment collected at the outlet of the plots after each rainfall event must have been corrected for soil background concentrations. Corrected REE concentration (Cc^j_i) can be calculated according to the followings for each tracer (*i*) for each rainfall event (*j*):

$$Cc^j_i = Cm^j_i - Cb_i$$

where Cm^j_i is the measured concentration of tracer *i* in the sediment left the plot after rainfall event *j* and Cb_i is the background concentration of the tracer REE *i*.

Tracer REE concentrations measured in the sediment leaving the PT plot were always significantly higher (1.1-1.5 times) than the soil background concentrations, except in the case of Ho with concentration values 2.73 times higher than the soil background concentration (Fig. 9). It should be noted that background concentration was the lowest in the case of this element so that REE amount increased by dispersion was in its case in the highest degree.

However, concentrations in the sediment are usually higher than soil background also in the case of the reference REEs, though to a lower degree. No difference was detected only for Tm. Higher values were measured for La and Eu: in average 1.13 times soil background values.

A similar phenomenon was observed on the CT plot with the difference that Yb (and Lu from the reference elements) showed no significant difference here (Fig. 10). In the other cases the difference was 1.09-1.23-fold excluding again Ho with a 1.81-fold higher value in the sediment. Similar values were however found at some of the reference elements, as well: La, Ce, Eu, Tb showed 1.10-1.20 times higher values in the sediment than the soil background concentrations. Comparing to the maximum of the background values, enrichment of Sm, Ho, Er and Yb are obvious: these values are exceeding it in most cases by more than 10%. On the conservation plot only Sm and Ho acted similarly.

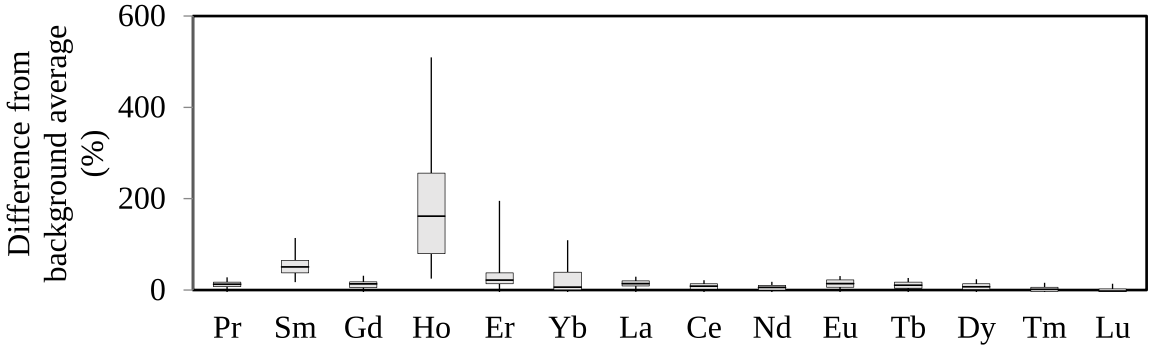


Fig. 9: *Difference of sediment REE concentrations from soil background concentrations on the PT plot.*

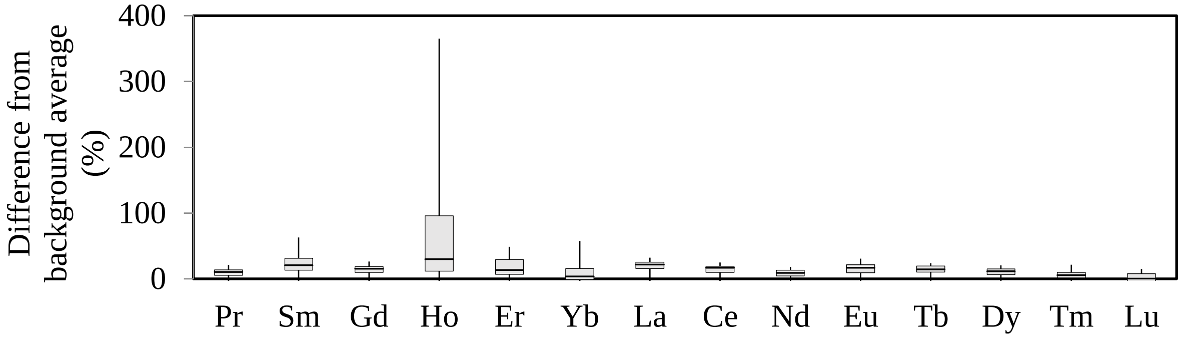
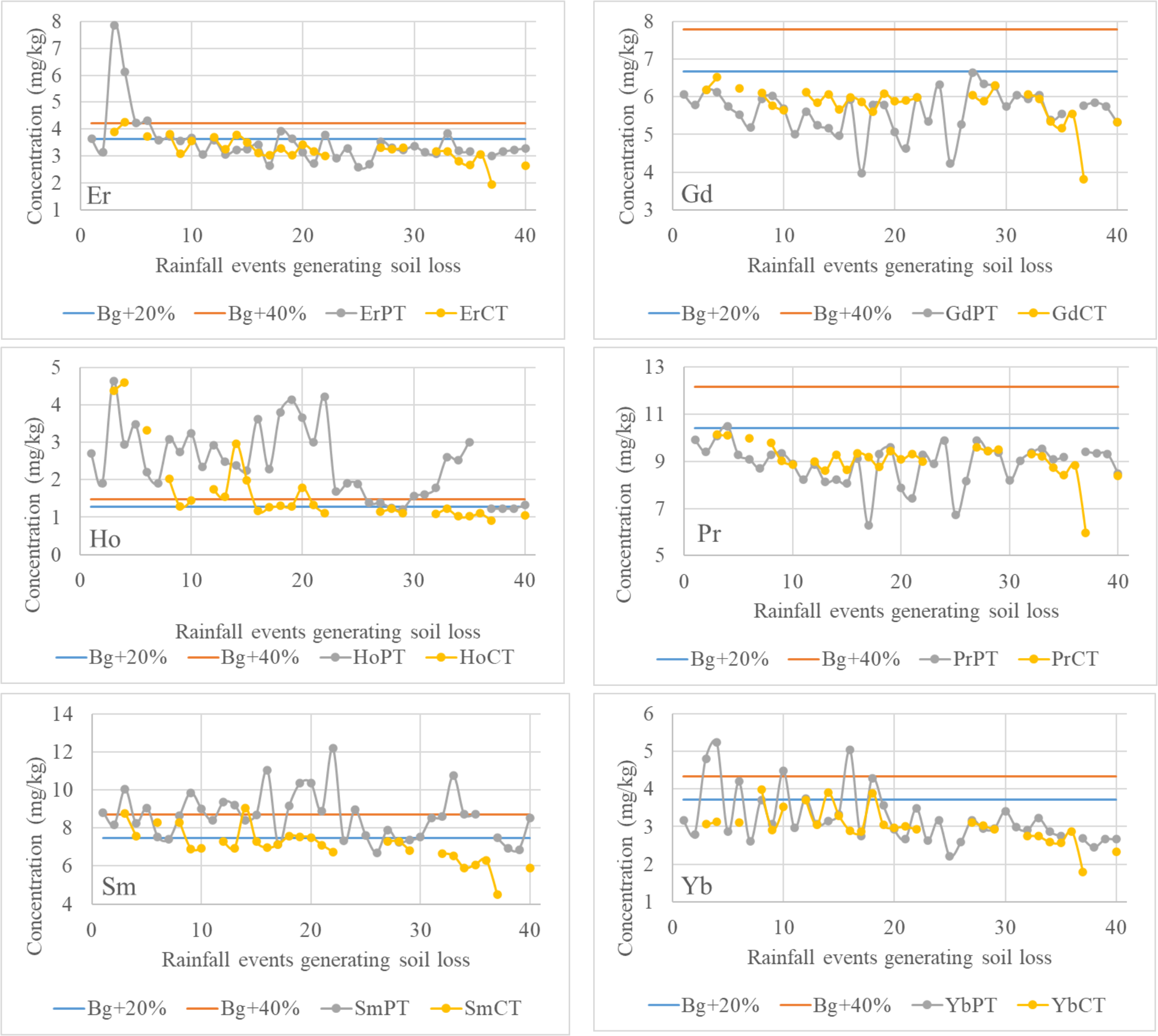


Fig. 10: *Difference of sediment REE concentrations from soil background concentrations on the CT plot.*

Analysis of the reference element’s concentrations measured in the sediment samples drew attention to the importance of the proper choice of the limit above which concentration values of tracer REEs can be regarded as a result of tracing with high reliability. Moreover, appropriate selection of the reference elements is of high importance as well. Fig. 11 shows the very different behaviour of these REEs: while concentration values of Lu in the sediment never reached soil background maximum +10% (even background maximum in only two cases out of 56), Eu and La exceeded background maximum +10% in 39 and 38% of the samples, respectively. In turn, they both reach background maximum + 20% in only two cases out of the 56. According to these observations, without examination of the behaviour of the tracer elements of this aspect, measured values in the sediment samples exceeding background maximum + 10% can not be considered as the result of tagging, while those above background maximums +20% with high probability can be. However, preliminary study of the elements planned to use in an experiment of this kind would make data evaluation more certain.



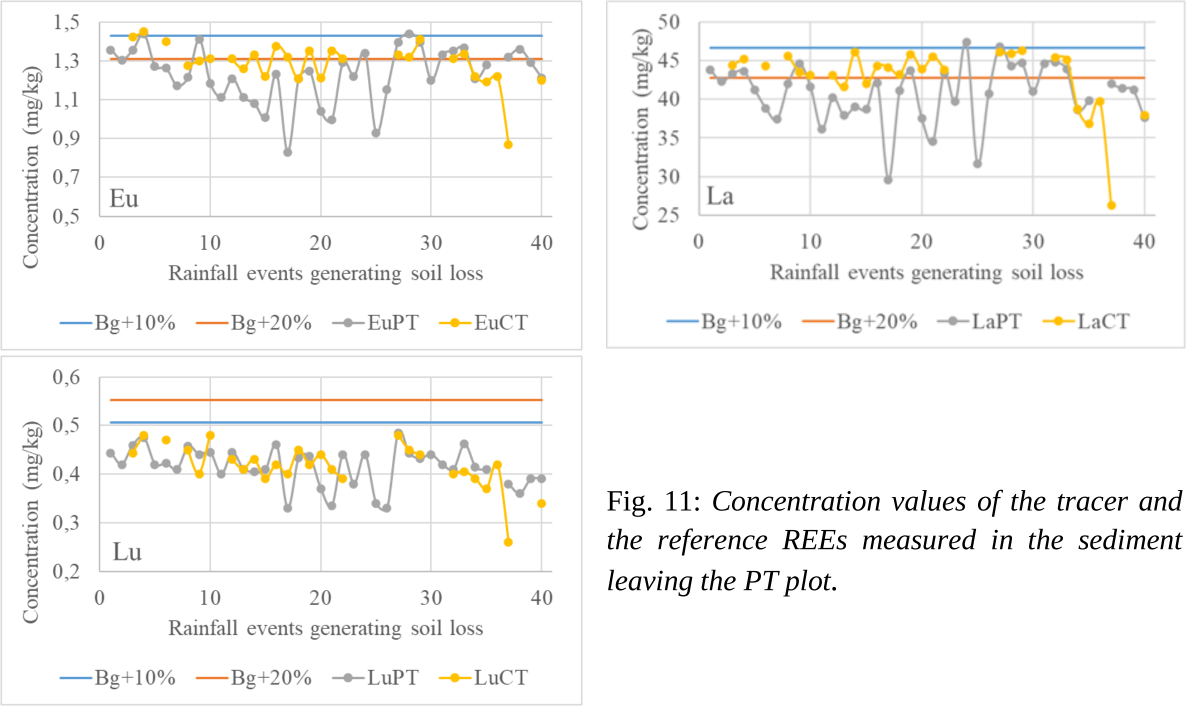


Fig. 11: *Concentration values of the tracer and the reference REEs measured in the sediment leaving the PT plot.*

Comparing REE concentrations in the sediment of the two plots significantly higher values of Sm, Ho, Er, and Yb were measured on the PT plot. The rate of the difference is between 1.1 and 1.5, increasing in the following order: Yb, Er, Sm, Ho. No significant difference was found in the case of the reference elements: it is lower than 4% in all cases so that the concentration values were equal. This verifies that differences experienced in the case of the tracer elements are the consequence of the tagging.

Analysing these data it can be stated that REE values in the sediment of the PT plot are only in the cases of Sm, Ho, Er, Yb convincingly higher than the soil background concentrations, i.e. material deriving from the tracing appear surely in the sediment leaving the plot. On the CT plot, the same can be stated only in the cases of Sm and Ho, and also in these cases to a lower extent.
Different behaviour of the REEs on the two plots are also obvious, but different amounts dispersed due to different tillage depth make the comparison more difficult.

The importance of the appropriate choice of reference elements appears here very well as the difference in behaviour of the different elements may be determining. In our case, La shows very similar, while Lu very different characteristics than the applied tracer elements. If only the latter one (and similars) would have been used as a reference element, results would suggest a more significant enrichment of all the dispersed REEs in the sediment.

It was examined how the amount of sedimented REEs changed with the singular rainfall events. Reference REEs show very similar distribution, so that this can be considered as a basic condition and any difference compared to this in the cases of the tracer elements can report that the sediment originates from a tagged stripe.

Correlations were calculated among the REE amounts eroded by the single precipitation events on both plots. On the PT plot amount of reference elements correlate very well with each other, showing that these elements probably were transported together, appointing thus the background. Pr and Gd correlate well with them, implying similar behaviour, i.e., dispersed amounts do not appear here. In the cases of Sm, Ho, Er, and Yb, however, no correlation was found. Their behaviours are consequently different, which is obviously due to the dispersed amounts. Ho, Er and Yb correlate very well also with each other, while Sm only with Ho and has a weaker relationship with the others.

On the CT plot, only Ho has a very different behaviour compared to the reference elements, which however correlates with some of the tracer elements (Pr, Sm, Er), but not with the reference elements. All other REEs show a very strong correlation, i.e., dispersed elements behave like the reference ones. This means that it can not be stated here that concentrations changed due to tagging. However, at some rainfall events, there are differences that may indicate erosion deriving from the dispersed material.

The relationship between the REE concentrations in the runoff sediment and the amount, intensity and length of the precipitation was also studied. Remarkable change in REE concentration was regarded when the change representing the average amount (24.2 mm), length (11 hours) and intensity (3.3 mm/hour) of the precipitation resulted in a change of REE concentration of 10% at least.

No remarkable change in the REE concentration in the runoff sediments was found with the increase of the precipitation amount in the most cases. The only exception is again Ho, of which concentration showed a slight decrease (6%) in the PT, and a remarkable increase (16%) on the CT plot.

Similarly, in most cases no remarkable change in the REE concentrations was observed with the increase of the precipitation length. However the concentration of Sm and Er in the runoff sediment showed nearly 10% increase with the increase in the precipitation length, whereas this change in Ho concentration was remarkable (14%) on the PT plot. Contrarily, Ho also showed a remarkable decrease (nearly 50%) in its sediment concentration on the CT plot with the precipitation length.

Again, an increase of precipitation intensity did not result in a remarkable change in the concentrations of the REE in the runoff sediment. The only exception is still Ho, which exhibited a remarkable increase (14%) in its concentration with precipitation intensity.

Consequently, the runoff sediment concentrations of REE could not be related to the precipitation amount, length and intensity in most of the cases. The only exception is Ho on the CT plot, where its concentration decreased remarkably with precipitation amount and length, and it increased with precipitation intensity.

Rainfall simulation experiments

To gain additional information on soil redistribution extending the originally planned data deriving from the field samples, a rainfall simulation experiment was also carried out next to the studied plots, also to compare the loss and redistribution of soil sediments under conventional and conservation agricultural practices. Five different rainfall intensities (up to 80 mm/h) were applied to both plots. Sources and pathways of sediments within the two plots were studied using REE-oxides as tracers. Approximately 1,000 mg/kg of Er2O3, Ho2O3 and Sm2O3 (calculated to the upper 1 cm of the soil) were dispersed to the soil surface with banded distribution; each transversal band covered the third of the surface area of the plots. The concentration of the REE-oxides in the sediment leaving the plots, and that of the surface soil before and after the experiment were analysed by a portable X-Ray fluorescence spectrometer.

Significant sediment losses were found for both plots after the experiments, with slightly different characteristics between the PT and CT plots. The highest difference in loss of added REEs was found in the upper third of the plots with 81 ± 19% in the PT and 71 ± 21% in the CT ones. These values have been equalized downwards with almost complete losses in the lower third of the plots (99 ± 2% and 97 ± 4%, respectively). Only a very small part of the removed sediment has been accumulated in the lower parts of the plots, they rather mostly left the study area. These accumulation zones showed patchy distribution and could be characterized by slightly higher REE concentrations in the CT plot. Also, large variances in the REE amounts removed from the study plots were found during the experiment with slight differences between the two plots. Thanks to the use of the REE tracers, information was received not only on the sediment amounts leaving the area due to the individual rainfall events but also on the source of them within the plot. Our data also suggest that differences between the PT and CT plots can be observed even in the short term.

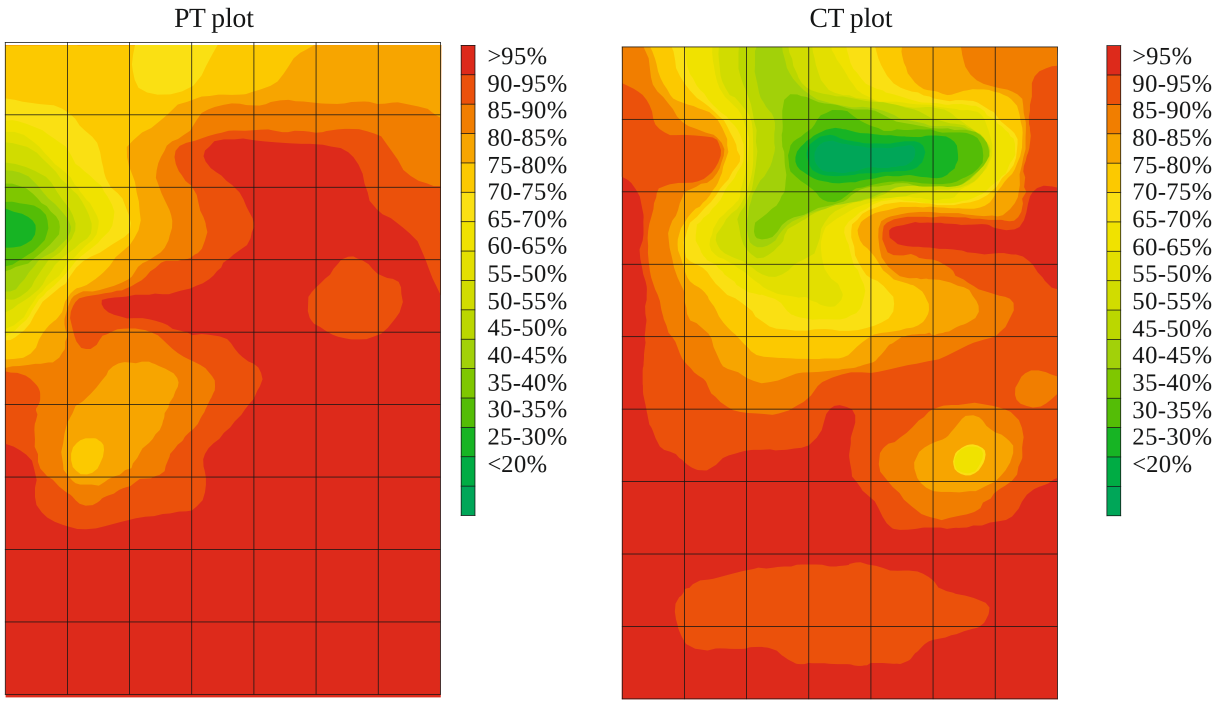


Fig. 12: *REO loss (%) after the rainfall simulation experiment on the PT and CT plots*

Moreover, laboratory rainfall simulation was also carried out at the Eötvös Loránd University to get data also from small scale plot. The main objective of this study was to examine the feasibility of REO tracking combined with 3D surface modelling and soil crust analysis by scanning electron microscopy.

The laboratory plot was filled with soil from the Szentgyörgyvár research station where also the field experiment was carried out. Its surface was divided into horizontal stripes and tagged with four different REOs. Sediment leaving the plot was collected and samples were taken from the surface after the experiment to follow redistribution within the plot.

Four 40 mm/h rainfall simulations (divided into two blocks) were conducted at 9% slope steepness. In a block, two successive simulations were run on a tilled, and then, on a crusted surface. Before the first rainfall simulation of a block, the tilled surface was prepared by hoeing and application of four REO tracers (Pr6O11, Sm2O3, Ho2O3 and Yb2O3) to the freshly tilled surface. REOs divided the parcel into two back, and two front sub-parcels. The REE runoff content was measured by pXRF, while the redistributed REEs were measured by SEM on polished crust samples taken after the second experiment in each block. Additionally, before and after 3D models of the surface were created for determining runoff direction and redistribution pattern.

According to the REE content of the soil loss samples, the soil washed down from the front sub-parcels, while back parcels started contributing to soil loss only during the second block experiments. The surface microtopography changed between the experiments. The runoff path from the back sub-parcels headed to one side of the parcel. This strong, cross-side runoff pattern explained the lack of the back sub-parcel REEs in the soil loss. Meanwhile, in the crust samples, several forms of the redistribution were identified. The REEs of the back sub-parcels were found in the samples that were collected in runoff paths, and the leaching pattern became traceable with REOs. Moreover, we were able to reconstruct the original surface easily on SEM images.

It could be concluded that REO tracing applied with surface modelling and runoff direction modelling, and SEM analysis of crust samples was suitable to follow the path of water runoff, and explain the soil redistribution pattern horizontally and vertically. Intensive erosion and sedimentation were detected parallel, whereas the REOs were useful for soil redistribution estimation, and promising for soil loss value estimation and tracking infiltration in the soil. Small-scale redistribution seems to be independent from the overall slope length and incline emphasizing the role of microtopography. Most of the delivered particles were deposited within decimetres resulting small changes in the original REO pattern. However, the investigation was carried out under unique topographical and soil conditions, the sensitivity of this method under various environmental circumstances is not yet known.

Results are published and can be read in detail in: Szabó, J.A., Király, C., Karlik, M., Tóth, A., Szalai Z., Jakab, G.. Rare earth oxide tracking coupled with 3D soil surface modelling: an opportunity to study small-scale soil redistribution. J Soils Sediments 20, 2405–2417 (2020). Open access: https://doi.org/10.1007/s11368-020-02582-7

Analysis of concentration data of elements not used for tracing

During the analysis of the soil samples, concentrations of 24 elements not used for tagging were also examined in the external laboratory. Comparison of the quantity of these elements in the soil on the plots and in the eroded sediment can help in characterisation of soil erosion processes. The number and location of samplings are the same as those of elements applied for tagging.

The concentrations of these elements on both plots and on the 12-12 sampling points within the plots were compared first. Ba and Rb showed significantly (but still only by 6-9%) higher concentration values in the downslope area of the CT plot, which may refer to light enrichment of feldspars in this part of the plot. On the whole, the study site can be considered as geochemically homogeneous on the basis of the concentration values of the 24 trace elements.

Although statistically significant difference could be detected in many cases between concentration values measured in the rill samples and the soil samples from the plots (Sr, Hf, Nb, Rb, Ta, Th, U, Zr, Ce, Nd and Eu on the PT, and Co, Hf, Ta Th, Zr and Eu on the CT plot), geochemically no real differences were found, either here.

Concentrations in the eroded sediment and the soil samples were also compared. Fig. 13 shows differences in % between the average concentrations measured in the two types of samples on the two plots. Hf and Zr showed 40-50% lower values in the sediment on both plots. These elements accumulate typically in heavy minerals resistant to weathering (mainly in zircon), which are in addition usually of relatively big size in soil (usually in silt fraction), so that selectivity of erosion may affect them more. Oppositely Ba, Co, Cs, Ga, Rb, Sn, Sr, Th, V, and W showed enrichment in the eroded sediment compared to original soils on both plots, with averages between 16 and 111%. These elements are partly common components of feldspars and their weathering products (e.g. Ba, Cs, Ga, Rb, Sr), which are due to their lower density or particle size more exposed to erosion. Co, Sn, V, and W are rather related to iron minerals, which have though higher density but are often of very small particle size, belonging thus also to the more erodible fraction of soil.

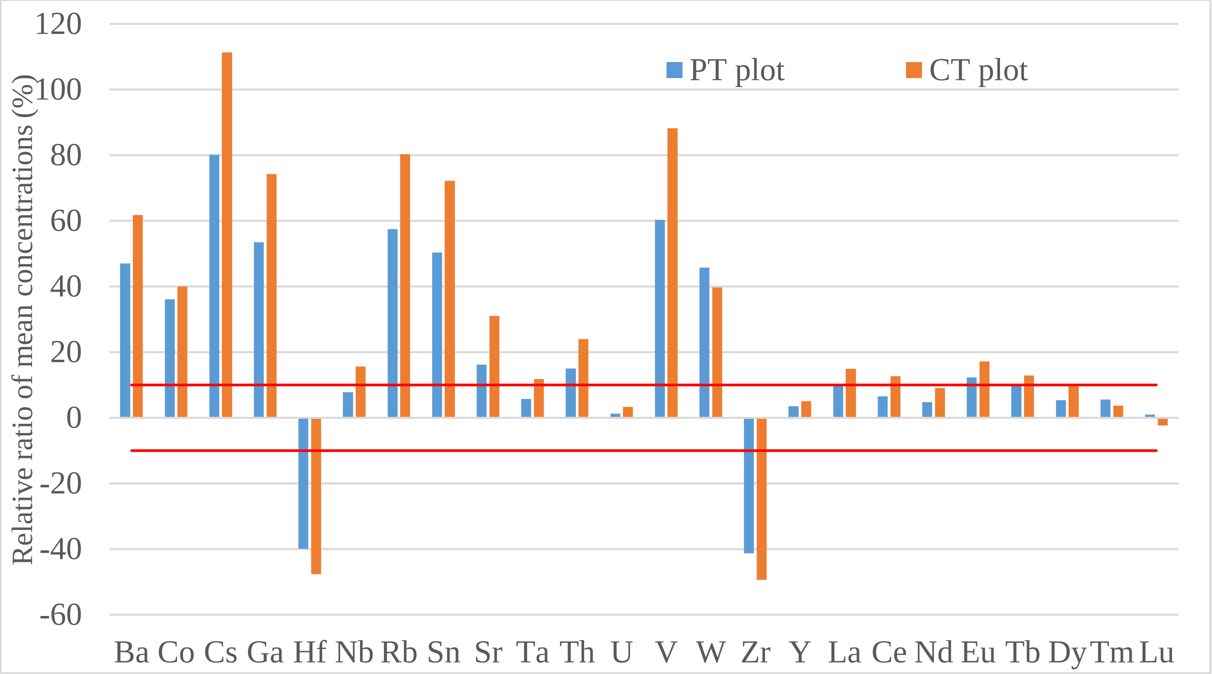


Fig. 13: *Difference in % between the average concentration of trace elements not used for tracking in the soil samples from the plots and in the eroded sediment.*

Concentration values in the eroded sediment from the two plots did not show significant difference in the case of most elements. Cs, Ga, Rb, Sn, Sr, and V had 10-15% higher concentrations on the CT plot, while significantly lower concentration values of Zr were detected here, with an average of 13%. As suggested from these data, elements related to more erosive phases enriched more, while those related to less sensitive phases enriched less in the sediment deriving from the CT plot as compared with that of the PT plot. This may refer to the difference between the intensity of the erosion processes occurring on the two, differently cultivated plots.

Correlation was also calculated among element concentrations measured in the eroded sediments. Most elements show strong ($r > 0.5$, if $\alpha = 0{,}05$) positive correlation with each other. Exceptions are Hf and Zr showing strong negative correlation with most of the elements, excepting each other and rare earth elements where no correlations exist. Tm and Lu correlate positively only with each other, while U only with Sr and Y. Results show that most of the studied elements move together in the eroded sediment, except Hf and Zr which enriches in heavy minerals, moreover U and some REEs show different behaviour. Behaviour of the latter ones can not be explained adequately based on the existing data.

Relationship among the behaviour of the elements was somewhat more clear when analysing with correlation the rate of average enrichment of the elements. Elements were divided into four groups in both plots. To the first group belong Ba, Cs, Co, Ga, Rb, Sn, Th, and V. These showed a strong positive correlation ($r > 0.75$) with each other. On the PT plot, these elements showed positive correlation ($r > 0.5$) also with Nb and the rare earth elements, while on the CT plot only with W. To the second group belong the rare earth elements, mostly showing strong positive correlation with each other, except Tm and Lu, which showed correlation only with each other. Additionally, they exhibited positive correlation with Nb and Y on the PT plot, and with U and Th on the CT plot. To the third group of the elements belong Zr and Hf, showing always very strong positive correlation ($r > 0.99$) with each other, and strong negative correlation ($r < -0.75$) with the elements of the first group. On the CT plot, Zr and Hf showed positive correlation also with Tm and Lu. To the fourth group of elements belong those usually not correlating with the others or with only a few of them: Sr showing only weak positive relationship with REEs; Ta showing weak positive relationship with the REEs only on the PT plot; U and W showing correlation with the other elements only on the CT plot, while on the PT plot with only very few from the first group. These relationships are usable only as guidelines without exact knowledge on the host phases of the elements, but it is obvious that erosion affects the movement of the several phases mostly very similarly on the two plots. There are however a few elements showing definite difference, indicating that the movement of some phases may be affected by erosion differently on the two, differently cultivated plots. Further study of these may be of interest.

The relationship between trace element concentrations in the eroded sediment, and quantity, duration, and intensity of precipitation was also studied. Precipitation event of average quantity, duration, or intensity causing concentration change in the sediment of at least 10% of background value (increase or decrease) was considered as a significant change. The increasing quantity of precipitation causes minor changes in most cases, usually increase in concentration, except Hf, Zr, and W, where the decrease of concentration can be observed. Significant difference was however observed in only a few cases, e.g. in that of Cs and Ga on both plots and Ba, Rb, and Sn on the PT plot. These elements showed an increase in concentration, while W showed significant decrease in concentration by increasing the quantity of precipitation on the CT plot. In addition, many of the elements showed minor (5-10%) changes, while concentration did not change (or only on the PT plot) in only a few cases (Ta, Y, La, Ce, Nd, Dy). Significant difference in element concentrations between the first and fourth quartile calculated from precipitation quantity data appeared only very rarely (in the case of Ba and Tb on the PT plot and of Co, Cs, Ga, Rb on the CT plot). With the increasing duration of the precipitation events minor changes, usually increase in concentration can be observed on both plots. Hf is an exception again showing a not significant decrease in concentration on both plots. Ba, Cs, and V are characterised by significant increase in concentration on both plots, while Ga, Rb, Sn, W, and Zr on the PT and Co on the CT plot. Most elements show again only a slight increase of concentration mainly on the PT plot, while only a few elements show no quantity change on either plot (U, Y, Nd, Dy, Tm). By increasing precipitation intensity, element concentrations usually decrease in the eroded sediment, mainly on the PT plot, except Hf and Zr with increasing concentrations. Changes in this case are however never significant, reaching rarely the 5% change in concentration (only Cs shows major decrease on the PT plot). Differences between the two plots of different cultivation can be thus noticed also by these data, but these trends are often weak, or even based on the more frequent lack of change in the case of the CT plot, which deviations are statistically hard to verify.

Patterns of element concentrations illustrated as a function of the individual precipitation events were also compared. The two main groups of elements: the group of Hf and Zr and that of the other elements are well marked also here. Previous ones show their maximum mostly in the cases where the others show their minimum, demonstrated also by their negative correlation with the other elements. In the latter group, some elements may also have further anomalies in the pattern, differing from the pattern of their group. This is conspicuous particularly on the PT plot. Elements representing such a different pattern are Cs, Sn, W, U, Tm, and Lu which sometimes showed local maximum where other elements of their group exhibited minimum values. These anomalies do not show any relationship with the characteristics of the precipitation events. The host phases of these elements may be eroded from the soil surface during these events differently from each other.

Conclusions

This is the first study showing that rare earth oxides (REOs) are suitable tracers for several-years-long soil erosion tracing experiments under field conditions. Former experiments (Zhang et al., 2003; Polyakov and Nearing, 2004; Kimoto et al, 2006) were based on the assumption that the concentration of the tracer in bulk soil is equal to that in the eroded sediment. The results of this study showed that this assumption is false, as different REOs exhibited varying behavior, mobility to a different extent during erosion.

That is why reliable application of the REO tracer method can be suggested only under the following requirements not mentioned in previous studies:

- Though REOs are generally considered as having good potential as sediment traces due to their advantageous characteristics (Zhang et al 2001; Polyakov et al., 2004; Kimoto et al. 2006; Deasy and Quinton, 2010; Michaelides et al., 2010), this study made it obvious that rare earth elements in soils are hosted by phases showing different erdodibility, thus they may show different mobility due to erosion. Ignoring this fact can lead to wrong conclusions, endangering the reliability of the whole experiment. Study of the differences in the mobility of REEs by runoff would so be crucial for the proper choice of the elements used for tagging and of those used as reference. More knowledge in this field would make data evaluation more certain.
- We suggest that threshold concentration values instead of background concentrations have to be assessed for each tracer element to be able to define what concentration values measured in the samples can be derived from the tracing with high reliability.
- As reported among the results tagging was not fully successful on the CT plot, probably due to the inefficient mixing effect of the moderate cultivation on this plot. In the case of further experiments of this type this experience should be taken into consideration and mixing should be solved in a different, reliable way.
- It was noticed from several aspects that Er, Ho and Sm showed more intensive mobility compared to the other tracers on both the PT and the CT plots. As this parallelism between the two plots can not be explained by cultivation characteristics or analogies in relief, it must be concluded that it is caused by the different behaviour of the REEs during erosion. This recognition draws attention again to the importance of preliminary

study of the different mobility of REEs due to runoff, and of the use of well-chosen reference elements elements for reliable control of the results.

By the results of the laboratory rainfall simulation experiment it can be concluded that REO tracing applied with surface modelling and runoff direction modelling, and SEM analysis of crust samples is suitable to follow the path of water runoff, and explain the soil redistribution pattern horizontally and vertically. REOs are useful for soil redistribution estimation, and promising for soil loss value estimation and tracking infiltration in the soil. Small-scale redistribution seems to be independent from the overall slope length and incline emphasizing the role of microtopography. However, the investigation was carried out under unique topographical and soil conditions, the sensitivity of this method under various environmental circumstances is not yet known.

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