

Rare Earth Element Accumulation in Annually Flooded Soils along the Mississippi River in Southeastern Missouri

Michael T. Aide, Christine C. Aide

Department of Agriculture, Southeast Missouri State University, Cape Girardeau, USA
Email: mtaide@semo.edu

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Abstract

Throughout the history of modern soil science, protocols have been advanced to identify and characterize parent materials. More recently, rare earth element signatures have been employed to show uniformity or differences between parent materials. The morphology and evolution of the Mississippi River and its tributaries have created numerous parent materials and depositional environments. We observed representative 1) upland soils, 2) alluvial soils not receiving annual flooding and 3) annually flooded soils adjacent to the Mississippi River. Rare earth element signatures and appropriate rare earth element ratios support the premise that erosional materials from the upland soils after deposition becomes the parent material for soils on floodplains that receive upland sediment and for Mississippi River floodplain soils whose sediment originated in the upland regions, even if the sediment was partially transported by the Mississippi River. The rare earth element signatures do not corroborate the premise that Mississippi River floodplain soils obtain their parent materials from upland soils whose alluvial sediment is substantially composed of coarse-grain bedload.

Keywords

Mississippi River Alluvium, Rare Earth Elements, Sediment Provenance, REE Signature, REE Bioavailability

1. Introduction

The rare earth elements (REE) are listed as cerium (Ce), praseodymium (Pr), neodymium (Nd), promethium (Pm), samarium (Sm), europium (Eu), gadolinium (Gd), terbium (Tb), dysprosium (Dy), holmium (Ho), erbium (Er), thulium (Tm),

ytterbium (Yb), lutetium (Lu). Yttrium (Y) is frequently included as a rare earth element because of its small ionic radius and its association with REE minerals. Lanthanum is frequently with the REE's because of its association in the Periodic Table and its trivalent oxidation state and similar chemical affinity. Promethium undergoes rapid radioactive decay and is absent from the natural environment (Lee, 1992). The REE series has ground state electronic configurations with at least one 4f electron. REE³⁺ species result from having electrons removed from their d, s and f orbitals. The REE's display considerable ionic bonding character and are considered hard acids (Lee, 1992).

Europium has a ground state electronic configuration that provides enhanced stability for the Eu²⁺ species. Cerium exhibits oxidation-reduction and exists as either Ce³⁺ or Ce⁴⁺. The "Lanthanide Contraction" exists because of f-orbital incomplete electric field shielding and increases in nuclear charge on transition across the lanthanide series, resulting in increasing hydrolysis and complex stability on progression to greater atomic numbers (Lee, 1992). The light rare earth elements (LREE) consist of the elements La to Eu and the heavy rare earth elements (HREE) are comprised of the elements Gd to Lu. Some authors include middle rare earth elements MREE (Sm, Eu, Gd, Tb, Dy, Ho) (Vermeire et al., 2016).

1.1. Rare Earth Element Rock, Soil, and Water Abundances

Rare earth element soil abundances are influenced by the minerals in the parent material, texture, weathering history, pedogenic processes, organic matter contents, and anthropogenic disturbances (Aide & Smith-Aide, 2003; Aide & Aide, 2012). In a review of trace element occurrences, Kabata-Pendias (2020) compiled typical REE concentrations of selected parent materials, soils, plants, and surface soil horizons. Rock REE concentrations vary distinctly with rock classification and source area, with typical REE compositions ranging from 0.1 to 100 mg kg⁻¹. Argillaceous sediments generally have greater REE concentrations than limestones and sandstones (Kabata-Pendias, 2020).

For soils, the LREE concentrations are generally greater than the HREE concentrations and the REE concentration distribution by atomic number typically obey the Oddon-Harkin rule. Kabata-Pendias (2020) documented that REE concentrations may be slightly greater in alkaline soils than acidic soils and the clay and organic fractions may limit REE soil profile leaching. Exchange and adsorption reactions may dominate in soil and sediment accumulation. Oxidation-reduction reactions may result in cerium anomalies.

1.2. Rare Earth Elements and Soil Development

The importance of the REE's rests with their "signature", which may be defined as either the actual REE concentrations or their normalized concentrations arrayed regarding their atomic number. Normalized concentrations arise when the analytical concentrations are divided by the rare earth element concentrations

from underlying or similar parent materials. Analysis of the REE signatures typically involves evidence of fractionation, i.e., LREE/HREE ratios, La/Yb ratios, Nd/Sm ratios, and the presence of Ce or Eu anomalies. REE signatures have been compared to reveal lithologic discontinuities (Aide & Smith-Aide, 2003), aeolian or anthropogenic additions (Aide et al., 2002), and weathering intensity (Nesbitt & Markovics, 1997; Tyler, 2004; Aide, 2018). Soil profile lithologic discontinuities are changes in the parent material and typically and substantially alter soil behavior. Soil characteristics that imply the presence of lithologic discontinuities include: 1) abrupt textural changes, 2) contracting sand sizes, 3) shape and orientation of rock fragments, 4) stone lines, and 5) mineral composition (Buol et al., 1997).

Table 1 lists selected references focusing on REE fractionation.

Table 1. Supplementary articles articulating rare earth elemental fractionation in soil.

Author	Pertinent Information
Vázquez-Ortega et al. (2015)	Annual chemical denudation of REE's occur during snowmelt-driven organic carbon pulses with the REE translocated as organo-mineral colloidal species into the underlying regolith.
Vermeire et al. (2016)	The rate of REE soil loss correlated with overall elemental loss rates and intensities of mineral weathering.
Laveuf and Cornu (2009)	REE fractionation occurred because of REE release by primary mineral weathering, adsorption by organic matter and secondary minerals, and clay illuviation and redox processes.
Nesbitt (1979)	CO ₂ and organic matter supported REE carbonate complexes leaching into the regolith. In the regolith the HREE were enriched to a greater degree than the LREE.
Land et al. (1999)	In Spodosols, the E horizons were REE depleted, and REE depletion was greater for the HREE. Selective extractions demonstrated that crystalline Fe-oxyhydroxides and labile organic fractions accumulated HREE to a greater extent than the LREE, whereas humic and fulvic acids preferentially accumulated the LREE
Öhlander et al. (2000)	Nd and Sm have similar environmental chemistries, and the consistency of the Nd/Sm ratio is an effective tool to discern sediment source areas or parent material differences.
Aide and Smith-Aide (2003)	In a review, La/Yb ratio has been utilized to estimate parent material similarity.
Vermeire et al. (2016)	REE were rapidly released because of Spodosol weathering, resulting in REE soil depletion. Soil organic matter and oxyhydroxides did not alter the REE signatures.

2. Mississippi River Sediments

Many publications describe channelization and sedimentation by the Mississippi River, particularly near the Mississippi River delta. Many manuscripts feature descriptions involving natural and accelerated erosion as source materials (Hassan et al., 2017; Aide, 2021). Russell et al. (2021) noted that the Mississippi River's natural evolution involved meandering and formation of floodplains and sediment deposits. Bini et al. (2011) noted that sediments typically contain heavy metal concentrations inherited from the parent material and conditioned by soil forming processes, the number of pedogenic cycles, and human activities.

Human activities have altered the Mississippi River system with the construc-

tion of levees and dredging-channelization to reduce flood damage and alter sediment deposit potential. The Mississippi River basin has been transformed because of land use practices, dam construction, and river navigation projects. Hassan et al. (2017) developed relationships involving sediment yield per unit area and drainage area to isolate regional sediment yield patterns. Current sediment is sourced primarily along the river valleys in agricultural regions and because of stream bank and channel erosion.

Sionneau et al. (2008) proposed that the relative abundances of smectite, illite, kaolinite and chlorite were associated with large, clay-mineral provinces, such as: 1) the north-western Mississippi River watershed is smectite rich, 2) the Great Lakes area and eastern Mississippi River watershed is illite and chlorite rich, 3) the south-eastern United States is kaolinite rich, and 4) south-western Mississippi River watersheds are illite and kaolinite rich.

The purpose of this manuscript was to determine the rare earth element signatures of 1) soils receiving annual flood derived sediment from the Mississippi River, 2) soils receiving sediment primarily from upland soils without receiving annual Mississippi River sediment and 3) upland soils. The main intent was to assess if the rare earth element signatures indicate or confirm the sediment source areas. A secondary aim was to determine the exchangeable rare earth element concentrations to assess bioavailability.

3. Materials and Methods

3.1. The Study Area

Aide and Aide (2025) discussed in detail the study area and its location in south-eastern Missouri along the annual floodplain of the Mississippi River. They also described the soil profile morphologies and associated chemical properties of two pedons of the fine-textured Commerce series (Fine-silty, mixed, superactive, non-acid, thermic Fluvaquentic Endoaquepts) and two coarse-textured pedons of the Caruthersville series (Coarse-silty, mixed, superactive, calcareous, thermic Typic Udifluvents). The Commerce pedons presented A-Bw-Bg horizon sequences and displayed ochric and cambic horizons, whereas the Caruthersville pedons presented A-C-Cg horizon sequences and displayed ochric epipedons. In addition, nine surface horizons of the Wilbur series (Coarse-silty, mixed, superactive, mesic Fluvaquentic Eutrudepts) were sampled. The Wilbur pedons have A-Bw-Cg silt loam horizon sequences and reside on silty floodplains. The ochric and cambic soil horizons receive silty stream sediment arising from erosion of the surrounding loess uplands. For reference to the REE distribution of the adjacent uplands, two soil profiles of the Menfro series (Fine-silty, mixed, superactive, mesic Typic Hapludalfs) were characterized. The Menfro series consists of very deep, well drained, moderately permeable soils formed in thick loess deposits having Ap-E-Bt-C horizon sequences. This soil series is the dominant upland soil series, with similar upland soil series differing from the Menfro series in soil depth or drainage classification.

The climate is humid continental. The Cape Girardeau County weather database provides that the annual rainfall is 1.07 m and the daily annual air temperature of 14°C (Festervand, 1981). Annual flooding durations are generally 1 to 7 days; however, long flood intervals may occur during spring snowmelt in the upper Mississippi River watershed (Aide, 2021).

3.2. Laboratory and Statistical Protocols

An aqua-regia digestion was performed to estimate elemental concentrations associated with soluble, exchangeable, organically-complexed, adsorbed/occluded in Fe-oxyhydroxide and the partial lattice degradation of phyllosilicate fractions (Aide & Fasnacht, 2010). In this procedure, 0.25 g of finely-ground fine earth fraction was digested in 0.01 liter of aqua regia for one hour, followed by 0.45 µm filtering with an aliquot analyzed using inductively coupled plasma-atomic emission spectrometry. Quality assurance and analytical accuracy involved four certified reference materials for mining and exploration and duplicate samples.

For the Na-acetate leach, a 0.75 g soil sample of passing a 60-mesh sieve was leached with a sodium acetate matrix at 30°C for one hour. Two reference samples and selected duplicate samples were also Na-acetate leached. The solutions are analyzed using inductively coupled plasma-mass spectrometry. Soil analysis for the aqua regia digestion and Na-acetate extraction were performed by Activation Laboratories (Ancaster, Ontario). Statistical analysis for mean separation used a t-test statistic (Excel).

4. Results

The Caruthersville pedons exhibit silt loam to sandy loam textured soil horizons, featuring slightly alkaline pH levels and limited soil organic matter contents (0.4 to 1.6%). The cation exchange capacity varies from 10.0 to 23.3 cmol kg⁻¹ (centimole of protonic charge per kilogram). The Commerce pedons exhibit silty clay to clayey-textured horizons, having slightly alkaline pH levels and moderate soil organic matter contents (2.2 to 3.4%). The capacity of the cation exchange varies from 23.6 to 40.4 cmol kg⁻¹.

The Caruthersville and Commerce soils have aqua regia digestion REE distributions (Table 2) that indicate that: 1) the light REE have distinctly greater concentrations than the heavy REE, 2) the standard deviations are comparatively small for the two soils, and 3) the mean ratio of Nd/Sm is 5.2 for the combined Caruthersville pedons and 5.8 for the combined Commerce pedons, suggesting similarity. The corresponding ratio of La/Yb is 39.2 for the combined Caruthersville pedons and 24.0 for the combined Commerce pedons, indicating that the soil series are not entirely equivalent.

The aqua regia digestion REE distributions of the Caruthersville and Wilbur soils (Figure 1) exhibit appreciable differences, with the Wilbur series having greater REE abundances. The Caruthersville horizons have greater sand content, suggesting greater sand content was a result of preferential accumulation of bed-

load material from the Mississippi River. The particle size distribution of the Caruthersville pedons clearly indicates that accumulation of sand-sized and transport rounded quartz grains acts to dilute the rare earth element content that is affiliated with the clay separate.

Table 2. Mean and standard deviations (STD) in units of mg kg⁻¹ for two soil series.

Element	Caruthersville		Commerce	
	Mean	STD	Mean	STD
Y	6.64	0.55	12.3	1.0
La	19.6	1.7	26.4	1.5
Ce	36.8	3.4	50.8	2.3
Pr	4.4	0.4	6.1	0.3
Nd	16.2	1.2	23.1	1.4
Sm	3.1	0.4	4.9	0.5
Eu	0.5	0.1	0.9	0.1
Gd	2.4	0.2	3.8	0.2
Tb	0.3	0	0.5	0
Dy	1.5	0.1	2.7	0.2
Ho	0.3	0	0.5	0.1
Er	0.7	0.1	1.4	0.1
Tm	0.1	0	0.2	0
Yb	0.5	0	1.1	0.1
Lu	0	0	0.1	0.1

The mean and standard deviations were assessed for two soil profiles per series across soil horizons.

The aqua regia REE distribution of the Commerce and Wilbur soils (**Figure 2**) are very similar, suggesting that both soil series received sediment materials originating from the upland regions. The mean Nd/Sm ratio of the Wilbur samples is 5.5, which is like that of the Commerce series, further suggesting the parent materials have a common origin. The t-test for mean separation for the Caruthersville-Wilbur soil horizon Nd/Sm ratio is not significant ($\alpha = 0.09$), whereas the t-test for the Commerce-Wilbur soil horizon Nd/Sm ratio is highly significant ($\alpha = 1.4 \times 10^{-7}$). The t-test for the Caruthersville-Wilbur soil horizon La/Yb ratio is significant ($\alpha = 0.001$) and the t-test for the Commerce-Wilbur soil horizon La/Yb ratio is highly significant ($\alpha = 1.5 \times 10^{-5}$).

The Wilbur and Commerce parent materials are silty and clayey alluvium, respectively. The alluvial Wilbur soils are associated with small river systems draining upland soils. The Commerce soils are in backswamp positions largely receiving annual Mississippi River sediment and possible infusion of small stream sediments from upland regions. The Caruthersville soils show coarse-textured bed-

load sediments from the annual flooding of the Mississippi River.

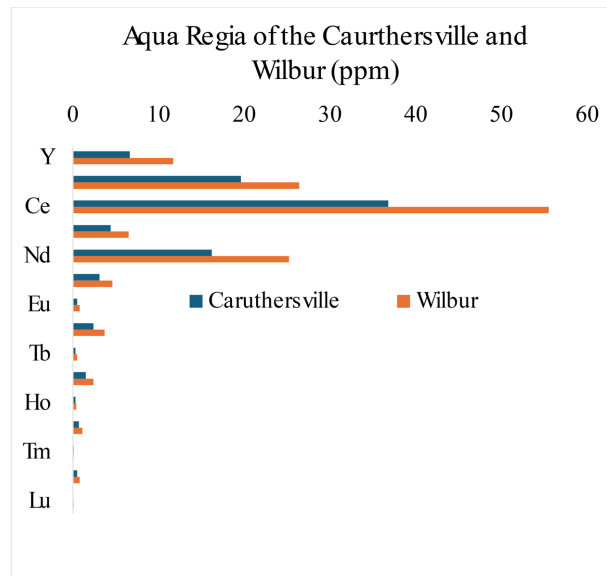


Figure 1. Aqua regia digestion concentrations (ppm) for the Caruthersville and Wilbur pedons.

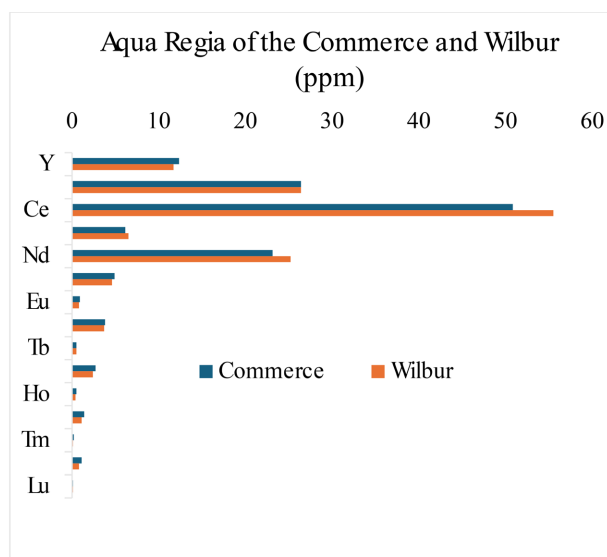


Figure 2. Aqua regia digestion concentrations (ppm) for the Commerce and Wilbur pedons.

For the two upland Menfro pedons, the mean Nd/Sm ratios are very similar at 5.2 and 5.4, respectively. The corresponding La/Yb ratios are 35.7 and 29.6 (**Figure 3**). The La/Yb ratios may also be considered similar, because the Yb concentrations are small and slight concentrations variations may substantially influence the ratio values. The t-test values for the Caruthersville-Menfro soil horizon Nd/Sm ratio is not significant ($\alpha = 0.44$), whereas the t-test for the Commerce-Menfro soil horizon Nd/Sm ratio is highly significant ($\alpha = 4.22 \times 10^{-27}$). The t-test

for the Caruthersville-Menfro soil horizon La/Yb ratio is significant ($\alpha = 0.015$), and the t-test for the Commerce-Menfro soil horizon La/Yb ratio is highly significant ($\alpha = 8.8 \times 10^{-4}$). Thus, the La/Yb ratios involving the Commerce and Menfro soils indicate parent material similarity. Suggesting that the upland Menfro soils provided acceleration erosion sediment to both the Wilbur and Commerce pedons.

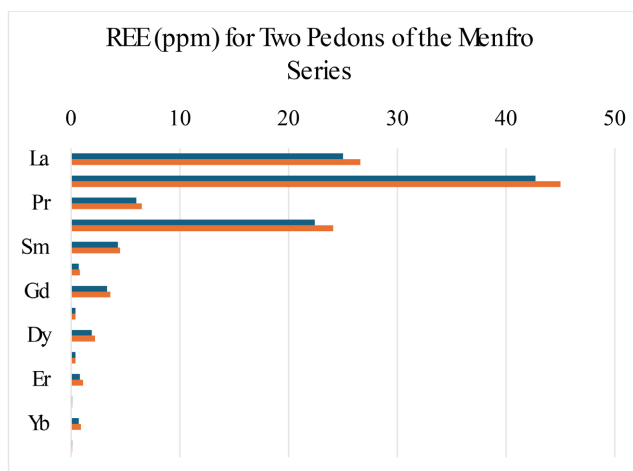
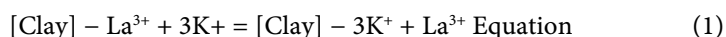


Figure 3. Aqua regia digestion concentrations (ppm) for the two Menfro pedons.

Acetate REE Extractions to Estimate Bioavailability

Phyllosilicate minerals possess net negative charges because of isomorphous substitution. Some clays exhibit greater quantities of net negative charge, such as Smectite > Illite >> Kaolinite. A process termed cation exchange permits cations to exchange positions between the adsorbed phase and the aqueous phase. In Equation (1), Lanthanum, which is initially an adsorbed cation, is displaced by potassium.



Thus, cations providing charge balances for the net negative charges of the phyllosilicates may readily be desorbed and more available for plant uptake or transport as either eluviation-illuviation within the soil profile or accelerated erosion. Given that exchangeable cations are considered largely bioavailable because they may be readily displaced to the soil's aqueous phase, then the quantity of these exchangeable cations essentially determines their plant bioavailability.

Table 3 provides the sodium acetate REE extraction concentrations. The acetate extraction REE concentrations are greater for the Light REE's than the heavy REE's, reflecting the trend observed for the aqua regia digestion concentrations. Allowing for ppb and ppm differences, the acetate extraction concentrations for La and Yb represent only 2.2 and 5.0% of the aqua regia digestion concentrations. Thus, compared to the total REE pool, only relatively small REE concentrations are readily available for water transport or plant uptake. The inference is that sediment transport by the Mississippi River likely does not readily support further REE fractionation.

Table 3. Acetate extractable mean and standard deviations (STD) for two soil series ($\mu\text{g kg}^{-1}$).

Element	Caruthersville		Commerce	
	Mean	STD	Mean	STD
Y	879	164	1017	97
La	579	117	583	48
Ce	1035	320	709	92
Pr	161	33	170	13
Nd	754	147	865	64
Sm	183	36	223	18
Eu	43	8	54	4
Gd	187	36	243	21
Tb	26	5	31	3
Dy	154	29	179	16
Ho	29	5	33	3
Er	79	16	87	8
Tm	9	2	10	1
Yb	53	10	55	5
Lu	8	1.7	9.2	1

The mean and standard deviations were assessed for two soil profiles per series across soil horizons.

5. Summary and Conclusion

Substantial literature exists showing that row crop tillage causes accelerated erosion, providing sediment to the Mississippi River. In southeast Missouri, we developed a sampling protocol involving upland soils, soils developed in silty alluvium mostly derived from deep loess upland soils, and aggrading soils experiencing annual flooding by the Mississippi River. The premise was that the rare earth element signatures for the soils receiving annual flooding would provide connections to the sediment source areas.

Soils of the upland Menfro series generally show substantial erosional features, with sediment-bearing waters transporting material to either the Mississippi River or to floodplains of smaller rivers leading to the Mississippi River. Soils of the Wilbur series were selected to represent the floodplains that do not receive sediment directly from the Mississippi River and soils of the Commerce and Caruthersville series representing floodplain soils receiving annual sediment directly from the Mississippi River.

The rare earth element signatures and selected rare earth element ratios indicate the likelihood that erosional material from the upland Menfro soils supports sedimentation in the Wilbur floodplains. Additionally, the rare earth element signatures and selected rare earth element ratios indicate the likelihood the upland sed-

iment and sediment derived from the Wilbur floodplains are the primary parent materials for the Commerce pedons. The rare earth element signatures and selected rare earth element ratios do not support the likelihood that upland soil material is the dominant parent material for the Caruthersville pedons. Coarse-textured Mississippi River bedload is likely a major component of the Caruthersville parent material.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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