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Using ion-exchange resins to monitor nitrate fluxes in remote semiarid stream beds

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Abstract Monitoring in remote areas can represent a real challenge in environmental studies. Numerous techniques have been developed over the last decades to monitor nutrients and other elements in different systems. However, not all of them are suitable for field applications, particularly when the locations are difficult to access or its accessibility depends on seasonal climate conditions. This study was aimed to test the applicability and efficiency of resin samplers and resin bags to monitor nitrates fluxes (NO_3-N) in two small semi-arid catchments in Northwestern Mexico. Resin samplers were installed in the hyporheic zone below the river bed in order to monitor the vertical fluxes of NO_3-N and remained there for 5 months (during the summer rains). Resin bags were anchored in rock outcrops upstream of the resin samplers before the onset of the summer rainfall season and replaced every 2 weeks during 4 months to capture pulses of NO_3-N in ephemeral streams. NO_3-N pulses in the stream are a potential source of NO_3-N that can infiltrate into the soil. Results of the resin samplers found a difference of up to 12 kg ha^{-1} season^{-1} between the two catchments. The resin bags showed a higher accumulation of NO_3-N in the catchment with lower vegetation cover (160.3 mg L^{-1} season^{-1}) compared to the one with higher vegetation (67.8 mg L^{-1} season^{-1}). Measured nitrate fluxes at both sites responded to rainfall pulses recorded during the monitoring period. Resin samplers and resin bags can be used together, to assess nutrient fluxes on the surface and in the soil and can be tested in any type of ecosystem. In this particular case, these methods demonstrated an efficient way of determining spatio-temporal nitrate fluxes in semi-arid ecosystems in remote areas that are difficult to access, monitor, and collect data.

Keywords Remote field measurements · Resin samplers · Resin bags · Catchment · Nutrient fluxes · Tropical dry forest

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Introduction

The physical characteristics of arid and semi-arid ecosystems play an important role in the accumulation and distribution of nitrogen (N) in the soil. Mineralogical characteristics of soils have a direct influence on ecosystem productivity, the regulation of biogeochemical cycles and water flows (Austin et al., 2004; Barajas-Guzmán et al., 2006; Padien & Lajtha, 1992). Differences in soil texture and porosity greatly influence the spatial variability of water infiltration rates and rainfall redistribution (Austin et al., 2004; Cregger et al., 2014). Nitrate fluxes (NO_3-N) in tropical arid and semi-arid ecosystems are governed by complex catchment characteristics such as drainage area, topography, aspect, slope, geology, soil type, the density of vegetation, climatic conditions, and N deposition history (Belnap et al., 2005; Kopáček et al., 2005). In addition, changes in land use and vegetation cover as a result of anthropogenic activities have drastically modified soil characteristics and NO_3-N concentrations in ecosystems throughout the world (Barajas-Guzmán et al., 2006; Jaramillo et al., 2003; Maass et al., 2005). For this reason, the movement of NO_3-N fluxes (vertical and horizontal) has become an important topic among researchers, especially in remote areas with the influence of seasonal rains and where access and instrumentation of study sites are very difficult.

In highly seasonal ecosystems, such as tropical dry forest (TDFs), one or two wet-dry cycles can take place within a year with each period lasting between four to six months (Austin et al., 2004). These wet-dry cycles affect all aspects of nutrient fluxes, including N mineralization (Austin et al., 2004; Cregger et al., 2014). Water pulses affect the frequency and duration of wet-dry cycles in the soil, but these wet-dry cycles may indirectly control the biological activity of soil organisms, which will determine N turnover (Austin et al., 2004). In the TDFs, the diversity of life forms is likely to be associated with water and nutrient availability (Barajas-Guzmán et al., 2006). Certainly, seasonality in water and nutrient availability plays a major role in the functioning of these ecosystems (Campo et al., 2001).

In situ monitoring of nutrients in soil and surface waters is of basic interest for studies concerning ecology, water management, agriculture, forestry, and environmental protection (Singh et al., 2018; Willich & Buerkert, 2016). However, measuring nutrient fluxes in natural ecosystems and remote areas represents a challenge due to costs, logistics, and difficult access to study sites (Gallant, 2015; Willich & Buerkert, 2016). Several techniques have been used to assess the availability of nutrients under field conditions (Lu et al., 2020; Wey et al., 2022); nevertheless, none are universally applicable due to their specific applications and limitations (Bhogal et al., 1999; Desormeaux et al., 2019; Fares et al., 2009).

Lysimeters are a common technique used to understand the process of water flow and solute transport (Fares et al., 2009; Pütz et al., 2018; Singh et al., 2018; Wey et al., 2022), but have been found to underestimate percolation in-field monitoring (Wey et al., 2022). Suction cups (SCs) are adaptable and can easily be installed in the field; however, SCs have been criticized for only sampling the soil matrix (Wey et al., 2022). Despite their applicability, they are limited for long study periods particularly when accessibility to the sites is limited and highly variable climate conditions are present (Fares et al., 2009). On the other hand, portable autosamplers are novel systems that allow to analyze water samples and soil water concentrations in an automated way at high temporal resolution (Thompson et al., 2021; Yeshno et al., 2021). In the case of remote areas, its installation is restricted due to the flooding of the rivers and its high operating and maintenance costs (Carvalho, 2020; Thompson et al., 2021; Von Freyberg et al., 2021). Autosamplers for nitrate determination are still rare and not available yet for remote areas (Wey et al., 2022). They require intense maintenance (e.g., for the mini-photometer determining NO_3-N) and have a high energy demand. Autosamplers can generate estimates of concentrations over a period of time but you also need continuous measurements (daily or several times a day) of the exact amount of water flowing to be able to calculate/model quantities of mineral N (this disadvantages also occurs with suction cups) (Thompson et al., 2021; Wey et al., 2022). Lysimeters, SCs, and autosamplers often contain mechanisms and electrical circuits to obtain relatively high-resolution measurements but require regular maintenance, which results in higher costs (Fisher, 2012).

Ion-exchange resin (IER) samplers have been used in intensive agricultural systems (Grahmann et al., 2018; Predotova et al., 2011; Siegfried et al., 2011; Wey et al., 2022), mixed forest vegetation (Willich &

Buerkert, 2016), tropical rainforests (Lehmann et al., 2001), and urban land use (Desormeaux et al., 2019) to measure nutrients and trace elements (heavy metals) in soils (Lang & Kaupenjohann, 2004; Schweiker et al., 2014). IER samplers have lower cost and maintenance requirements in comparison to suction cups and lysimeters allowing a high number of replicates, which is a prerequisite to adequately account for temporal and spatial variability (Singh et al., 2018; Wey et al., 2022; Willich & Buerkert, 2016). IER samplers can also be used to determine the availability of nutrients at the plot scale if proper care is taken to maintain soil structure and in absence of stagnant water (Grahmann et al., 2018; Predotova et al., 2011; Siegfried et al., 2012; Wey et al., 2022). Resins exhibit surface characteristics and nutrient sorption phenomena that simulate a plant root surface (Durán et al., 2013). Several authors have confirmed the reliability of IER samplers, especially in cumulative nutrient-leaching studies in agroecosystems (Grahmann et al., 2018; Siegfried et al., 2011; Wey et al., 2022). For example, Siegfried et al. (2011) estimated nitrate (NO_3-N), ammonia (NH_4-N), and phosphate (PO_4-P) in organic vegetable production on irrigated lands with sandy soils in northern Oman. Grahmann et al. (2018) measured nitrate leaching using ion-exchange resin samplers in agricultural fields in northwest Mexico reporting averages of NO_3-N leaching losses from 40 to 70 kg-N ha^{-1}. Wey et al. (2022) conducted a comparative study using IER samplers, suction cups, and soil coring measuring nitrate leaching in four agricultural fields. In this study, the authors described the advantages and disadvantages to use each technique. Another approach that uses resins to capture nutrients are resin bags containing exchange resins that are enclosed in permeable nylon mesh to allow the passage of a solution (Adair et al., 2004; Harms et al., 2009; Lajtha, 1988). However, the application of resin bags has only been reported in litter studies in forests (Kochy & Wilson, 1997), denitrification studies (Harms et al., 2009), nutrient mineralization studies (Goenster et al., 2014; Noe, 2011; Willich & Buerkert, 2016), and nutrient dynamics in soils (Lajtha, 1988; Schade & Hobbie, 2005; Schuur, 2001). Resin samplers and resin bags can be used together, in order to understand nutrient processes in different ecosystem components (water surface and hyporheic zones) assessing fluxes at plot scale. However, few studies (Adair et al., 2004; Goenster et al., 2014; Harms et al., 2009; Noe, 2011; Qian & Schoenau, 2002) report the applicability of IERs in natural catchments, and particularly in semiarid ecosystems dominated and characterized by seasonal fluxes.

Despite the variety of techniques used to measure nutrient fluxes, in highly seasonal remote ecosystems (arid or semi-arid), NO_3-N monitoring has become a challenge due to difficult access and long distances to reach the monitoring sites. This makes the deployment of monitoring equipment expensive and requires constant supervision periods. With this in mind, the goal of this study was to test the functionality of resin samplers and resin bags estimating NO_3-N fluxes in two small semi-arid catchments in northwestern Mexico. Two hypotheses are being tested in this study. The first one is that the resin samplers will adequately accumulate leached NO_3-N as a result of the vertical water movement within the soil structure. The second one is that the resin bags are capable of measuring NO_3-N concentrations in response to surface runoff pulses. The objectives of this study are as follows: (a) to evaluate the combined use of the ion-exchange resin samplers and resin bags to better understand the dynamics of NO3-N at micro-basin scale and (b) to corroborate the application of ion-exchange resins to monitor nutrient dynamics in natural ecosystems. In addition, results from this work will establish a baseline of knowledge of NO_3-N fluxes in remote semiarid catchments found in tropical dry forests (TDFs) and the use of alternative methodologies for short- and long-term monitoring of NO_3-N in soils and surface waters.

Materials and methods

Description of the area study

This study was conducted in northwestern Mexico, within the boundaries of the Alamos-Rio Cuchujaqui Natural Reserve located in the southeastern portion of the state of Sonora (Fig. 1). The main type of vegetation is TDFs (64%) which is an important and dominant ecosystem across Latin America (Stan & Sanchez-Azofeifa, 2019), oaks (19%) and pine (7%) forests, and induced and cultivated pastures (10%) (Álvarez-Yépiz et al., 2008). Two small catchments, known locally as “El Guayabo” (0.36 km^2) and “El Halcon” (0.87 km^2), were selected based on their land cover conditions (Table 1). Land conditions between

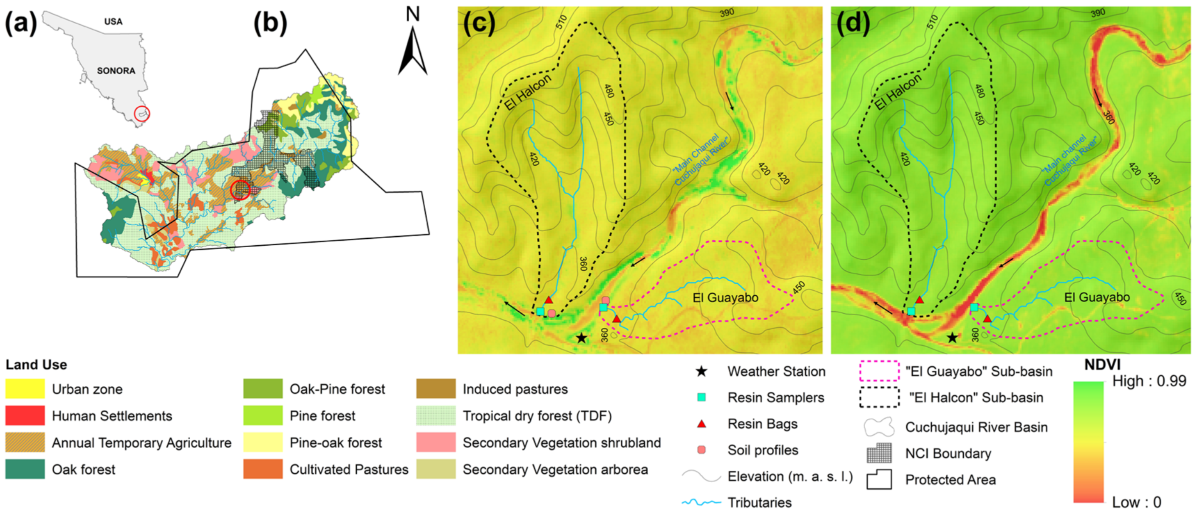


Fig. 1 (**a**) Location of the protected area of flora and fauna Sierra de Alamos-Rio Cuchujaqui; (**b**) land use distribution within the protected area; (**c**) location of resin samplers, resin bags and soil profiles within "El Guayabo" and "EL Halcon" catchments during the dry season; (**d**) location of catchments "El Guayabo" and "EL Halcon" during the wet season. The normalized difference vegetation index (NDVI) index was calculated to show the differences in vegetation before and during the monsoon (Table 1)

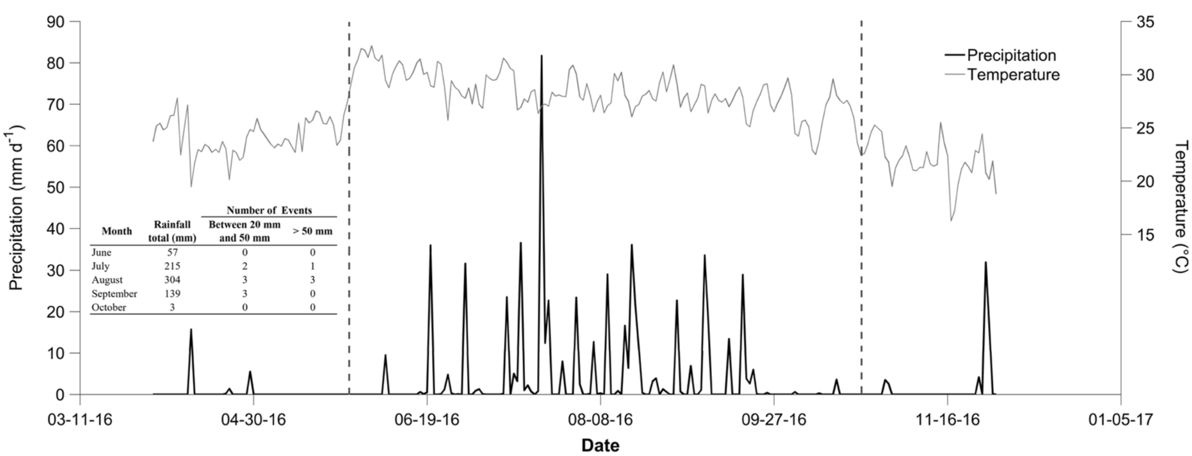
these catchments were evaluated by preliminary inspection of 10-m resolution Sentinel -2 imagery and later confirmed by exploratory field surveys (Fig. 1).

Catchment elevation, slope, stream classification (Strahler method), and aspect were calculated using a 15-m digital elevation model provided by the National Institute of Statistics and Geography (INEGI). Drainage length, land use categories, soil types, and geologic parent materials were determined using available information from INEGI.

A meteorological station was installed in early 2016 adjacent to the two catchments to measure several climate variables (rainfall, wind speed, relative humidity, and air temperature) (Fig. 2). Results from this weather station were compared to the nearest meteorological station from the Mexican Weather Service that has long-term records (1927–2017) of rainfall and air temperature (Servicio Meteorologico Mexicano) located 40 km away. The long-term records were used to determine the influence of the North American Monsoon (NAM) and to classify our study period as a wet year with an influence of 76% of the NAM during the months of July, August, and September (shown in Table 1). During our study period, a total of 738 mm were recorded in 2016; throughout the year, there were 56 events with less than 20 mm, 20 events were between 20 and 50 mm, and only 4 events were greater than 50 mm (Fig. 2). According to Maass et al. (2018) who conducted a long-term study of rainfall-runoff in a similar tropical dry forest ecosystem in Chamela, Mexico, on a monthly basis, rainfall above 200 mm generates 10 mm of runoff. In addition, these authors also found that only about 20% of the events are greater than 20 mm and can potentially generate runoff depending on soil moisture conditions. Large events that result in immediate runoff are greater than 50 mm and account for only 5.5% historically. Results from this study also determined that the probability of runoff occurring in the dry months is less than 10% with 90% of the runoff occurring during the core months of the monsoon (July to September), with the month of September accounting for the highest contribution of runoff historically (48%) (Maass et al., 2018). This coincides with our study site, our station recorded 4 events with precipitation greater than 50 mm, and these occurred in late July and August. Those 2 months were also the only ones that recorded accumulated rainfall totals above 200 mm. In the month of September, the total rainfall was 139 mm with only 3 events that had precipitation between 20 and 50 mm, most likely generating runoff because of the antecedent soil moisture

Table 1 Watershed characteristics

Characteristics (unit)	"El Guayabo" catchment	"El Halcon" catchment
Area (km^2)	0.36	0.87
Channel drainage length (m)	1,318	2,317
Stream classification (Strahler)	Order: 1, 2	Order: 1, 2
Edaphology (classification)	Lithosol, fluvisol, regosol	Lithosol, fluvisol, regosol
Geology (classification)	Rhyolitic tuff-andesite	Rhyolitic tuff-andesite
Land use (classification)	Broadleaf forest Tropical dry forest Roads	Broadleaf forest Tropical dry forest
Vegetation cover (%)	Dense: 57.00 Medium dense: 36.00 Sparse: 7.00	Dense: 84.57 Medium dense: 15.02 Sparse: 0.41
NDVI (mean values)	Dry season: 0.18 Wet season: 0.77	Dry season: 0.20 Wet season: 0.80
Elevation (m)	High: 406 Median: 380 Low: 350	High: 506 Median: 373 Low: 340
Slope (degree)	5.77 degrees	14.34 degrees
Climatic information of the study area		
Precipitation:	Annual historic SMN (1927–2017)	Summer historic (July, August, September)
(Minas Nuevas–SMN)	697 mm	532 mm (76% NAM)
Precipitation:	Annual (our weather station)	Summer (our weather station) (July, August, September)
2017	574 mm	485 mm
2016	730 mm	658 mm
2015	642 mm	510 mm



Month	Rainfall total (mm)	Between 20 mm and 50 mm	> 50 mm
June	57	0	0
July	215	2	1
August	304	3	3
September	139	3	0
October	3	0	0

Fig. 2 Precipitation and temperature were recorded using a high-resolution climate station (Campbell Scientific instruments) installed in the field at the beginning of 2016. Dotted lines show the monitoring period of the resin samplers during the rainy season. The total rainfall during the study period was 730 mm year^{-1}and the annual averaged air temperature was 25.6 °C

conditions (shown in Fig. 2). Although we do not have streamflow measurements to demonstrate days with flow and non-flow, we estimate based on the intensity of the rainfall events that 7 or 8 runoff events occurred during our study period.

The normalized difference vegetation index (NDVI) was determined for both catchments using the red and near-infrared bands from two Sentinel-2 images acquired from the remote pixel repository (https://remotepixel.ca). One image represents dry surface conditions and was captured by the satellite in May 2017, while the other image represents wet surface conditions and was captured in August of the same year (Fig. 1). Once NDVI was calculated, an unsupervised classification was conducted to classify catchment vegetation in three categories (dense, medium, and sparse) using the ISO Cluster approach within ArcGIS 10.8 (ESRI Inc., Redlands, California). The percentage of each vegetation class in both catchments was calculated by dividing the area of each vegetation class by the total catchment area (Table 1).

Resin samplers and resin bags

To estimate the accumulation and fluxes NO_3-N fluxes in the two catchments mentioned above, resin samplers were used capture the NO_3 concentrations that infiltrate with the water. Resin bags on the other hand were used to capture nutrient fluxes on the surface. Using these two methods together allowed us to better understand the dynamics of NO_3-N at the micro-basin scale on the surface and in the soil to demonstrate that it can be used in any type of ecosystem.

Soil sampling and analysis

In both experimental catchments ("El Guayabo" and "El Halcon"), one soil profile per site was excavated following the soil description protocol described in Siebe et al. (2006) and classified under the standard guidelines of the World Reference Base for Soil Resources (IUSS, 2007). Soil samples were taken using a soil auger (5 cm in diameter) in each identified horizon for physical and chemical analyses. Bulk density (g cm^{-3} dry soil) was carried out by cylinder method (Blake, 1965), and pH was measured with a portable pH-meter (Hanna, HI8424, Washington, USA) on a 1:2.5 (soil:solution) soil–water extract. Soil organic matter (OM) was determined by wet oxidation (Walkley & Black, 1934), and inorganic N was extracted with 2 M KCl and determined using a segmented flow chemical analyzer (SEAL Analytical Inc. Mequon, WI, USA). The tone and intensity of the colors of the horizons were determined using the Munsell color system (Nickerson, 1940; Schmidt & Ahn, 2019).

Resin and silica sand preparation

Lewatit® MonoPlus M 600 (Lanxess AG, Leverkusen, Germany), a strong anion resin, was used in the Cl^- form to fill the samplers and the bags. The resins were washed with a solution of NaCl (1 M) for 1 h and then rinsed five times in deionized water prior to installation. Silica sand, a neutral dilution medium, was washed with a solution of HCl (0.1 M) for 1 h followed by thorough rinsing with distilled water three times. Resins and sand stayed moist before installation to maintain high ion-exchange capacity.

Resin sampler construction and installation

PVC cartridges with a diameter of 0.1 m and a height of 0.1 m were used to construct the resin samplers which contained a mixture (2:1) of silica sand and anion exchange resins (Fig. 3). The mixture was prepared under laboratory conditions, packed densely into the cartridges, and stored until installation in ice coolers. IER samplers were installed in "El Guayabo" and "El Halcon" catchments on May 26, 2016, and removed on October 12, 2016, which encompassed the entire length of the monsoon season.

The IER samplers were installed according to the guidelines of TerrAquat Consultancy (Stuttgart, Germany; www.terraquat.com). Trench pits of 2.1 m^3 for "El Guayabo" and 1.6 m^3 for "El Halcon" were dug to install the resin samplers in the field. At "El Guayabo" site, five horizontal tunnels were dug at 0.70 m depth, five at 0.35 m depth, and nine tunnels on the stream bed near the surface. At "El Halcon" site, fifteen tunnels were dug at 0.43 m depth. A total of 34 resin samplers were installed at both sites. In the "Halcon" site, bedrock outcrops were found close to the surface (0.5 m depth), which limited the possibility to install the resin samplers in both catchments

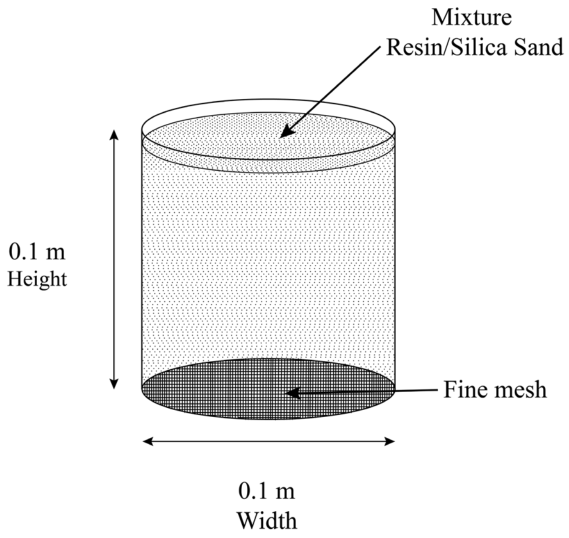


Fig. 3 Resin sampler dimensions

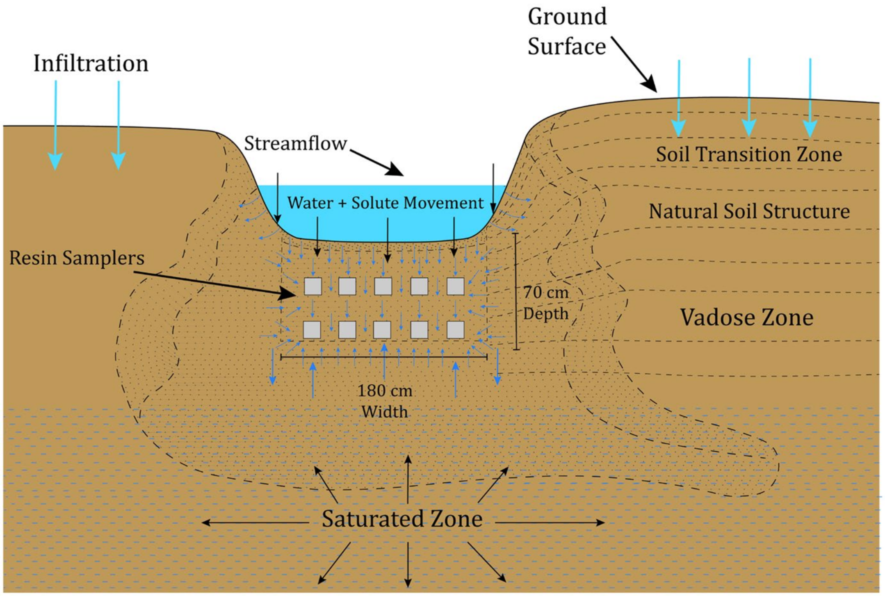
at the same depths. The samplers were placed below an undisturbed part of the plot by digging horizontal access tunnels between 0.10 and 0.70 m depth in the soil profile under the streambed of the intermittent streams. After installation, the horizontal tunnels were tightly refilled with silica sand (used as neutral media to ensure horizontal water flow), and the pits were closed with soil found in the respective horizon (Fig. 4).

After the removal of the resin samplers, the content of each resin sampler was divided into four layers (0–4, 4–6, 6–8, and 8–10 cm) to assess the nutrient-concentration profile for each sampler (Grahmann et al., 2018). The first layer (0–4 cm) received NO_3-N ions infiltrated from above and they are fully absorbed until two-thirds of their adsorption capacity is reached (according to specifications by the manufacturer). Once this capacity is reached, ions move to the following layer, and this process applies in the same manner to all layers. The lowest layer (8–10 cm) is designed to capture capillary rise from the bottom of the sampler, which needs to be separated in case there is contamination of stagnating water or flow of water from below (rising water table) (Fig. 5). Each layer was weighed, labeled, and stored at 5 °C until extractions were conducted.

Construction and installation of resin bags in intermittent streams

The concentration of NO_3-N in the stream was estimated with resin bags. Resins bags had an area of 0.10 by 0.10 m and were built and filled with 5 g of the anion resins MonoPlus M 600. The bags were made using a resistant nylon mesh that allowed water to flow through. The bags were anchored in rock outcrops upstream of the location where the resin samplers were installed using steel cables and connectors

Fig. 4 Installation diagram of the resin samplers in the river bed



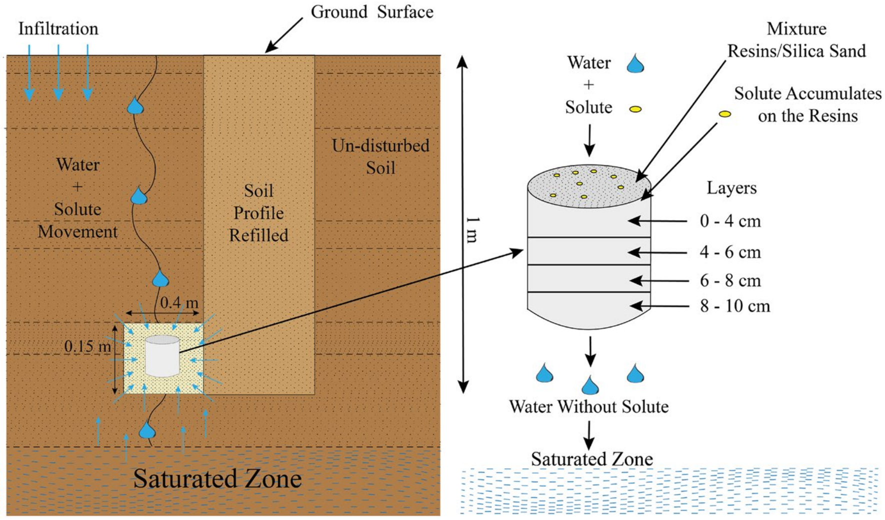


Fig. 5 Operation diagram of resin samplers and NO_3-N adsorption gradient (0–4, 4–6, 6–8, and 8–10 cm)

(Fig. 6). Resin bags have to stay moist in order to work adequately; therefore, regular visits were made to keep them wet with deionized water during the first week until the first rains occurred (July 5, 2016). After their installation (June 28, 2016) and regardless of rain events, resin bags were changed every 2 weeks throughout the summer until October 12 in 2016.

For each of the seven sampling dates, ten resin bags were installed per site resulting in a total of 140 samples at the end of the rainy season. After removal, resins in each bag were weighed, labeled, and stored in a cooling chamber at 5 °C until extraction. Each sample was then analyzed three times in the segmented flow analyzer, and the average of the triplicates was used as the corresponding value for that particular sample.

Precipitation data throughout the rainy season were obtained from the high-resolution meteorological station installed near the experimental sites. Streamflow in the two tributaries was not measured due to the impossibility to access the sites as a result of the high runoff on the main channel of the Cuchujaqui River. However, the runoff generated during the monsoon season occurs with precipitations during the evenings with streamflow occurring very late at night or early in the mornings. Figure 2 shows data of precipitation that can be associated with generation of streamflow.

Ion extraction and analysis

Previous studies (Desormeaux et al., 2019; Grahmann et al., 2018; Siegfried et al., 2012) found that two extractions and the use of sodium chloride (1 M NaCl), sulfuric acid (0.5 M H_2SO_4), or potassium chloride (2 M KCl) as an extraction agent were optimal to extract 90% or more of the ions accumulated in the resins. Therefore, 15 g of the resin-sand mixture of each layer and 60 mL of 0.5 M H_2SO_4 were mixed in a plastic vessel and

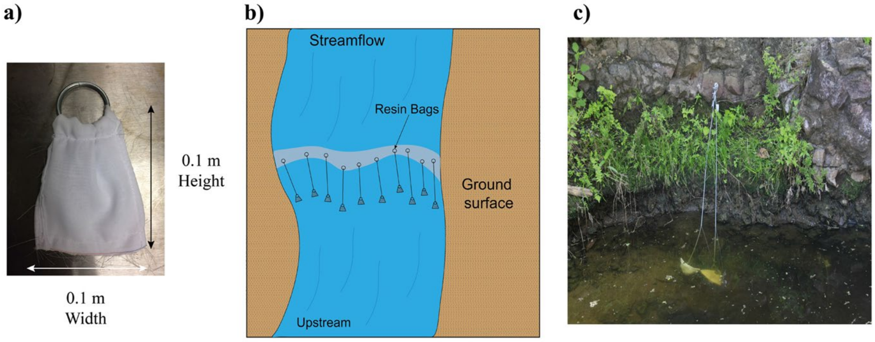


Fig. 6 (**a**) Resin bag dimension; (**b**) installation scheme of resin bags in tributaries; (**c**) Resin bags anchored to the rock

stirred at 180 rpm for two hours. The solution was filtered through a #40 Whatman filter paper and the filtrate was recovered in a plastic tube (aliquot 1). The material retained in the filter paper was extracted again with 60 mL of 0.5 M H_2SO_4 and stirred for 2 h at 180 rpm. The second extraction was filtered again on the same paper and stored in another plastic tube (aliquot 2). In the end, the two aliquots were mixed, and a subsample of 50 mL was taken for further analysis. The same procedure was carried out with the resin bags with 5 g of resin sample and 20 mL of the extractant. Blank samples of washed silica sand and pure anion-exchange resins were extracted similarly. All extractions were diluted using 1 mL of subsample and 14 mL of deionized water (1:14) and stored at 5 °C. For NO_3-N analysis, a Continuous Segmented Flow Analyzer-SEAL Autoanalyzer 3 HR (SEAL Analytical Inc. Mequon, WI, USA) was used. The concentrations of NO_3-N were measured according to method No. G-200–97 Rev.9 provided by SEAL Analytical and three replicates were obtained for each sample.

Calculation of NO_3-N leaching derived from resin samplers

Each resin sampler was examined for the existence of a "zero-layer" with minimum or negligible NO_3-N accumulation in the third layer. The NO_3-N concentrations in the 4–6 and 6–8 cm layers were not considered "leached NO_3-N" to exclude possible contamination from the bottom layer (Bischoff, 2007; Grahmann et al., 2018; Schweiker et al., 2014). The amount of leached NO_3-N in the resin samplers was calculated from the concentrations measured in the extracts from the first resin layer (0–4 cm). A subsample of 15 g for each resin layer was dried over 48 h at 60 °C to determine the gravimetric moisture content (dry weight-based, W_d). The amount of leached NO_3-N per resin layer was determined with the following equations:

$$NO_3 - N(mg\ g^{-1}) = \frac{NO_3 - N\ conc.\ (mg\ L^{-1})\ x\ 120(mL)}{\frac{1000}{W_d(g)}} \quad (1)$$

where NO_3-N per gram of sample NO_3-N (mg g^{-1}) is equal to the NO_3-N concentration (mg L^{-1}), 120 (mL) is the volume of H_2SO_4 added during the extraction process, and 1000 is the conversion factor to ppm. The following step consisted in estimating the dry layer weight (DLW) which was calculated using the total fresh layer weight (FLW) following the equation:

$$DLW(g) = FLW(g)\ x\ \frac{100}{\%\ subsample\ moist + 100} \quad (2)$$

Once the NO_3-N per gram of sample was obtained (Eq. (1)), the NO_3-N content in each layer can be calculated and presented as NO_3-N leaching in kg ha^{-1} $season^{-1}$ with the following equation:

$$NO_3 - N\ content\ \left(kg\ ha^{-1}\right) = \frac{NO_3 - N\left(mg\ g^{-1}\right)\ x\ DLW(g)\ x\ 100}{78.5\left(cm^2\right)} \quad (3)$$

where NO_3-N content in kg ha^{-1} is equal to NO_3-N per gram of sample NO_3-N (mg g^{-1}) multiplied by the dry layer weight (DLW) and divided by 78.5 (the resin sampler surface area). To convert the units from mg cm^{-2} to kg ha^{-1}, a conversion factor of 100 was used.

For the resin bags, the amount of NO_3-N accumulated over 14 days was calculated from the concentrations measured in the extracts in each bag in mg L^{-1} 14 day^{-1}. All data were evaluated for normality using the Shapiro–Wilk test. Outlier values were not deleted from the dataset to show the natural variation of the soil system. One-way ANOVA with Tukey multiple comparison tests were used to check for significant differences ($P < 0.05$) between resin sampler layers. Statistical analyses were performed using Minitab 18 (Minitab LLC, State College, PA, USA).

Results and discussion

Soil profile classification

The soil profile descriptions and chemical and physical properties of the two profiles ("El Guayabo" and "El Halcon") are presented in Table 2. Both sites share the category of fluvisol due to their formation over intermittent streams. Fluvisols are characterized by soils that are developed on alluvial deposits (Food & Agriculture Organization of the United Nations, 2014). Each site has particular conditions that differentiate them from each other. The soil in "El Guayabo" site is characterized by a slightly acid sandy clay

Table 2 Soil profile description and soil analysis results

Site	Soil type	Depth	Texture[1]	Color (wet)	In situ humidity	Volumetric Humidity	Porosity	pH	$CaCO_3$	OM[2]	BD[3]	NO_3-N	Horizon[4]
		cm				%			%	%	g cm^{-3}	ppm	
El Guayabo	Thaptomollic fluvisol	0–20	RA	5YR-3/2	Very dry	5.8	0.63	6.95	0	3.6	0.99	19	Ah
		20–42	RA	5YR-3/3	Very dry	10.9	0.57	6.78	0	2.8	1.13	15	C
		42–50	CRA	5YR-4/4	Fresh	14.4	0.70	6.86	0.5	1.4	0.81	19	C1
		50–65	RA	5YR-3/2	Fresh	17.9	0.60	6.76	0.5	1.6	1.07	15	C2
		65–75	CA	5YR-4/6	Fresh	16.0	0.60	5.71	0.5	0.6	1.06	14	2Ah
		75–83	RA-R	5YR-2.5/2	Fresh	32.7	0.52	6.62	0	1.2	1.26	13	3Ah
		83–90	CA	5YR-3/3	Fresh	28.2	0.51	6.05	0.5	1.4	1.31	13	Ag
		90–96	CR	5YR-2.5/1	Wet	38.2	0.50	6.72	0.5	0.6	1.33	13	Ag
		96–115	CRA	5YR-3/4	Wet	22.1	0.52	6.74	0.5	0.4	1.28	13	2C2
		115–135	CA	5YR-4/4	Wet	22.5	0.53	6.42	0.5	0.7	1.25	11	3C2
		135–150	RA	5YR-4/1	Wet	-	-	-	0	-	-	-	4C2
El Halcon	Calcareous fluvisol	0–7	RA	5YR-3/2	Fresh	6.9	0.61	6.86	0.05	2.6	1.04	11	Ah
		7–26	RA	5YR-2.5/2	Wet	9.6	0.57	7.02	0.05	2.2	1.13	9	C
		26–35	RA	5YR-3/2	Dry	12.2	0.58	7.68	2–10	1.2	1.11	13	C1
		35–45	CR	5YR-5/3	Dry	11.8	0.58	7.74	25–50	1.4	1.12	12	C2
		45–63	CA	5YR-6/3	Fresh	12.2	0.61	7.75	25–50	1.2	1.03	9	C3
		63–80	RA	5YR-6/2	Fresh	14.1	0.60	7.77	> 50	1	1.05	27	C4
		80–100	Parental material	-	-	-	-	-	25–50	-	-	-	R

[1] *RA* sandy clay, *CRA* sandy clay loam, *CA* sandy loam, *RA-R* sandy clay-clay, *CR* clay loam (Siebe et al., 2006)

[2] *OM* organic matter content

[3] *BD* bulk density

[4] Nomenclature and international designation of horizons (IUSS Working Group, 2007)

loam soil developed on a colluvial material (thapto-mollic fluvisol) (IUSS, 2007) and soil pH ranges from 5.71 to 6.95. The percentage of carbonate calcium ($CaCO_3$) and OM in the soil profile ranged between 0 to 0.5% and 0.4 to 3.6%, respectively. Soil organic matter is mainly concentrated between 0 and 42 cm. Porosity ranged from 0.50 to 0.70. On the other hand, the "El Halcon" site is characterized by being formed on a colluvial material with a carbonated sandy clay formation (calcareous fluvisol) and the soil pH ranges from 6.86 to 7.77. The percentage of $CaCO_3$ and OM in the soil profile ranged between 0.05 up to 50% and 1 to 2.6%, respectively, and OM is mainly concentrated in 0–26 cm soil depth. The porosity ranged between 0.50 and 0.70 and high concentrations of $CaCO_3$ found in the soil profile were determinants for the designation of the soil type.

The soil profile in "El Guayabo" presents a greater number of horizons suggesting a high morphological dynamic, which allows the recurrent accumulation of sediments to the site. The chemistry characteristics such as pH, carbonates, and texture are related to the presence of more water in this site. The low accumulation of carbonates is associated with a longer residence time of the water in the soil, which does not allow the processes of precipitation of CO_3. Another important factor is the presence of finer mineral particles like clay or loamy clay texture which is evidence of greater development of soils that lead to improved physical conditions such as greater porosity, water retention in the soil, and changes in mobility of ions in solution. In "El Guayabo" site, the morphological dynamics and soil properties show that this location has the greatest potential for accumulation of OM and potentially mineralizable nitrogen. On the contrary, the profile of the "El Halcon" site has less edaphic development, thicker fractions, slightly alkaline and alkaline pH, and a clear concentration of carbonates. The description of soil profiles carried out in both study sites, including a broad analysis of physical and chemical parameters, was crucial for understanding the NO_3-N dynamics in the soils and its effect on the wet-dry cycles of TDFs and their tributaries.

Cumulative capture of NO_3-N leachate in the stream bed

The NO_3-N leaching found in both sites, "El Guayabo" and "El Halcon," were highest in the first and fourth layers of the resin samplers. The first layer ("leaching layer") of the resin samplers represents NO_3-N leached from above for each site (Fig. 7). The concentrations in the fourth layer in both sites confirmed NO_3-N movement from below caused by a temporary increase in the water level, especially in "El Guayabo" (78.4 kg ha^{-1} season^{-1}) due to the influence of the Cuchujaqui River floodplain spilling over the tributary. ANOVA showed significant

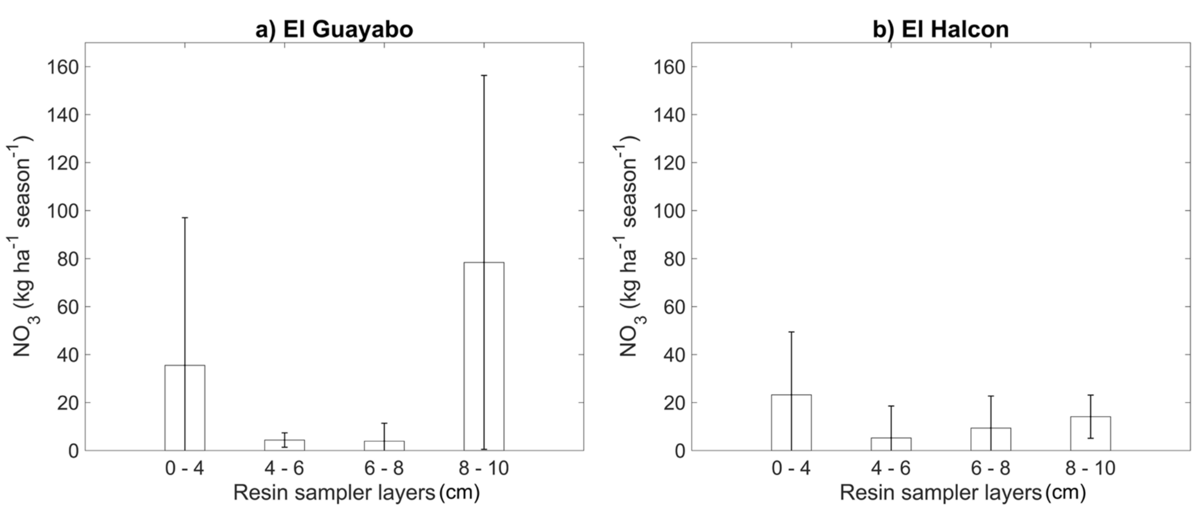


Fig. 7 NO_3-N fluxes (kg ha^{-1} season^{-1}) in four layers (0–4, 4–6, 6–8, 8–10 cm) for two catchments during monsoon season. (**a**) "El Guayabo" (n = 19, 5 months) and (**b**) "El Halcon" (n = 15, 5 months). Error bars show ± one standard deviation of the mean NO_3-N concentration per site

differences between the layers (“El Guayabo”, $P < 0.05$; “El Halcon”, $P = 0.03$) and verify the efficiency of the resin sampler methodology. As shown in Fig. 7, results showed that water was moving predominantly vertically (downwards), thus yielding reliable estimates of NO_3-N leaching (Bischoff, 2007; Grahmann et al., 2018; Predotova et al., 2011).

When analyzing the leaching layer in both sites, total NO_3-N leached an average of 35.5 and 23.2 kg ha^{-1} $season^{-1}$ for “El Guayabo” and “El Halcon,” respectively (Fig. 7). Differences in soil texture, porosity, and the seasonal climate contribute to water infiltration rates, rainfall redistribution, and surface runoff. These differences are regulators of N fluxes in soils (Austin et al., 2004; Cregger et al., 2014). Sandy soils have less runoff due to the great infiltration capacity; infiltration is deeper and loss by evaporation is smaller (Austin et al., 2004). NO_3-N is an anion that can readily leach through the soil profile. Soils with significant quantities of silt, clay, and OM will also retain more NO_3-N than soils without much silt and clay (Gaines & Gaines, 1994). As a result, NO_3-N accumulation in many arid and semi-arid ecosystems should be greater on fine-textured than coarse-textured soils due to relatively higher water availability in sandy soils during the same seasons (Austin et al., 2004; Cregger et al., 2014).

A study conducted between different land uses (crop, pasture, and urban land) (Desormeaux et al., 2019) measured NO_3-N leaching. Results showed NO_3-N leaching of 23, 69, and 152 kg ha^{-1}, respectively, for turf, crop, and pasture land-use types. Although the results obtained do not represent a natural area, their values can be used as a reference with those obtained in the present work (35.5 and 23.2 kg ha^{-1} $season^{-1}$). Our NO_3-N leaching results are below the results found in crops (69 kg ha^{-1}) where a significant amount of fertilizers is added. Another study (Grahmann et al., 2018) measured NO_3-N leaching in wheat and maize crops in an experimental field located in a semi-arid region in Sonora, Mexico. The results of this study show a total NO_3-N leaching loss averaged 53.5 kg ha^{-1} $season^{-1}$ for wheat and 68.2 kg ha^{-1} $season^{-1}$ for maize. Wey et al. (2022) conducted a comparative study using IER samplers, suction cups, and soil coring measuring nitrate leaching in grass-clover leys, maize, cereal, and canola crops. N fluxes ranged from 44 to 219 kg N ha^{-1} using IER samplers, 24 to 540 kg N ha^{-1} using soil cores, and 193 kg NO_3-N ha^{-1} using suction cups between the different crops. The results obtained were reliable and comparable to each other. For monitoring and comparison strategies, the IER samplers technique seems to be the preferred approach due to the high and versatile spatial coverage and low material costs compared with the other techniques. The results obtained in the three studies are comparable to each other, and can also be used as a reference with the present work. On the other hand, a laboratory experiment about the effect of soil texture on NO_3-N leaching in soil was conducted (Gaines & Gaines, 1994). The results of this study show higher NO_3-N leaching (176 mg kg^{-1}) in the sandy clay loam soil texture compared to the loamy sand (149 mg kg^{-1}), green mix (128 mg kg^{-1}), and sand (119 mg kg^{-1}) soil texture. Soil profile results in “El Guayabo” (deeper soil, a higher percentage of sandy clay loam particles, high porosity, and a lower percentage of OM in the deeper soil layers) explain the presence of greater leaching of NO_3-N compared to “El Halcon” site.

Monitoring of NO_3-N concentrations dynamics in intermittent streams

Resin bag results showed NO_3-N being carried in the stream runoff water in bi-weekly intervals over the rainy season (Table 3) representing a potential source of nitrogen that can be leached into the soil. Total NO_3-N fluxes averaged 160.35 and 67.88 mg L^{-1} $season^{-1}$ for “El Guayabo” and “El Halcon,” respectively (Table 3). “El Guayabo” obtained higher pulses during the first dates because, as in many arid ecosystems, NO_3-N accumulates in catchment soils during dry periods, and its concentrations or fluxes can be very high during the first precipitation events (Fig. 8) (Austin et al., 2004; Cregger et al., 2014). As the monsoon progresses, the concentrations of NO_3-N decreased over the time due to a dilution effect produced by large volumes of stream discharge caused by precipitation (Fig. 9) (Austin et al., 2004). On the other hand, as previously reported, NO_3-N accumulated during the dry season is easily leached due to high precipitation and runoff events, which could increase N loss and decrease N retention in soils (Cregger et al., 2014). High NO_3-N concentrations found in resin bags in “El Guayabo” site can be an indicator of total NO_3-N leached on the same site. At the end of the monitoring period, high concentrations

Table 3 Total NO_3-N measured by resin bags during the summer. The NO_3-N concentrations represent the number of sampling days (14 days, except from August 9 to 31, which represents the accumulation of 22 days due to the difficult access to the sites caused by bad weather conditions) and the area of the catchments ("El Guayabo": 0.32 km^2; "El Halcon": 0.87 km^2)

Date	"El Guayabo" NO_3-N (mg L^{-1} day^{-1})	"El Guayabo" NO_3-N (mg L^{-1} 14 day^{-1})	"El Halcon" NO_3-N (mg L^{-1} day^{-1})	"El Halcon" NO_3-N (mg L^{-1} 14-day^{-1})
07/12/2016	18.65	261.17	8.59	120.33
07/26/2016	16.04	224.63	1.74	24.49
08/09/2016	11.24	157.49	8.79	123.08
08/31/2016	7.09	156.08	4.80	105.68
09/14/2016	5.95	83.41	3.21	44.98
09/28/2016	4.32	60.57	2.30	32.23
10/12/2016	12.79	179.11	1.74	24.36
Average	10.87	160.35	4.45	67.88

of NO_3-N were found in "El Guayabo" site, which could be due to ponding zones. Ponding zones can raise the concentration of NO_3-N (Newman et al., 1997).

The use of resin bags in monitoring nutrients in surface waters has not been reported to our knowledge. Some studies have used resin bags for nitrate mineralization studies in soils. For example, Harms et al. (2009) and Schade and Hobbie (2005) buried resin bags between 5 and 10 cm depth into the soil to determine NH_4-N and NO_3-N in Arizona. Another study of NH_4-N and NO_3-N mineralization in wetland soils was conducted by Noe (2011). The resin bags were constructed and placed inside a PVC tube which was buried in the ground 7.5 cm depth for 1 month.

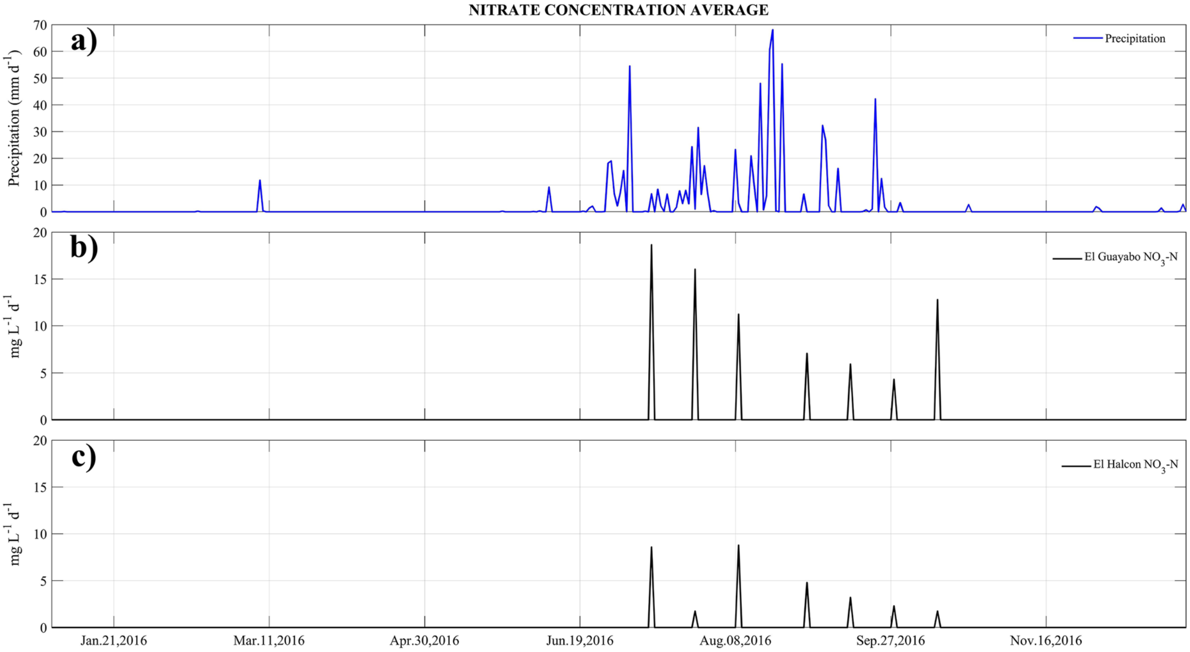


Fig. 8 **a)** Time series of daily precipitation and **b)** NO_3-N fluxes in mg L^{-1} 14 day^{-1} in "El Guayabo" (n = 70; 4 months) and (**c**) "El Halcon" (n = 70; 4 months) during monsoon season

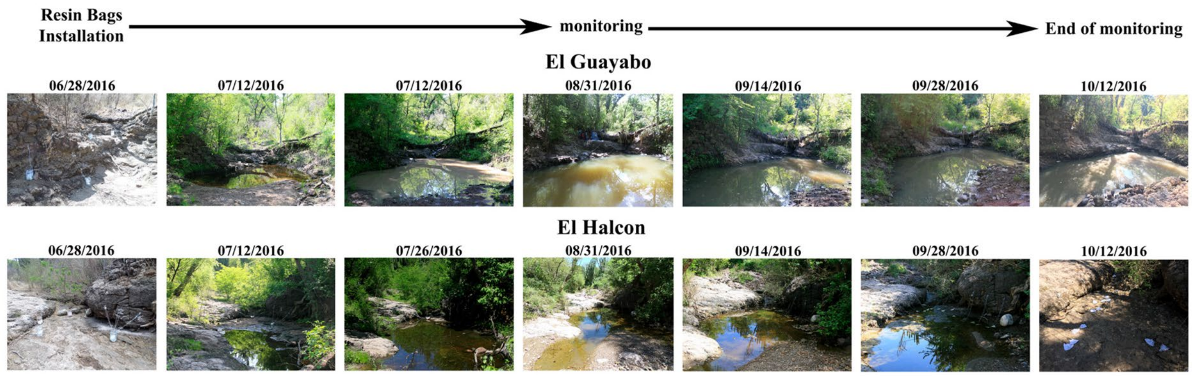


Fig. 9 Timeline of resin bags. The accumulated water in the tributaries is proportional to the recorded rainfall events

Other studies used water samples to measure the accumulation of NO_3-N on single-day events. For example, Arce et al. (2013) estimated nitrate accumulation in a Mediterranean headwater stream in Spain and found that NO_3-N concentrations ranged from 0.7 to 1.6 mg L^{-1}. Another study found measured NO_3-N concentrations in 11 streams during single-day samplings in different ecosystems around the world (Webster et al., 2003). Two sites were selected within arid ecosystems (Sycamore Creek, Arizona, and Gallinas Creek, New Mexico). In both sites, NO_3-N concentrations were 0.0168 mg L^{-1} and 0.0075 mg L^{-1} respectively. Another study also conducted in Sycamore Creek, Arizona, measured NO_3-N concentrations using water samples during a single flash flooding event (Fisher & Minckley, 1978). NO_3-N concentrations ranged from 0.9 to 1.9 mg L^{-1} during 5 h of flooding. Our normalized results per day found NO_3-N concentrations ranging between 1.74 and 18.65 mg L^{-1} day^{-1}. These results are comparable with the results found by Arce et al. (2013), Webster et al. (2003), and Fisher and Minckley (1978) (Table 4). However, there are no control/comparison results with other standard methods used in similar environments. Nevertheless, it shows the applicability of ion-exchange resins in remote/intermittent areas and it also provides an estimate of leaching fluxes and their movement that are in the same order of magnitude of observations previously reported in the literature.

The resin samplers facilitated to monitor the vertical flow or the infiltration of NO3-N in the river bed. Although the resin bags showed satisfying results in the capture of NO3-N pulses, this method cannot account for losses of mineralized N by denitrification, especially if the resin bags experienced wet and dry periods and high variability in air temperature during their installation period of 2 weeks due to increasing and decreasing river water table. These changes could lead to denitrifying conditions for NO3-N, and hence, NO3-N concentrations were underestimated. This is a common weakness of the method itself, for the same reason, the bags were kept moist before the first rains, and managed to accumulate the nitrates that were passing through the flow of the streams. With this, it was possible to monitor the NO3-N concentrations that passed in periods of approximately 14 days. This allowed us to observe the dynamics of the NO3-N superficially in time in contrast to point data that represent a snapshot of what happened at the time of sampling in the field. After removal of resin samplers and resin bags, the resins were transported inside a cooling box to maintain low temperatures until laboratory extraction to minimize microbial activity. In the past, researchers reported the addition of mercury to resins in order to restrict microbial activity; this is not possible anymore due to the polluting and toxic effects of this element.

Conclusions

Our results show that NO3-N fluxes in semi-arid ecosystems can be estimated by resin samplers and resin bags. The resin bags captured NO3-N pulses transported in the streamflow; the same ones that potentially infiltrated in the river bed as vertical flow. Once the NO3-N infiltrated into the ground, the resin samplers had the ability to accumulate NO3-N, triggered by leaching over long periods of time (during the monsoon) without the need for maintenance or observation at the study sites. The NO_3-N concentrations in the resin samplers and resin bags showed that TDF nutrient dynamics were strongly linked to precipitation and the volume of runoff generated during the monsoon. Taking into account the challenge to conduct research in a remote study area, these methods proved to be very effective for the quantification of NO_3-N leaching in soils and NO_3-N loads transported by stream discharge obtaining acceptable and sound data in arid ecosystems. The combination of both methodologies allowed us to connect the dynamics of both components (surface and soil), therefore contributed to a system understanding of nutrient dynamics during the entire length of one season of the North American Monsoon. The resin samplers allowed us to better understand how the flux of nutrients after storm events can lead to the accumulation of nutrients found in the hyporheic zone via the vertical flow or the infiltration of NO_3-N in the river bed.

Although the resin bags showed good results in this work, the resin bag method cannot account for losses of mineralized N by denitrification, especially if the resin bags experienced wet and dry periods and high variability in air temperature during their installation period of 2 weeks due to increasing and decreasing river water table. These changes could cause that NO_3-N was denitrified, and hence, NO_3-N concentrations were underestimated. This is a common weakness of the method itself, for the same reason, the bags were kept moist before the first rains, managed to accumulate the nitrates that were passing through the flow of the streams. With this, it was possible to monitor the NO_3-N concentrations that passed in periods of approximately 14 days. The reactivity of the nutrient pulses can be very different if the two catchments tested in our study had more contrasting land use covers. Potentially, catchment areas with higher vegetation cover would generate more biomass and may result in higher loads of nutrients washed in the streambed during rain events which will then be infiltrated into the soils.

Results from this work demonstrate that the methodological combination of the resin samplers and resin bags can be used to better understand how an ecosystem with intermittent streamflow's NO3-N concentration in the surface water column can then be infiltrated and accumulated in the soil. This adds new knowledge of how NO3-N fluxes in semi-arid ecosystems work, particularly those dominated by seasonal rainfall regimes.

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Availability of data Data access is available through Mendeley Data at https://data.mendeley.com/datasets/8srgt2szvr/1.

Declarations

Conflict of interest The authors declare no competing interests.

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