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Sugarcane cultivation as a major surface source of sediment in catchments from a coastal zone of Pernambuco, Brazil

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ABSTRACT: Identifying sediment sources is fundamental for protecting and improving soil and water quality. Conventional fingerprinting studies have often collected sediments at the overall watershed outlet only, resulting in an important spatial scale dependency. This study aimed to identify and to assess the delivery patterns of sediment sources within three priority sub-catchments (Sapocaji, Piedade and Minas) located in the downstream portion of the Ipojuca River watershed in Brazil. This research would build on understanding sediment sources in the studied watershed by elucidating source type contributions on a sub-catchment basis. Both bed and suspended sediment samples were collected in these sub-catchments, and two types of sources were sampled: surface and subsurface sources. A total of 21 geochemical tracers were measured. The tracers were evaluated for their conservation and discriminatory ability (Kruskal-Wallis test and linear discriminant analysis), and the best set of tracers was selected for source apportionment modeling using MixSIAR. Surface (i.e., sugarcane croplands) sources contributed the highest to suspended sediments in the Piedade and Minas sub-catchments. There was a difference in the quality of riverbank management, which helped explain contrasts in the importance of this source. Overall, sub-catchment-specific sediment control management measures, such as the revegetation of riparian forests, need to be implemented, mainly in the Sapocaji sub-catchment. These results underscore the importance of connectivity for surface source contributions.

Keywords: watershed management, erosion, hydrology, sediment fingerprinting.

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INTRODUCTION

Brazil has emerged as a dominant force in the global food and agriculture sector, solidifying its position as one of the largest producers worldwide (Pellegrina, 2022). However, this achievement was not obtained without unintended consequences, such as elevated soil erosion rates due to intensive agricultural practices (Guerra et al., 2014) and land-use change (Santos et al., 2017). Elevated soil erosion can accelerate sediment delivery to river systems, resulting in detrimental impacts on water resources (Walling et al., 2003; Wu et al., 2020). To address such problems, improved knowledge of sediment sources is crucial for targeting erosion and sediment delivery control as part of integrated watershed management (Minella et al., 2008; Collins et al., 2017).

Adoption of mathematical modeling previously led to quantitative approaches that estimate sediment source relative contributions to target sediment samples, resulting in a more widespread uptake of the fingerprinting approach (Collins et al., 2020; Evrard et al., 2022). Here, the fingerprinting approach is founded on collecting representative soil samples at different points in the watershed and of sediment in transport, and then analyzing both in the laboratory to determine a set of fingerprint properties, which are then used to compare and identify the similarities between the sampled sources and target sediment (Davis and Fox, 2009; Owens et al., 2016). This approach assumes the analyzed sediment preserves the characteristics of its sources even after detachment, mobilization, and transport, i.e., the chosen tracers need to be conservative (Peart and Walling, 1986), and the characteristics of the sources are significantly different, thereby allowing for the quantification of source contributions (Sherriff et al., 2015). Accordingly, fingerprint properties that have proved useable in this regard include, amongst others, heavy metals, stable carbon and nitrogen isotopes, and radionuclides (Liu et al., 2017; Collins et al., 2020).

Ipojuca River was previously considered one of the most polluted fluvial systems in Brazil (Silva et al., 2015; Nascimento et al., 2019). This issue can be explained by a cocktail of inputs reaching the river system, such as pesticides, insecticides and petroleum derivatives, among others (Silva et al., 2019). A fingerprinting study previously conducted on the Ipojuca River underscored the importance of regional sediment source contexts, showing that specific geochemical patterns were generated for each region of this large watershed (Nascimento et al., 2023). According to that recent study, 80 % of suspended and 87 % of bed sediment sampled at the outlet of the Ipojuca River originate from the downstream region. Such findings highlight the importance of studying the main downstream sub-catchments in the Ipojuca watershed in more detail.

This study aimed to identify and assess the delivery patterns of sediment sources within priority sub-catchments located in the downstream portion of the Ipojuca River watershed. Our study was driven by the objective of improving the current understanding of sediment sources within sub-catchments releasing sediment to the watershed outlet and the near-coastal zone.

MATERIALS AND METHODS

Study area

Ipojuca watershed is located in the Pernambuco State, Northeastern Brazil. It originates in a semiarid region and flows through a humid forest zone before reaching the coast, covering an approximate area of 3,430 km² with a length of 320 km (Condepe/ Fidem, 2005). Three sub-catchments located in the downstream portion of the Ipojuca River watershed were selected for this study: Sapocaji (14 km²), Piedade (15 km²), and Minas (9 km²) (Figure 1).

Figure 1. Maps showing the location of the Ipojuca watershed in Brazil and of the sub-catchments and their respective soil classes, plus source material and target sediment sampling points.

The downstream region of the Ipojuca River watershed, or coastal zone, where the Sapocaji, Piedade, and Minas sub-catchments are located, has an average annual precipitation between 2000 and 2400 mm (Condepe/Fidem, 2005). The sub-catchments are in an environment of plains and lowered coastal plateaus formed from fluvialmarine sedimentation. The geomorphology downstream of the Ipojuca River is marked by remobilized surfaces, floodplains, alluvial terraces, and local typical slopes known as "Mares de Morros". The three sub-catchments contain mostly Ferrasols (*Latossolos*), Acrisols (*Argissolos*) and Gleisols (*Gleissolos*). In the Sapocaji and Piedade sub-catchments, Acrisols (*Argissolos*) are dominant, covering 55 and 63 %, respectively, while Ferrasols (*Latossolos*) (79 %) are the main soil class in the Minas sub-catchment (Figure 1).

Source and target sediment sample collection

Target sediment sampling encompassed both suspended and bed sediments. Bed sediments were collected as composite samples by scraping the non-consolidated material available in the main watercourse bed of the sub-catchments, without exceeding a depth of 0.05 m. Suspended sediments were collected using time-integrated samplers (Phillips et al., 2000) at points from each sub-catchment outlet. Suspended sediment samples were composites combining material collected in three or four samplers during the same period. Four composite samples of suspended sediment were collected in the Minas sub-catchment, two in the Piedade sub-catchment, and four in the Sapocaji sub-catchment. Suspended sediment sampling period spanned the hydrological dynamics of 2019 to 2020, representing periods of low and high water discharge (Table 1). Three samples of the composite bed sediment were collected in the Sapocaji sub-basin, five in Piedade, and six in Minas.

The source material samples were collected to represent sugarcane croplands, unpaved roads, and channel banks (Figure 2). More broadly, we considered two sediment sources: surface and subsurface. Surface source samples were collected from surface soils under sugarcane. Channel banks and unpaved roads represented subsurface sources. In this region, roads mainly represent subsurface layers exposed at the foothills in the sub-catchments.

In the Minas, Piedade and Sapocaji sub-catchments, 28, 32, and 28 source material samples were collected, respectively. For each selected point, 10-20 sub-samples were collected from the 0.00-0.05 m layer for sugarcane croplands and unpaved roads. On the other hand, channel banks samples represented deeper soil layers, which can be mobilized by processes such as lateral erosion. The locations of sampling points are shown in figure 1. Overall, for the Sapocaji sub-catchment, there were 12 surface source samples and 16 subsurface source samples. In the Piedade sub-catchment, there were 12 surface source samples and 20 subsurface source samples, and in the Minas sub-catchment, 11 samples were collected from surface and 17 from subsurface sources.

Sample preparation

All source material samples were air-dried, ground, and sieved to <2 mm. Sediment samples were dried in a circulating oven at 50 °C, gently disaggregated and passed through a 2 mm sieve. Particle size distribution of the suspended sediment samples was determined using a liquid dispersion particle analyzer (Microtrac S3500) in the range of 20 to 2.0 mm. Prior to this step, organic matter was burned off by adding 20 mL of 25 % H₂O₂ in an oven at 50 °C for at least 24 h; then the particles were dispersed by adding 10 mL of 6 % NaOH and shaking at 130 rpm for 12 h. As observed in Nascimento et al. (2023), this analysis indicated that 90 % of the particles had a diameter smaller than 24.3 μm. Therefore, all samples were sieved to <32 μm to minimize potential errors arising from particle size contrasts during comparison of source and sediment sample properties for sediment source apportionment.

Geochemical analyses

We analyzed the total content of 21 metals (Al, Ba, Ce, Cr, Fe, Gd, La, Nd, Ni, Pb, Pr, Sc, Sm, Sn, Sr, Th, Ti, V, Y, Zn, and Zr), that were tested as potential sources tracers. Following the methodology of Estévez-Alvarez et al. (2001), 0.5 g of each sample (source material or target sediment) was pre-digested in 10 mL of HF for 12 h. Subsequently, HNO₃ (5 mL) and HClO₄ (3 mL) were added and heated to 180 °C to ensure complete dissolution of samples. Finally, the extract was diluted in 5 mL of HCl and ultrapure water to reach a total volume of 25 mL.

Figure 2. Photographs of source sampling areas: (a) sugarcane croplands, (b) sugarcane directly connected to the river, (c) channel bank, and (d) unpaved roads close to the river.

Metal concentrations were measured using an ICP-OES (Optima DV7000, Perkin Elmer) coupled with a cyclonic chamber system, increasing the readings' accuracy. Total levels of Zr were measured using a portable X-ray fluorescence spectrometer (Bruker S1 Titan 800 Handheld XRF Analyzer). Tools such as calibration curves, use of high-purity acids, and analysis of blanks and reference materials (SRM 2709 Montana Soil; NIST, 2002) were employed for better quality evaluation and quality control of the digestions and analyses.

Statistical analysis and source apportionment modelling

Three sequential statistical procedures were used to evaluate and select the geochemical tracers: (1) conservatism test (box plot) for eliminating chemical elements that did not exhibit conservative behavior during sediment delivery; (2) Kruskal-Wallis test (p<0.10) to select individual tracers with high potential to distinguish the sampled sediment sources, and; (3) linear discriminant analysis (forward stepwise) (p<0.10), for selecting the minimum set of geochemical tracers giving the highest discrimination between sources to be used in the source apportionment modeling. A Bayesian model (MixSIAR) was used to estimate the relative contributions of sediment from individual sources (Stock and Semmens, 2016). This model has been used extensively for studying sediment source tracing (Latorre et al., 2021; Batista et al., 2022). No results generated by the model were rejected, as all showed Gelman-Rubin values above 1.01.

Evaluation of the performance of this modeling was undertaken using virtual mixtures and a comparison of known and predicted contributions based on root mean square error (RMSE) and mean absolute error (MAE). Finally, eleven virtual mixtures for each

group of tracers and sub-catchments were established: 1) 50 % surface sources and 50 % subsurface sources; 2) 60 % surface sources and 40 % subsurface sources; 3) 40 % surface sources and 60 % subsurface sources; 4) 70 % surface sources and 30 % subsurface sources; 5) 30 % surface sources and 70 % subsurface sources; 6) 75 % surface sources and 25 % subsurface sources; 7) 25 % surface sources and 75 % subsurface sources; 8) 80 % surface sources and 20 % subsurface sources; 9) 20 % surface sources and 80 % subsurface sources; 10) 90 % surface sources and 10 % subsurface sources; 11) 10 % surface sources and 90 % subsurface sources. Statistical tests and modeling procedures were performed using R software (version 4.3.1).

RESULTS AND DISCUSSION

Tracer conservation

Figures 3, 4 and 5 present the conservatism tests for the Sapocaji, Piedade and Minas sub-catchments, respectively. Elements Ce, Gd, La, Nd, Pr, Sc, and Sm exhibited conservative behavior in all three sub-catchments. Some elements exhibited conservative behavior in one or two of the sub-catchments, such as Ba, Cr, Mn, Ni, Sr, Th, Ti, V, and Y. Other elements were not conservative in any of the sub-catchments, such as Fe, Sn, and Zn, mainly showing enrichment during sediment transport. High contents of sedimentassociated Zn have been reported for the Ipojuca River (Silva et al., 2015), possibly originating from point anthropogenic sources, which could explain the non-conservative behavior of this specific element.

Sediment source discrimination

Kruskal-Wallis H-test was applied to indicate if the elements that passed the conservatism test have potential to differentiate the sampled sediment sources. A p-value<0.10 indicates tracers with the ability to distinguish the two sources (Table 2) significantly. Accordingly, only Ce showed potential for discriminating sediment sources in all three sub-catchments. Only Cr, Mn, Ce, Sc showed conservative behavior and potential for source discrimination in the Sapocaji sub-catchment. However, a broader set of potential tracers were identified for both the Piedade (Al, Ba, Ce, Gd, La, Nd, Pr, Sm and Th) and the Minas (Al, Ni, V, Ce, La, Nd, Sm, Y) sub-catchments.

Linear discriminant analysis (LDA) results are presented in table 3, which summarises the set of elements that showed the highest potential for source discrimination in each sub-catchment, and critical LDA parameters: the Wilks' Lambda test, F-value, and cumulative error for the selected tracers. Here, a Wilks' Lambda value of 1 indicates no discrimination, while 0 indicates total discrimination.

Generally, the geochemical tracers provided Wilks' Lambda values below 0.5 and cumulative source discrimination errors of <15 %, indicating moderate or significant discrimination of the source material samples. F-value showed the contribution of each tracer in discriminating the source samples. Higher F-values indicate that a given tracer is more important for source discrimination. Some tracers, such as Mn in the Sapocaji and Nd in the Minas sub-catchment, proved to be particularly useful for discriminating the two sediment sources.

Distribution and contribution of sediment sources

Average contributions of the sampled sources in the sub-catchments (Figure 6) were analyzed for both suspended sediments (SS) and bed sediments (BS). Sugarcane was considered a surface source (SFS), while unpaved roads and channel banks were considered subsurface sources (SBS). Overall, surface source contribution to both SS and BS was higher (Figure 6), except for the Sapocaji sub-catchment, where subsurface sources showed a slightly higher contribution for BS.

Figure 3. Boxplots of the geochemical tracer contents that were approved by the conservatism test in the Sapocaji sub-catchment. SFS: surface sources; SBS: subsurface sources; SS: suspended sediments; BS: bed sediments.

In the Sapocaji sub-catchment, approximately equal source contributions were estimated: 55 % surface source and 45 % subsurface source contributions to SS, compared with 49 % from surface sources and 51 % from subsurface sources for BS. Piedade sub-catchment showed a more pronounced divergence in source contributions to SS, with 90 % originating from surface sources and only 10 % from subsurface sources. However, BS sources showed more balance, with 50 % originating from surface sources and 50 % from subsurface sources. Minas sub-catchment exhibited more consistent similarity in the source contributions to SS and BS, with the majority originating from surface sources, accounting for 62 %, in both cases. Subsurface sources in the Minas sub-catchment contributed 38 % of SS and BS.

Sugarcane agriculture areas, particularly in the Piedade sub-catchment, have a significant impact on sediment contributions, agreeing with the results reported by another study on the origin of sediments sampled in the main stem of the Ipojuca River (Nascimento et al., 2023). A key factor involved with sugarcane sediment dynamics in this region occurs after harvesting, when sugarcane is burned and harvested manually, a practice known to expose the soil to higher erosion rates (Thomaz et al., 2022). However, when

Figure 4. Boxplots of the geochemical tracer contents that were approved by the conservatism test in the Piedade sub-catchment. SFS: surface sources; SBS: subsurface sources; SS: suspended sediments; BS: bed sediments.

Figure 5. Boxplots of the geochemical tracer contents that were approved by the conservatism test in the Minas sub-catchment. SFS: surface sources; SBS: subsurface sources; SS: suspended sediments; BS: bed sediments.

Table 2. Mean tracer values and Kruskal-Wallis p-values for each tracer in the three sub-catchments

SFS: surface sources; SBS: subsurface sources.

sugarcane cultivation follows best management practices, these can provide important protection against elevated soil erosion in the region (Bezerra and Cantalice, 2006). Riparian vegetation is much better preserved in the Piedade than in the other two sub-catchments, accounting for the lower contribution of subsurface sources in this sub-catchment system.

There was a pronounced discrepancy in source contributions between SS and BS in the Piedade sub-catchment. Such a finding triggers the assumption that most of the sediments delivered by surface sources are being quickly transported to the outlet instead of being deposited on the river bed. For the Minas sub-catchment, surface sources were also more important (Figure 6), but with similar contributions for both SS and BS.

Although they generally contributed less than surface sources, subsurface sources (i.e., channel banks and unpaved roads) were nevertheless important. Here, channel banks were generally more important than unpaved roads, reflecting the existence of tall uncovered bank faces formed by the river network, with limited vegetation protection (Amorim et al., 2021; Nascimento et al., 2023). A lack of riparian vegetation is prevalent in the Sapocaji sub-catchment, favoring sediment detachment (Figure 7), and thereby explaining the higher contributions of subsurface sources to the BS in this sub-catchment system.

Each sub-catchment demonstrated variations in sediment source contributions. Therefore, sub-catchment-specific sediment management measures need to be implemented. However, riparian forests revegetation, in compliance with current Brazilian legislation, could be an efficient common sediment control measure in the sub-catchments, and

Tracers Wilks' Lambda F-value Accumulated error $\frac{0}{0}$ Sapocaji Mn 0.48 25.43 7.69 Cr 0.40 16.89 11.53 Piedade Ce 0.78 8.51 25.00 Al 0.60 9.55 18.75 Ba 0.55 7.56 12.50 Th 0.43 8.74 12.50 Gd 0.38 8.50 9.37 Minas Nd 0.66 13.32 21.42 Al 0.55 10.29 21.42 Ni 0.49 8.13 14.28

Table 3. Results of the Linear Discriminant Analysis (LDA) for selecting the best set of tracers for each sub-catchment

especially in the Sapocaji sub-catchment. This practice can stabilize riverbanks and reduce sediment inputs in the longer term. Currently, the law requires the maintenance of native vegetation protection areas with a fixed width along watercourses. In this regard, Guidotti et al. (2020) reported that a 15 m wide vegetative filter strip (native forests) between the stream bank and agricultural areas is sufficient to guarantee river system protection.

Accuracy of the modeling using virtual mixtures

Results of the virtual mixing analyses, as well as the RMSE and MAE values for each sub-basin, are detailed in table 4. The overall average for RMSE and MAE, considering all cases of modeling with virtual mixtures, was 14 %, ranging from 0 to 35 %. Most contribution estimates were considered acceptable, resembling the accuracy reported in

Relative contribution for each sub-basin

Figure 6. Average relative contributions of surface sources (SFS) and subsurface sources (SBS) for each sub-catchment: Sapocaji (Sap), Piedade (Pie), and Minas (Min), for both suspended sediments (SS) and bed sediments (BS).

Figure 7. Channel banks evidencing collapse or exposed without riparian vegetation in the middle course of the Sapocaji subcatchment.

previous sediment source studies that used a Bayesian approach (Raigani et al., 2019; Nosrati et al., 2021).

Comparative analysis of the relative contributions estimated and known by means of virtual mixtures revealed that the RMSE and MAE were substantially lower in the Sapocaji sub-basin (9 %), indicating greater effectiveness of the tracer set and the model in predicting the results in this sub-basin (Table 4). On the other hand, the RMSE and MAE averages in the other sub-basins showed similar values, equating to 16 % (range 0 to 29 %) for the Piedade sub-basin and 16 % (range 0 to 35 %) for the Minas sub-basin.

Limitations and uncertainties associated with the study

Fingerprinting modeling of sediment sources in the three sub-basins of Ipojuca River has been statistically validated. However, we consider it crucial to recognize some limitations and uncertainties associated with the results found in this study. According to the virtual mixture tests, it is likely that the Piedade and Minas sub-basins exhibited greater errors in the estimates of source contributions (Table 4). This can be attributed to different factors, including the number of suspended sediment composite samples, a possible erroneous tracer inclusion in the final set for each sub-basin, and the limitation in the ability of geochemical tracers to discriminate the two sources in these sub-basins significantly.

Results for the Piedade sub-basin showed the pattern of sediment delivery for a relatively short observation period. Unlike the other sub-basins, suspended sediment sampling in the Piedade sub-basin was restricted to the high water discharge period, notably in May and June, with only two campaigns being carried out. The low number of samples made up of suspended sediments runs counter to the trend observed in most fingerprinting studies and could have an adverse impact on the quality of obtained data. However, the riverbed composite sediment sampling ($n = 5$) satisfactorily meets the representativeness of these sediments. In this sense, we encourage additional studies to explore the temporal variability of sediment source delivery to assess the effects of interannual rainfall variations in this study area.

A significant proportion of the chemical elements showed conservative behavior. However, the range test used in this modeling has inherent limitations, and there are currently no fully effective statistical tests for assessing the complex behavior of tracers during sediment transport (Collins et al., 2020). Thus, some non-conservative tracers might be incorporated into the final composite signatures used in this modeling. Here, it is feasible to consider that a selected tracer in the final composite signatures has undergone chemical

Table 4. Results of the predicted and known contributions from surface sources (SFS) and subsurface sources (SBS) to the virtual mixtures and the RMSE (Root Mean Square Error) and MAE (Mean Absolute Error) for the sub-catchments

> transformations and variations in its concentration during sediment transport, which, although not of sufficient magnitude to fail the range test for conservative behavior, might be enough to impact the modeled sediment source contributions (Nosrati et al., 2019).

> Reduced ability to differentiate sediment sources in the Minas sub-basin may have contributed to significant errors in the source estimates. Therefore, we encourage the exploration of other properties that could potentially strengthen the discrimination among sediment sources. A viable tracer for this study region is phosphorus (P) and its fractionation, since surface soil layers under cultivation generally have higher P concentrations than subsurface layers. In addition, other options to discriminate surface and subsurface sources, which have not been tested in this tropical environment, include Pb²¹⁰ levels and the stable isotopes of carbon (C) and nitrogen (N).

Modeling in the Sapocaji catchment could also be more accurate. As already pointed out, a substantial proportion of tracers did not show conservative behavior in this sub-catchment, suggesting that including a larger number of tracers could improve source discrimination and increase the model accuracy. We believe that a small urban area adjacent to the outlet of the main course may have exerted a significant influence on the sediment load and/or the input of chemicals. So, three scenarios can be considered: (1) a potential source of sediment may not have been considered in this study; (2) the urban area had a substantial impact on the geochemistry of the sediments transported in this part of the river; (3) both scenarios may have acted simultaneously. Preventing this scenario would have been possible by collecting suspended sediment samples before the urban area, or by including this area as a potential sediment source in the sampling strategy.

CONCLUSIONS

Identification of sediment sources provided new insights for sediment management in the downstream portions of the Ipojuca River in Brazil. Surface soils under sugarcane were responsible for the highest contribution to SS. This is a particularly significant finding for the state of Pernambuco, Brazil, since the coastal humid areas of its territory are marked by the extensive presence of this particular agricultural practice.

The source apportionment results also suggested a small change in the quality of riverbank management accounted for substantial differences in the contributions of this source. Accordingly, the degraded, unvegetated channel banks of the Sapocaji sub-catchment were fundamental for the supply of river sediments, while the more preserved margins of the other sub-catchments generated smaller subsurface contributions.

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