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1 Quantification of sediment source contributions in two paired catchments of the 2 Brazilian Pampa using conventional and alternative approaches

3
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18 19 Abstract

20 The knowledge of the contribution of sediment sources to river networks is a
21 prerequisite to understand the impact of land use change on sediment yield. We
22 calculated the relative contributions of sediment sources in two paired catchments, one
23 with commercial eucalyptus plantations (0.83 km²) and the other with grassland used
24 for livestock farming (1.10 km²), located in the Brazilian Pampa biome, using different
25 combinations of conventional (geochemical - G, radionuclide - R and stable isotopes
26 and organic matter properties - S) and alternative tracer properties (spectrocolorimetric
27 visible-based-color parameters - V). Potential sediment sources evaluated were stream
28 channel, natural grassland and oat pasture fields in the grassland catchment, and
29 stream channel, unpaved roads and eucalyptus plantation in the eucalyptus catchment.
30 The results show that the best combination of tracers to discriminate the potential
31 sources was using GSRV tracers in the grassland catchment, and using GSRV, GSV and
32 GS tracers in the eucalyptus catchment. In all these cases, samples were 100% correctly
33 classified in their respective groups. Considering the best tracers results (GSRV) in both
34 catchments, the sediment source contributions estimated in the catchment with
35 eucalyptus plantations was 63, 30 and 7% for stream channel, eucalyptus stands and
36 unpaved roads, respectively. In the grassland catchment, the source contributions to
37 sediment were 84, 14 and 2% for natural grassland, stream channel and oats pasture
38 fields, respectively. The combination of these source apportionment results with the
39 annual sediment loads monitored during a 3-year period demonstrates that commercial
40 eucalyptus plantations supplied approximately ten times less sediment (0.1 ton ha⁻¹ yr⁻¹
41 ¹) than the traditional land uses in this region, i.e. 1.0 ton ha⁻¹ yr⁻¹ from grassland and
42 0.3 ton ha⁻¹ yr⁻¹ from oats pasture fields. These results demonstrate the potential of
43 combining conventional and alternative approaches to trace sediment sources
44 originating from different land uses in this region. Furthermore, they show that well-
45 managed forest plantations may be less sensitive to erosion than intensively grassland
46 used for livestock farming, which should be taken into account to promote the
47 sustainable use of land in this region of South America.

48
49 **Keywords:** Soil erosion; Eucalyptus; Sediment fingerprinting; Spectroscopy.

1. Introduction

Soil erosion is a major process of land degradation worldwide. In the Pampa biome in Southern Brazil, soils developed on sedimentary parent materials are characterized by a sand-to-silt sized texture (Roesch et al., 2009). These relatively coarse soils are particularly sensitive to degradation processes (Becker et al., 2009; Kaiser, Reinert, Reichert, Streck, & Pellegrini, 2010; Reichert, Amado, Reinert, Rodrigues, & Suzuki, 2016; Reichert et al. 2015).

The Pampa biome in Brazil is exclusively found in the southernmost state of Rio Grande do Sul where it covers 63% of the surface corresponding to 2% of the national area (MMA, 2018). The so-called "Campanha" region corresponds to a significant portion of this biome, and it is located in the southern half of the State, naturally covered by herbaceous vegetation and sparse trees. Natural grassland play an important role in the economy, culture and the way of life of the "gaucho" society (Boldrini et al., 2010). During the Iberian Colonization, cattle was introduced in this region by the Jesuits around 1634 and, since then, livestock farming on natural grassland has provided the main economic activity in this region (MMA, 2018).

In contrast, forestry has a much lower spatial extent (3%) although it is currently carried out on 780,900 hectares. Among these activities, 55% of forested areas correspond to eucalyptus plantations, 34% to pinus, and 11% to acacia plantations (AGEFLOR, 2017). In 2000, 253,000 hectares were covered with pine and eucalyptus (SBS, 2012). Then, in 2004, additional plantations were installed in the Pampa biome in areas that were previously devoid of significant forest activity. This expansion is a response to the increased demand for forest-based raw products, although it raised numerous concerns associated with the potential problems occurring when introducing exotic and fast-growing tree species in this region of natural grassland (Andriollo, Redin, Reichert, & Silva, 2017; Mateus & Padilha, 2017; Reichert et al., 2017). Since then, the increase in eucalyptus plantations in this region has raised many questions regarding the possible impacts of this activity on the degradation of water and soil resources, as there is limited information on their environmental impact in South America (Portela et al., 2015; Bormann et al., 2016).

The identification of those areas that deliver sediment to rivers draining this region may be achieved through the comparison of the physical and chemical properties of both soils and sediments. For this purpose, investigations using fine sediment fingerprinting methods have become increasingly popular over the last several decades, after their initial development in the 1970s (Haddadchi, Olley, & Pietsch, 2015; Koiter et al., 2013).

The most frequently used tracers in the conventional method are fallout radionuclides (e.g. ^{137}Cs and unsupported ^{210}Pb or $^{210}\text{Pb}_{\text{ex}}$), geochemical elements and organic matter properties (Evrard et al., 2013; Laceby, Huon, Onda, Vaury, & Evrard, 2016; Laceby et al., 2017; Mabit et al., 2009; Minella, Merten, & Reichert, 2007; Minella, Clarke, Merten, & Walling, 2008; Schuller et al., 2013). The selection of a combination of discriminant properties and the use of a multivariate mixing model provide a common technique to quantify the source contributions to sediment. This statistical procedure has been

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3 97 successfully applied to a wide range of contrasted environments. In addition to these
4 98 conventional methods, faster, cheaper and non-destructive alternative techniques
5 99 were developed such as those based on spectroscopic properties. Among other
6 100 benefits, these techniques require small quantities of sample (~ 100 mg). Both
7 101 conventional and alternative properties may be combined to increase the
8 102 discrimination between the sources and the precision of the model outputs (Collins &
9 103 Walling, 2002).
10 104

11 105 To date, few studies compared the use of different parameter combinations when
12 106 conducting sediment fingerprinting approaches in Brazil (Tiecher, Caner, Minella, & dos
13 107 Santos, 2015). Furthermore, this type of approach has not been tested in catchments
14 108 covered with natural grassland or commercial eucalyptus plantations. Although the soil
15 109 surface is better protected against rainfall and runoff by the canopy of the forest
16 110 plantations, soil tillage and management, tree harvesting, construction and
17 111 maintenance of forest roads may increase the sensitivity of soils to erosion in these
18 112 systems (Ferreira, Gonçalves, & Dias, 2008; Oliveira, Bertol, Campos, & Júnior, 2014;
19 113 Sheridan, Noske, Whipp, & Wijesinghe, 2006). Forest roads frequently provide the main
20 114 sediment sources in forested areas (Croke & Mockler, 2001; Hairsine, Croke, Mathews,
21 115 Fogarty, & Mockler, 2002) and, in those areas with inappropriate road planning, forest
22 116 roads may supply more than 90% of the sediment produced (Grace III, Rummer, &
23 117 Stokes, 1998; Madej 2001). Sediment fingerprinting has also proved to be efficient in
24 118 areas with pasture and livestock farming (Collins, Walling, & Leeks, 1997; Collins,
25 119 Walling, Webb, & King, 2010; Owens, Walling, & Leeks, 2000). Sediment mobilization in
26 120 pastures and livestock farming areas is commonly related to soil compaction because of
27 121 animal trampling (Ambus, Reichert, Gubiani, & Carvalho, 2018; Holthusen, Brandt,
28 122 Reichert, & Horn, 2018). When natural grassland is well managed, only low soil loss rates
29 123 are observed (Motha, Wallbrink, Hairsine, & Grayson, 2002; Nosrati et al., 2011).
30 124

31 125 Most studies devoted to the quantification of sediment sources in Brazil were
32 126 conducted in agricultural catchments (e.g. Franz, Makeschin, Weib, & Lorz, 2014; Le Gall
33 127 et al., 2016; Miguel, Dalmolin, Pedron, & Moura-Bueno, 2014a; Miguel, Dalmolin,
34 128 Pedron, Moura-Bueno, & Tiecher, 2014b; Minella, Merten, & Clarke, 2009; Minella,
35 129 Walling, & Merten, 2014; Tiecher et al., 2014, 2015, 2017a; b; 2018). Tiecher et al.
36 130 (2017b) investigated the sediment source contributions in two paired agricultural
37 131 catchments with different proportions of riparian vegetation and wetlands and similar
38 132 proportions of crop fields. Stream channel were the main source of sediments ($49 \pm$
39 133 26%) in the catchment with the lower proportion of riparian vegetation and wetlands,
40 134 while in the other study site with a higher proportion of riparian vegetation, the stream
41 135 channel contribution amounted to only $23 \pm 14\%$. Furthermore, in another eucalyptus
42 136 forested catchment of this region, Rodrigues et al. (2018) identified stream channel as
43 137 the main sediment source using a set of geochemical variables.
44 138

45 139 Although the soil surface is better protected under forest plantations, practices such as
46 140 soil preparation, management, harvesting, construction and maintenance of forest
47 141 unpaved roads increased the susceptibility of soils to erosion in these systems (Ferreira
48 142 et al., 2008; Lima, 1996; Oliveira, 2014; Sheridan et al., 2006). Several studies
49 143 demonstrated that forest roads are the main source of sediment in forested areas
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3 144 (Croke et al., 2001; Hairsine et al., 2002; Grace III et al., 1998; Madej, 2001). High values
4 145 of soil (22 t ha^{-1}) and water losses (44% of rainfall) were also observed on forest unpaved
5 146 roads by Oliveira et al. (2014).
6 147

7 148 The identification and the quantification of the sediment source contributions and their
8 149 potential spatial and temporal variations constitute a prerequisite for designing
9 150 effective management measures in order to reduce the supply of sediment and
10 151 contaminants to the river systems (Brosinsky, Foerster, Segl, & Kaufmann, 2014;
11 152 Cooper, Krueger, Hiscock, & Rawlins, 2014). Accordingly, the objective of the current
12 153 study was to investigate the relative contribution of potential sediment sources in two
13 154 paired catchments, with different land uses (eucalyptus plantation and livestock
14 155 farming) representative of those found in the Brazilian Pampa biome. As different
15 156 results in sediment source proportions can be obtained when different tracer sets or
16 157 mixing models are used (Martinez-Carreras et al., 2010; Haddadchi et al., 2013; Cooper
17 158 et al., 2014; Pulley et al., 2015; Laceby and Olley, 2015; Palazón and Navas, 2017; Nosrati
18 159 et al., 2018; Uber et al., 2019), conventional and alternative fingerprinting approaches
19 160 were applied and the differences obtained were discussed, making a critical analysis on
20 161 the selection of tracers and their implication on the results obtained when calculating
21 162 the contribution of sediment sources, as well as on the potential use of alternative
22 163 spectroscopy-based methods.
23 164

24 165 **2. Materials and methods**

25 166

26 167 **2.1. Study site and general setup**

27 168

28 169 The study was conducted in two paired catchments (Figure 1). The first is covered with
29 170 eucalyptus plantations (*Eucalyptus saligna*) managed by the CMPC Riograndense
30 171 Cellulose company. The second is covered by natural grassland and livestock farming
31 172 located at Farm Alvorada, a private property (see Supplementary material).
32 173

33 174 These catchments are located in the municipality of São Gabriel, in the Campanha
34 175 physiographic region in the Rio Grande do Sul State, southern Brazil. Both catchments
35 176 flow into the Vacacaí and Vacacaí-Mirim rivers, which drain a total area of $11,077 \text{ km}^2$
36 177 and are located in the center-western portion of the State, between the
37 178 geomorphological provinces of “Depressão Central” and “Escudo Sul Rio-Grandense”
38 179 (SEMA, 2017), which in turn drain into the Guaíba River Basin, and finally to the Atlantic
39 180 Ocean.
40 181

41 182 Climate is classified as humid subtropical (Cfa), and characterized by the absence of
42 183 drought with an average annual temperature of 18.6 °C and an average annual rainfall
43 184 of 1356 mm (Moreno, 1961; Alvares, Stape, Sentelhas, Gonçalves & Sparovek, 2013).
44 185 Soils in both catchments are derived from the weathering of metamorphic and granite-
45 186 gneiss rocks (amphibolite metamorphism; orthogneiss lithologies, metadiorite, and
46 187 metaperidotite), according to Ramgrab et al. (2004). These soils are predominantly
47 188 physically fragile and characterized by a low natural fertility and a low agricultural
48 189 potential (Morales, 2013).
49 190

191 The soils are classified as Argissolo Vermelho (Ultisols), Cambissolo (Inceptisols), and
192 Neossolo (Entisols), respectively according to the Brazilian Soil Classification System
193 (EMBRAPA, 2006) and the US Soil Taxonomy System (USDA, 1999).

195 The catchment with eucalyptus plantations (EC) has a drainage area of 0.83 km² and a
196 perimeter of 4.17 km, with a 1.51 compactness coefficient and an average time of
197 concentration of 2:52 h calculated based on average rainfall events (Reichert et al.,
198 2017). The EC landscape is characterized by elevations comprised between 230 and 315
199 m asl, with an average elevation of 272 m and a mean slope of 7.7%.

201 Land use is dominated by *Eucalyptus saligna* (40% of total area planted in 2006 and 21%
202 in 2014 replacement after cutting), natural grassland with brush weeds (22%), riparian
203 vegetation (8%), unpaved roads (6%), and rock outcrops (3%). Eucalyptus plantations
204 had 3.0 × 3.3 m spacing and the stand planted in 2006 had an average diameter at breast
205 height of 0.17 m and an average height of 25 m. The natural grassland with *Baccharis*
206 spp. consisted of grasses and shrubs, in which *Aloysia gratissima* (Verbenaceae) and
207 *Heterothalamus alienus* (Asteraceae) were the most abundant species. The riparian
208 vegetation consisted of arboreal stratum of native species, with individuals of 6–8 m in
209 height, namely *Sebastiania commersoniana*, *Rollinia salicifolia*, *Styrax leprosus*, *Eugenia*
210 *uniflora*, *Luehea divaricata*, *Casearia decandra*, *Diospyros inconstans*, *Myrcianthes*
211 *pungens* and *Ocotea* spp. (Peláez, 2014).

213 The catchment with grassland (GC) has a drainage area of 1.10 km² and a perimeter of
214 4.32 km, with a 1.22 compactness coefficient and an average time of concentration of
215 1:47 h. The GC landscape is characterized by elevations between 255 and 310 m asl with
216 an average elevation of 273 m and a mean slope of 3.1%.

218 Land use mainly corresponds to degraded natural grassland (62% of the total area of
219 the GC), pasture composed of oats pasture fields (*Avena strigosa*) (31%), eucalyptus
220 patches and isolated individuals (3%), riparian vegetation (2%) and reservoir (2%). The
221 GC upper vegetation stratum in degraded natural grassland was composed of
222 *Saccharum angustifolium*, *Aristida laevis*, *Baccharis riograndensis*, *Andropogon lateralis*
223 and *Eryngium pandanifolium*, whereas the lower stratum consisted of *Paspalum* spp.,
224 *Axonopus affinis* and *Fimbristylis autumnalis* (Peláez, 2014). The degraded natural
225 grassland has a low vegetation cover, which is due to intensive grazing and the absence
226 of liming and fertilizer application to improve soil fertility for pasture growth. Cattle
227 grazing reduced species diversity and soil quality due to animal trampling, which
228 reduced the quantity of biomass and further exposed the soils to erosion processes.
229 Animal stock in this area is on average one head of cattle per hectare. The cultivated
230 pasture is sown annually with black oats followed by disc harrowing to incorporate
231 seeds in the soil once a year, generally in April or May. This area has received lime
232 application although fertilization was not performed.

234 **2.2 Hydro-sedimentary monitoring**

236 The monitoring was conducted from March 2014 to February 2017 using automated
237 monitoring sections, equipped with instruments measuring water level (limnigraphs),

238 turbidity (turbidimeters) and rainfall (pluviograph) installed near the catchment outlets
 239 and the data loggers recorded data at 10-minute intervals. Then, SSC was estimated
 240 using an equation on the basis of data available for the rainfall–runoff events
 241 establishing the relationship between turbidity (nephelometric turbidity units) and SSC
 242 (mg L^{-1}), more details are provided in Valente (2018). The sediment discharge (kg s^{-1})
 243 was obtained by multiplying water discharge (L s^{-1}) by the SSC (mg L^{-1}). Suspended
 244 sediment yield (SSY; ton year^{-1}) was then calculated integrating these values over time,
 245 according to Equation 1.

$$246 \quad SY = \sum_{i=1}^n k(SS_c * Q_i) \quad (1)$$

247 where: SY is the sediment yield (ton), SS_c is the sediment concentration (g L^{-1}), Q_i is the
 248 flow (L s^{-1}), k is a unit conversion factor, n is a number of instantaneous measurements
 249 of SS_c and Q performed at a given time.

250

251 **2.3. Sediment source sampling**

252

253 Potential sediment sources were identified through the observation of sediment
 254 mobilization and transport processes during storm events in the catchments. Three
 255 main sediment source types were identified in each catchment. In the eucalyptus
 256 catchment, eucalyptus plantations (EP), unpaved roads (UR) and stream channels (SC)
 257 were considered, while in the grassland catchment oats pasture fields (OF), natural
 258 grassland (NG) and stream channels (SC) provided the main potential sources.

259

260 Source material was collected between May 2015 and January 2016 (Figure 1). A plastic
 261 spatula was used to collect samples and avoid potential metal contamination by
 262 scraping the upper layer of the soil (2–3 cm). Each source sample was composed of at
 263 least ten sub-samples. All samples were oven-dried at low temperature ($<40\text{ }^{\circ}\text{C}$) to avoid
 264 the degradation of organic matter (Remusat et al., 2012) and sieved to 0.063 mm for
 265 further analysis. The number of source samples analysed for each type of tracing
 266 property and the respective sample quantities are described in Table 1. It is important
 267 to note that, due to logistical reasons, the number of measurements carried out for
 268 each approach was different depending on the catchment.

269

270 **2.4. Sediment sampling**

271

272 River sediment samples were collected between March 2014 and February 2017 at the
 273 catchment outlet following four strategies: (i) *suspended matter samples* were collected
 274 with a US-DH-48 sampler (3/16" diameter) during rainfall-runoff events; (ii) *time-*
 275 *integrated suspended sediment* samples were collected through the deployment of two
 276 sediment traps consisting of a 75 mm diameter polyethylene pipe with 80 cm in length
 277 in each catchment (Philips, Russell & Walling, 2000); (iii) *bed load sediment* samples
 278 were collected with a US-BLH-84 sampler, whereas 20 to 40 sub-samples were
 279 composited to provide representative samples (Edward & Glysson, 1999); and (iv) *lag*
 280 *deposits* were collected after storm-events in the vicinity of the catchment outlets,
 281 according Horowitz, Meybeck, Idlafkih & Biger (1999). After collection, all samples were
 282 evaporated following the method of Shreve & Downs (2005) and sieved to 0.063 mm
 283 for further analysis.

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4 285 **2.5. Soil and sediment analyses**

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7 287 Total concentrations in B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Na, Ni, Sr, Ti, V
8 288 and Zn, were analysed by ICP-OES after microwave assisted digestion for 9.5 min at 182
9 289 °C with concentrated HCl and HNO₃ in the ratio 3:1 (aqua regia) for both soil and
10 290 sediment samples.

11 291
12
13 292 For radionuclide measurements, the ¹³⁷Cs, ²¹⁰Pb_{ex}, ²²⁶Ra and ²³⁴Th activities were
14 293 determined by gamma spectrometry using low background N and P type GeHP
15 294 detectors (Canberra and Ortec). Measured activities were decay-corrected to the
16 295 sampling date and provided with 2σ-errors.

17 296
18
19 297 N and C content, and the δ¹³C and δ¹⁵N isotopic signatures, were determined using an
20 298 on-line continuous flow elemental analyzer Flash 2000 HT, coupled with an Isotopic
21 299 Ratio Mass Spectrometer Delta V Advantage via a conflow IV interface from Thermo
22 300 Fischer Scientific.

23 301
24
25 302 Spectrocolorimetric measurements were measured using a portable diffuse reflectance
26 303 spectrophotometer (Konica Minolta 2600d). The spectrophotometer was calibrated
27 304 before each set of measurements by making a zero and a white calibration. Control
28 305 measurements on red, green and yellow panels as well as six contrasting soil samples
29 306 were also taken regularly during a set of measurements. As recommended in the
30 307 literature (Legout et al., 2013), three measurements were taken for each sample (~0.1
31 308 g), where each sample respected the predefined composition ratio of source, as shown
32 309 in Figure 2, because of the rather small measuring area (3 mm radius circle), and to
33 310 account for the possible heterogeneity within the samples. Diffuse reflectance was
34 311 measured in the visible range between 360 and 740 nm with a 10 nm resolution. Raw
35 312 data collected were the spectral reflectance percentage for each of the 39 wavelength
36 313 classes. From the raw spectral data, 15 color coefficients were calculated using the
37 314 equations provided by the *Commission Internationale de l'Eclairage* (CIE) (1978). These
38 315 include the xyz chromaticity coordinates and parameters derived from various color
39 316 spaces. A detailed information regarding color coefficients used in our study (L*, a*, b*,
40 317 C*, h, x, y, z, L, a, b, u*, v*, u', v') can be found in Viscarra Rossel, McGlynn, & McBratney
41 318 (2006), Legout et al. (2013), Tiecher et al. (2015) and Uber et al. (2019).

42 319
43
44 320 **2.6. Sediment source discrimination and apportionment for discrete tracer properties**

45 321
46
47 322 The discrete properties (19 geochemical, 4 radionuclide, 2 stable isotope and 2 organic
48 323 matter properties and 15 spectrocolorimetric visible-based-color parameters) were first
49 324 submitted to an outlier and 'range test'. Samples and the variables with sediment
50 325 concentrations lying outside of the range of properties found in the sources were
51 326 identified and removed (Smith & Blake, 2014).

52 327
53
54 328 Afterwards, properties passing the 'range test' were submitted to a two-step statistical
55 329 selection. First, the Kruskal-Wallis *H*-test was performed to test the null hypothesis
56 330 (p<0.05) that the sources are belonging to the same population. Then, different

331 combinations of the selected variables were entered in a discriminant function analysis
 332 (DFA) in order to determine the minimum number of variables that maximizes the
 333 discrimination between the sources. Five combinations of tracer sets were tested
 334 including the conventional tracers [geochemical elements (G), stable isotopes and
 335 organic matter properties (S) and radionuclides (R)] and the alternative tracers [based
 336 on the VIS-based-color parameters (V)] were tested in the catchments (e.g. GSRV, GSV,
 337 GS, G and V).

338
 339 After defining the set of discriminant variables for each combination of tracer
 340 properties, the contribution of each source to the sediment samples was determined.
 341 Equation 2 describes the mathematical relationship between the proportions of
 342 contribution of each source and the variables measured in the sources and in suspended
 343 sediment (Walling & Woodward, 1995).

$$344 \quad y_i = \sum_{s=1}^n a_{is} P_s \quad (s = 1, 2, \dots, n) \text{ and } (i = 1, 2, \dots, m) \quad (2)$$

345 where: y_i is the value of the variable i measured in suspended sediment, a_{is} are the linear
 346 model coefficients (concentration of property in source s_i) and P_s is the contribution
 347 from the source s , which may be presented as a set of linear functions of m variables
 348 and n sources. To determine the P_s values, an objective function was used (Walling &
 349 Woodward, 1995). The solution was found through an iterative process with the
 350 objective to minimize the value of R (f mincon) (Equation 3).

$$351 \quad R = \sum_{i=1}^m \left\{ \frac{C_i - \left(\sum_{s=1}^n P_s C_{si} \right)^2}{C_i} \right\} \quad (3)$$

352
 353 where: m is the number of soil properties selected as variables tracer, n is the number
 354 of sources, C_i is the concentration of the tracer i in the suspended sediment sample, P_s
 355 is the proportion of contribution of the source s , C_{si} is the average value of the tracer i
 356 obtained at the source s .

357
 358 The mixing model was run using Matlab® software. During the minimization process, P
 359 values had to meet two constraints: (i) values of P_s must be higher than 0% and lower
 360 than 100%, (ii) and the sum of P_s values must be equal to 100%.

361
 362 The evaluation of the results was made by comparing the tracer values measured in
 363 suspended sediments and the value predicted by the model based on the contribution
 364 calculated for each source. Then, based on relative errors for each variable, the relative
 365 mean error (RME) was calculated to associate a unique error value with each suspended
 366 sediment sample according to Equation 4.

$$368 \quad RME = \sum_{i=1}^m \left\{ \frac{C_i - \left(\sum_{s=1}^n P_s C_{si} \right)}{m} \right\} \quad (4)$$

369

2.7. Sediment source apportionment for continuous visible raw reflectance spectra

The sediment fingerprinting approach used for continuous visible raw reflectance spectra was based on the construction of partial least squares regression models (PLSR), following the recommendations of Poulenard et al. (2009). The steps used in this alternative method were the calibration of partial least square regressions based on the preparation of artificial mixtures (n=74 samples for each catchment) of the sediment sources in known proportions. The distribution of the mixtures in the ternary diagram provided a compromise between the coverage of the whole triangle area and the preparation of a number of samples that were well distributed on each axis (Figure 2). Then, visible reflectance spectra were obtained for each composite sample. Relationships between visible spectra (x variate) and the corresponding weighted contribution of the sediment source datasets (y variate) were analyzed using partial least squares regression (PLSR). This data set was randomly divided into two parts, one half being used for calibration, the remaining half for validation. The statistical criteria used to assess the robustness of the model were the coefficient of determination (R^2) and the Root Mean Square Error (RMSE). It should be stressed that independent PLSR models were constructed for each of the three primary source material types, thus, each model estimates the proportion of one source, independently of the two others. Visible raw reflectance spectra of suspended sediment were then introduced into these VIS-PLSR models to estimate the contribution of each sediment source. Finally, the quality of the tracing procedure was checked by summing the three predicted proportions and verifying that this sum was close to 100% for each catchment (Legout et al., 2013).

3. Results

3.1. Sediment source discrimination in the grassland catchment

Table 3 shows all the 42 parameters analyzed in soils and sediment collected in the natural grassland catchment. Thirteen of these properties did not pass the range test, as sediment concentration was lying outside of the range of properties found in the potential sources, indicating that they may not be conservative and were thus removed in the next steps. Among the remaining parameters, B, $\delta^{15}\text{N}$, ^{137}Cs , $^{210}\text{Pb}_{\text{ex}}$, ^{228}Ra and ten VIS-based-color parameters passed the KW-test ($p < 0.10$) which were then considered as potential tracers.

The different parameters combinations (GSRV) comprising ten tracers selected by DFA analyses, lead to a final value of the Wilks' lambda value (Λ^*) of 0.0008. This means that 99.9% of the variance between the source groups was explained by these ten tracers (Table 3). Although three geochemical tracers (B, Mn and Zn) were entered in the DFA-analyses, none of them was selected. This means that for this catchment, geochemical tracers have less discriminating potential than tracers such as radionuclides, stable isotopes and organic matter properties and color. This observation is corroborated by the fact that the approach considering only geochemical tracers was associated with the worst source discrimination. Using only geochemical tracers the Wilks' lambda value (Λ^*) was 0.40, which means that only 60% of the variance between the source groups was explained by these ten tracers (Table 3).

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4 418 For the grassland catchment, the color parameters provided better discrimination than
5 419 the geochemical properties, and they performed also better than the combination of
6 420 geochemical properties and stable isotopes and organic matter properties, explaining
7 421 95% of the variability between sources and correctly classifying 95% of the samples into
8 422 their respective groups (Table 3).

9 423
10 424 Mahalanobis distance is a multi-dimensional measurement of dispersion of a certain
11 425 point from the mean. Thus, the greater the Mahalanobis distance between the
12 426 centroids of the sediment sources, the greater the difference between them. Although
13 427 the distances between all sources were significantly different, the scattering of the
14 428 points within each group may introduce a source of uncertainty. Accordingly, when a
15 429 sample is classified correctly, it is important to consider the distance between this point
16 430 and the group central point (Figure 3). Whilst the use of color (V) and color tracers
17 431 combined with geochemicals elements and stable isotopes and organic matter
18 432 properties (GSV) could explain most of the variability between sources (95 and 97%
19 433 respectively), the Mahalanobis distance between sediment sources was approximately
20 434 8 to 5 times smaller than in the GSRV approach, respectively (Table 3 and Figure 3).
21 435 Consequently, the uncertainties in the classification of the samples was much higher in
22 436 the approaches V and GSV (13.1 and 10.9%, respectively) compared to the GSRV
23 437 approach (<0.1%).
24 438

25 439 **3.2. Sediment source discrimination in the eucalyptus catchment**

26 440
27 441 In the eucalyptus catchment, only B, Ba, Ca, K, Sr and ^{228}Ra did not pass the range test
28 442 and were then excluded from further analysis (Table 4). Then, Co, Fe, Ni and $\delta^{15}\text{N}$ did
29 443 not pass the KW-test and were also excluded. DFA analysis (Table 5) performed using
30 444 GSRV, GSV and GS approaches indicates that all samples were correctly classified in
31 445 their respective groups (Table 5). Moreover, the Wilk's Lambda values of these three
32 446 approaches were very similar and indicate that between 99.6 and 99.9% of the variation
33 447 between sources was explained by the set of tracers selected by DFA-analyses.
34 448 Consequently the uncertainties associated with the sample classification of these three
35 449 approaches was very low (<0.1%).

36 450
37 451 Compared to approaches combining different sets of tracers (GSRV, GSV and GS), the
38 452 isolated use of color (V) and geochemical (G) tracers resulted in a lower power of
39 453 discrimination, with greater uncertainty (22-32%) and a shorter Mahalanobis distance
40 454 (Table 5 and Figure 3). Unlike grassland catchment, in the eucalyptus catchment the
41 455 source discrimination using only color or geochemical tracers was very similar (Table 5).
42 456 Using only three geochemical tracers and two color tracers, it was possible to explain
43 457 approximately 85 and 81% of the variation between sources, respectively, and correctly
44 458 classify 84 and 74% of the samples (Table 5).

45 459 46 460 **3.3. Source apportionment results in both catchments**

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48 462 Figure 4 shows the performance of the VIS-PLSR models constructed independently for
49 463 the three types of source materials. In the grassland catchment, the determination

coefficients were similar for each individual model (0.94, 0.95 and 0.94) predicting the contribution of oats pasture fields, stream channel and natural grassland. As mentioned in Legout et al. (2013) it should be stressed that these rather good values of determination coefficient obtained during the construction of the VIS-PLSR approach do not ensure a high performance of these models when applied to suspended sediments. However Table 5 shows that the mean predicted sum of 117% remains in the range of uncertainty (20%) that can be expected from such techniques. This approach leads to contributions of the natural grassland ranging between 25 to 98%, between 2-68% from the stream channel, and 0-57% from oats pasture (Table 6 and Figure 5).

Considering the results obtained with the best tracer combination (GSRV) in the catchment with natural grassland, the estimated source contributions to sediment was 84, 14 and 2% for natural grassland, stream channel and oats pasture fields, respectively (Table 6 and Figure 5).

In the eucalyptus catchment (Figure 6), considering the best tracers results (GSRV), the contribution estimated was 63, 30 and 7% for stream channel, eucalyptus stands and unpaved roads, respectively (Table 7 and Figure 6).

The sums of the sediment source contributions predicted by the three VIS-PLSR models were higher in the eucalyptus catchments than in the natural grassland, with a mean value of 126 and a maximum of 157% (Table 7).

487

488 **3.4. Weighted source contributions**

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Considering the percentage of source contributions obtained with the best approach in both catchments (GSRV group parameters – Tables 3, 5, 6 and 7), the weighted source contributions to total suspended sediment discharges during the monitored period, in the natural grassland catchment, were 67.6, 84.7 and 88.1 ton yr⁻¹ in 2014-2015, 2015-2016, and 2016-2017, respectively (Table 8). In the eucalyptus catchment, total suspended sediment discharges for each year amounted to 23.2, 29.6 and 15.7 ton yr⁻¹ (Table 8).

497

In the grassland, sediment yield from oats pasture fields ranged from 0.3 to 0.4 ton ha⁻¹ year⁻¹, and in the natural grassland it ranged from 0.8 to 1.1 ton ha⁻¹ year⁻¹. On the other hand, in the eucalyptus catchment, the sediment yield in eucalyptus plantations was lower, with values ranging from 0.1 to 0.2 ton ha⁻¹ year⁻¹.

502

The contribution of the stream channel was different in both catchments (2% to GC and 63% to EC), with the mean sediment yield values of 1.6 and 14.4 ton yr⁻¹ during the monitored period in the grassland and eucalyptus catchments, respectively.

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507

508 4. Discussion

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510 4.1. Sediment tracing methods

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512 These results suggest that the best set of tracers may be strongly site specific, even
513 when paired catchments with very similar relief, soil, and lithology characteristics are
514 investigated. One of the major challenges associated with the implementation of a
515 sediment fingerprinting approach is to provide physico-chemical information
516 supporting the tracer selection. The results obtained in the current researcher
517 demonstrate that even in close paired catchments, there is a large variability in those
518 tracers that are selected for source discrimination. For instance, in the grassland
519 catchment, few geochemical tracers provided discrimination between the sources (B,
520 Mn and Zn).

521

522 The low concentrations in geochemical elements made it difficult to outline significant
523 composition differences between the different types of sources in the grassland
524 catchment, which correspond to contrasted land uses. In contrast, despite relatively low
525 activities, fallout radionuclides (^{137}Cs , $^{210}\text{Pb}_{\text{xs}}$ and ^{228}Ra) provided an effective technique
526 for source discrimination in the grassland catchment, and the fallout radionuclides
527 $^{210}\text{Pb}_{\text{xs}}$, ^{137}Cs and ^{234}Th in the eucalyptus catchment, when low-background and efficient
528 gamma spectrometry detectors are available to conduct measurements given the low
529 levels of radiocesium found in the Southern Hemisphere.

530

531 The advantages of using composite signatures including tracers from several types of
532 tracer properties have been previously emphasized in the literature (Evrard et al., 2010;
533 Martínez-Carreras et al., 2010; Olley & Caitcheon, 2000). However, the widespread use
534 of some of these tracers, especially that of radionuclides, is limited by the long analyses,
535 their expensive cost and the large quantity of sample required compared to
536 spectrophotometric analyses, which are quick, inexpensive and require lower quantities
537 of samples.

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540 4.2. Sediment source apportionment

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542 In the grassland catchment, considering the approach with the best tracer combination
543 (GSRV), the main sediment source was natural grassland (84%), followed by oats
544 pasture fields (14%) and stream channel (2%). These results are in agreement with those
545 obtained in other studies conducted in rural catchments of Rio Grande do Sul, Southern
546 Brazil, which indicated that cropland ($91\pm 15\%$) was the main source of sediment and
547 low stream channel contributions ($5\pm 2\%$) (Tiecher et al., 2017b). The dominance of
548 natural grassland originating sediment is consistent with the large surface area occupied
549 by this potential source in the catchment and with the high erosion rates observed in
550 these areas compared to those found in the eucalyptus catchment. In Brazil, agricultural
551 activities often provide the main source of soil erosion, accelerating the sediment
552 transfer into water bodies (Tiecher et al., 2015). When synthesizing data from Brazil,
553 Anache, Wendland, Oliveira, Flanagan, & Nearing (2017) observed that cropland

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3 554 showed higher soil losses than grasslands and pasture in most studies, although this
4 555 result depended on the density of the soil cover by vegetation.
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7 557 In areas with extensive livestock, the degradation of soil physical conditions under
8 558 pasture is associated with cattle trampling leading to soil compaction (Müller, Ceccon &
9 559 Rosolem, 2001) and a drop in macroporosity. These processes reduce water infiltration
10 560 into the soil, generating a higher percentage of surface runoff. Cattle may become an
11 561 important bioerosive agent changing relief forms and accelerating geomorphological
12 562 processes in these areas (Thomaz & Dias, 2009).
13 563

14 564 The occurrence of stream channel collapses was observed in both catchments during
15 565 the study period, which demonstrates the importance of stabilizing stream channel to
16 566 reduce the availability of sediment sources. Furthermore, when looking for shade and
17 567 water, cattle may invade legal protection areas along rivers and their drainage channels,
18 568 thereby leading to trampling on areas where vegetation is regenerating (see the
19 569 illustrations of this phenomenon on the Supplementary material). Álvarez-Yépez,
20 570 Martínez-Yrizar, Búrquez, & Lindquist (2008) observed that the rivers and natural
21 571 reservoirs where animals are drinking have their stream channel unprotected due to
22 572 the frequent traffic of animals, which also leads to the accelerated siltation in river
23 573 channels and reservoirs, the degradation of riparian forests and their capacity for
24 574 renewal.
25 575

26 576 In the eucalyptus catchment, large amounts of lag deposits were found in the stream
27 577 channel after floods (see Supplementary material). Marttila & Klove (2010) observed
28 578 that the combined effects of weathering, groundwater seepage, and geotechnical
29 579 instability accelerated local stream channel collapse. However, all these processes are
30 580 not well understood in forested areas, and these issues require further research. Our
31 581 results indicate that the stream channel may provide a significant source of sediment,
32 582 especially in the eucalyptus catchment. In larger stream systems, stream channel
33 583 derived sediment can supply more than 50% of the sediment transiting these rivers
34 584 (Knighton, 1998). In one forested catchment of Rio Grande do Sul (Terra Dura Forest
35 585 Catchment), Rodrigues et al. (2018) found that stream channel provided the major
36 586 sediment contribution. Accordingly, management actions should be focused on those
37 587 areas located in the immediate vicinity of the river rather than in the eucalyptus
38 588 plantations themselves if they are well-managed forests/silviculture. Despite the low
39 589 sediment contribution from eucalyptus plantations and those stream channel sections
40 590 protected by a dense cover of riparian vegetation, the higher sediment contribution of
41 591 stream channel in other unprotected river sections illustrates the inherent fragility of
42 592 the soils in this region, independent of the surface area covered by riparian vegetation
43 593 or the type of riparian plants.
44 594

45 595 The lower contribution of eucalyptus plantations to sediment transiting the river
46 596 (average of $0.1 \text{ ton ha}^{-1} \text{ year}^{-1}$) compared that of the other main traditional land uses in
47 597 the grassland catchment (average of 0.3 and $1.0 \text{ ton ha}^{-1} \text{ year}^{-1}$ for oats pasture fields
48 598 and natural grasslands, respectively) (Table 8) demonstrates clearly the effectiveness of
49 599 the current management measures implemented in these eucalyptus plantations to
50 600 decrease soil erosion, including a sufficient density of surface litter and a good canopy

601 protection and runoff control. This indicates that there are better structural conditions
602 for water infiltration into the soil (Guimarães, 2015) under eucalyptus plantations, likely
603 reducing the sediment contribution from these areas. Similar observations were made
604 by Mello, Lima, & Silva (2007) and Rodrigues et al. (2018).

605
606 In forested catchments, the canopy acts as a barrier against the precipitation that
607 reaches the soil, reducing the amount of rainfall and redistributing it on a larger area on
608 the ground (Chang, 2012). Achieving a minimum soil cover by vegetation of 30% with
609 crop residues or forest canopy is fundamental to reduce soil losses due to water erosion
610 (Rodrigues, 2011). When comparing suspended sediment yields among various
611 undisturbed forest sites, Zimmermann, Francke & Elsenbeer (2012) clearly showed that
612 hydrological characteristics strongly influence suspended sediment dynamics in
613 forested areas. These authors observed that overland flow relocates leaf litter and
614 transports loose soil material. Thus, the transport of soil material probably accelerates
615 during the progressing wet season due to a positive feedback mechanism. The first rains
616 remove the leaf litter in flowlines while transporting relatively little amount of soil, while
617 subsequent events carry increasing amounts of fine soil material which seals
618 macropores in the flowlines. The authors also claim that major rainfalls cause substantial
619 storm-flow, which triggers localized stream channel failure. During these high-
620 connectivity events some hillslopes loose more than 200 kg ha⁻¹ of suspended-sediment
621 during a single rainfall event. This illustrates that a well developed tree cover is not
622 necessarily capable of preventing surface erosion and although the vegetation reduces
623 erosion to some degree and forests cannot inhibit erosion completely, a pronounced
624 soil anisotropy (expressed as the change of the saturated hydraulic conductivity with
625 depth) favors the activation of runoff.

626
627 Although resistance of soil to erosion is higher under forest plantations, soil tillage,
628 management, harvesting, and construction and maintenance of forest roads increase
629 the susceptibility to erosion of forest systems (Ferreira et al., 2008; Oliveira, 2014;
630 Sheridan et al., 2006). The low contribution of unpaved roads to sediment is likely due
631 to the small area covered by this land use in the investigated catchments. Usually, the
632 maintenance and the heavy traffic on unpaved roads increase the susceptibility of these
633 areas to soil erosion (Croke & Mockler, 2001; Ferreira et al., 2008; Hairsine et al., 2002;
634 Oliveira, 2014; Sheridan et al., 2006). In some catchments, forest unpaved roads may
635 contribute up to 90% of the sediment yield (Grace III et al., 1998; Madej, 2001), as a
636 result of inadequate planning.

637

638 **5. Conclusions**

639

640 The best discrimination between the potential sources in both catchments was using a
641 combination of geochemical, radionuclide, stable isotopes, organic matter properties,
642 and spectrophotometric visible-based-color tracers. The modelled source contributions
643 to sediments suggest that areas with commercial eucalyptus plantations contribute less
644 sediment to rivers than the traditional land use found in the region (natural grasslands
645 and oats pasture fields with extensive cattle). In the eucalyptus catchment, the mean
646 contribution for stream channel, eucalyptus stands and unpaved roads were,
647 respectively, 63, 30 and 7%, considering the GSRV parameters combination. In contrast,

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3 648 in the catchment mainly covered with natural grassland, the mean source contributions
4 649 to sediment were 84, 14 and 2% for natural grassland, stream channel and oats pasture
5 650 fields, respectively.
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8 652 The flux of sediment supplied by traditional extensive livestock areas to the river
9 653 network, whether under oats pastures ($0.3 \text{ ton ha}^{-1} \text{ year}^{-1}$) or under natural grasslands
10 654 ($1.0 \text{ ton ha}^{-1} \text{ year}^{-1}$), was larger than the soil losses occurring in forested land with
11 655 eucalyptus ($0.1 \text{ ton ha}^{-1} \text{ year}^{-1}$), indicating that additional efforts are necessary to
12 656 further reduce soil erosion in these areas. To improve this situation, an improved
13 657 pasture management should be introduced. Accordingly, there is an urgent need to
14 658 better plan livestock practices in Southern Brazil. The impact of the forest management
15 659 used in the plantation investigated in the current research showed to be effective in
16 660 reducing soil losses. Accordingly, the system including the rotation of the areas where
17 661 forest is cut could be expanded in similar areas. However, it is important to highlight
18 662 that during the evaluated period, there was no cut of eucalyptus. This operation can
19 663 certainly modify the contribution of the sediment sources due to intense traffic of heavy
20 664 machinery, both at the eucalyptus stands and on unpaved roads. Therefore, future
21 665 works must be developed in a longer time scale aiming to contemplate all the stages of
22 666 the eucalyptus cultivation.
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27 667

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37 677 the field.
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42 680 **Data Availability Statement**

43 681
44 682 The data that support the findings of this study are available from the corresponding
45 683 author upon reasonable request.
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Table 1.

Table synthesizing the number of samples and the sample quantities analysed for a range of potential tracing properties in both catchments.

Analysis	Sample quantity (g)	Source samples – Eucalyptus catchment			
		Unpaved road	Eucalyptus	Channel	Sediment
Geochemical	~0.05	9	11	21	23
Radionuclides	~1	7	6	6	5
Stable isotopes and organic matter properties	~0.5	9	11	21	23
Spectrocolorimetric	~0.1	9	11	21	23
Analysis	Sample quantity (g)	Source samples – Grassland catchment			
		Grassland	Oats	Channel	Sediment
Geochemical	~0.05	4	6	13	17
Radionuclides	~1	4	6	9	9
Stable isotopes and organic matter properties	~0.5	4	6	9	9
Spectrocolorimetric	~0.1	4	6	13	17

Note: Geochemical properties: B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Na, Ni, Sr, Ti, V and Zn; Radionuclides: ^{137}Cs , $^{210}\text{Pb}_{\text{ex}}$, ^{226}Ra and ^{234}Th ; Stable isotopes $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ and organic matter properties: N, C; Spectrocolorimetric properties: color-derived tracers (L*, a*, b*, C*, h, x, y, z, L, a, b, u*, v*, u', v').

Table 2.

Mean and standard deviation (SD) of potential discriminant parameters between land uses in the grassland catchment, including the significance level associated with the Kruskal–Wallis *H*-test and the range test comparing properties found in sediment and potential sources.

Variable	Kruskal-Wallis		Correct class. by DFA (%)	Oats		Grassland		Channel		Sediment		Sediment samples out of source range (%)	
	<i>H</i> -value	<i>p</i> -value*		Mea n	SD	Mea n	SD	Mea n	SD	Mea n	SD	Max ± SD	Min ± SD
<i>Geochemical (G)</i>				(n=6)		(n=4)		(n=13)		(n=17)		Higher	Lower
B (mg kg ⁻¹)	8.50	0.01	57	7.3	3.4	5.2	2.7	4.2	2.9	31.2	35.0	38	4
Ba (g kg ⁻¹)	†	-	-	0.1	0.0	0.1	0.0	0.2	0.1	0.4	0.2	63	0
Be (mg kg ⁻¹)	†	-	-	1.8	0.5	1.8	0.3	2.4	0.5	4.5	0.7	88	0
Ca (g kg ⁻¹)	†	-	-	0.2	0.1	0.1	0.0	0.1	0.1	0.9	0.6	46	0
Cd (mg kg ⁻¹)	†	-	-	0.4	0.2	0.6	0.3	0.5	0.2	1.0	0.4	78	0
Co (mg kg ⁻¹)	†	-	-	3.1	0.4	2.7	0.8	6.1	4.0	30.1	25.5	46	0
Cr (mg kg ⁻¹)	2.00	0.37	-	18.6	5.4	21.2	4.0	23.5	6.8	30.7	5.9	8	0
Cu (mg kg ⁻¹)	0.30	0.88	-	8.6	3.0	7.2	0.3	8.3	3.7	16.3	9.1	38	0
Fe (g kg ⁻¹)	†	-	-	13.9	3.5	14.0	1.3	14.1	2.8	26.6	6.9	75	4
K (g kg ⁻¹)	†	-	-	5.1	1.5	5.5	1.3	4.1	1.2	13.4	11.9	53	5
Li (mg kg ⁻¹)	0.50	0.76	-	20.8	8.7	17.0	4.6	20.9	6.7	29.6	9.2	13	0
Mg (g kg ⁻¹)	†	-	-	2.1	0.5	1.2	0.1	1.0	0.4	3.6	1.9	54	0
Mn (g kg ⁻¹)	5.20	0.07	-	0.3	0.1	0.2	0.0	0.4	0.4	1.6	1.1	38	0
Na (mg kg ⁻¹)	†	-	-	0.1	0.2	0.2	0.1	0.1	0.1	0.8	0.9	44	0
Ni (mg kg ⁻¹)	0.30	0.85	-	7.7	3.1	8.4	2.3	7.6	3.1	13.4	4.5	33	0
Sr (mg kg ⁻¹)	†	-	-	10.8	2.8	11.1	0.7	16.8	2.6	84.2	56.1	92	0
Ti (g kg ⁻¹)	1.30	0.52	-	1.6	0.4	1.6	0.1	1.3	0.8	1.7	0.4	0	0
V (mg kg ⁻¹)	0.00	0.98	-	41.1	9.9	43.7	5.9	40.6	11.7	52.8	10.4	8	0
Zn (mg kg ⁻¹)	5.50	0.06	-	19.2	6.8	15.3	2.5	12.9	3.3	36.9	20.9	33	0
<i>Radionuclides (R)</i>				(n=6)		(n=4)		(n=9)		(n=9)		Higher	Lower
¹³⁷ Cs (Bq kg ⁻¹)	12.40	<0.01	63	4.4	1.6	4.9	1.2	1.3	0.9	2.8	2.4	1	0
²¹⁰ Pb _{xs} (Bq kg ⁻¹)	12.90	<0.01	74	114.1	37.1	119.4	33.1	30.2	17.4	184.6	162.6	47	0
²²⁸ Ra (Bq kg ⁻¹)	5.20	0.07	68	115.0	25.9	83.7	12.6	85.4	10.5	106.3	111.8	23	0
²³⁴ Th (Bq kg ⁻¹)	2.40	0.31	-	116.8	39.1	84.6	25.7	98.3	17.3	127.9	139.1	44	0
<i>Stable isotopes and organic matter properties (S)</i>				(n=6)		(n=4)		(n=9)		(n=9)		Higher	Lower
δ ¹³ C (‰)	†	-	-	-19.3	0.8	-17.9	0.3	-16.4	1.0	-21.3	1.6	58	0
δ ¹⁵ N (‰)	10.00	0.01	57	6.5	0.6	5.1	0.6	7.2	1.1	4.2	1.5	0	33
C (%)	†	-	-	2.8	0.5	3.2	0.2	2.0	0.6	5.9	2.7	78	0
N (%)	†	-	-	0.3	0.0	0.3	0.0	0.2	0.1	0.5	0.2	78	0
<i>VIS-based-color parameters (V)</i>				(n=6)		(n=4)		(n=13)		(n=17)		Higher	Lower
L*	6.10	0.05	52	44.1	1.5	43.6	2.0	42.6	4.7	40.6	6.4	0	11
a*	6.20	0.04	65	7.7	1.7	6.4	0.9	6.5	0.7	7.6	2.0	5	0
b*	10.00	0.01	61	19.0	2.0	18.3	2.0	17.1	2.1	18.5	2.2	0	0
C*	9.10	0.01	61	20.5	2.5	19.3	2.2	18.3	2.2	20.0	2.7	5	0
h	22.10	<0.01	70	68.0	2.3	70.8	0.8	69.1	1.3	67.8	2.9	0	5
x	2.20	0.33	-	0.4	0.0	0.4	0.0	0.4	0.0	0.4	0.0	21	0
y	3.40	0.19	-	0.4	0.0	0.4	0.0	0.4	0.0	0.4	0.0	11	0
Z	1.70	0.43	-	0.2	0.0	0.2	0.0	0.2	0.0	0.2	0.0	0	16
L	6.10	0.05	52	37.3	1.4	36.8	1.9	36.0	4.3	34.1	5.7	0	11
a	7.10	0.03	65	5.9	1.3	4.8	0.7	4.9	0.6	5.6	1.5	5	0
b	19.50	<0.01	57	11.3	0.9	10.9	0.7	10.2	1.3	10.7	1.0	0	0
u*	10.60	0.01	61	20.1	3.2	17.7	2.0	17.2	2.0	18.8	3.5	5	0
v*	18.30	<0.01	57	20.8	1.7	20.2	1.5	18.8	2.4	19.9	1.7	0	0
u'	3.40	0.19	-	0.2	0.0	0.2	0.0	0.2	0.0	0.2	0.0	16	0
v'	2.00	0.37	-	0.5	0.0	0.5	0.0	0.5	0.0	0.5	0.0	11	0

†: variable removed after range test; bold values indicate significant difference between sediment sources at $p < 0.10$ by the Kruskal–Wallis *H*-test; -, not significant; "n" = number of samples.

Table 3.

Discriminant analysis results for the different groups of tracing parameter combinations in the grassland catchment.

DFA output	GSRV	GSV	GS	G	V
Wilks' Lambda	0.0008	0.0260	0.3028	0.3964	0.0553
Variability explained by variables (%)	99.9	97.4	69.7	60.4	94.5
Selected Variables	²¹⁰ Pb _{xs}	b	Zn	Zn	b
	a	b*	δ ¹⁵ N		b*
	a*	C*			C*
	b	L			L
	b*	L*			L*
	C*	u*			u*
	L	v*			v*
	L*	δ ¹⁵ N			
	v*				
	δ ¹⁵ N				
<i>Squared Mahalanobis distances</i>					
Oats vs. Grassland	447	69	3	1	48
Oats vs. Stream channel	352	15	5	8	12
Stream channel vs. Grassland	84	82	10	5	43
Average	260	52	7	6	34
<i>Source type classified correctly (%)</i>					
Stream channel	100	89	78	89	89
Oats	100	100	50	50	100
Grassland	100	100	75	100	100
Total	100	95	68	79	95
<i>Uncertainty associated with the discrimination of the source (%)</i>					
Stream channel	<0.1	12.3	28.1	33.0	15.2
Oats	<0.1	16.2	44.1	48.1	18.8
Grassland	<0.1	<0.1	23.8	46.7	<0.1
Average	<0.1	10.9	32.2	40.7	13.1

Table 4.

Mean and standard deviation (SD) potential discriminant parameters between land uses in the eucalyptus catchment, including the significance level associated with the Kruskal–Wallis *H*-test and the range test comparing properties found in sediment and potential sources.

Variable	Kruskal-Wallis		Correct class. by DFA (%)	Channel		Unpaved road		Eucalyptus		Sediment		Sediment samples out of source range (%)	
	<i>H</i> -value	<i>p</i> -value*		Mean	SD	Mean	SD	Mean	SD	Mean	SD	Max ± SD	Min ± SD
<i>Geochemical (G)</i>				(n=21)		(n=9)		(n=11)		(n=23)		Higher	Lower
B (mg kg ⁻¹)	†	-	-	5.2	2.5	5.5	2.0	7.9	2.0	22.1	22.2	44	0
Ba (g kg ⁻¹)	†	-	-	0.1	0.0	0.1	0.0	0.1	0.0	0.1	0.2	63	0
Be (mg kg ⁻¹)	13.40	<0.01	84	6.4	2.4	2.0	0.5	1.3	0.4	6.9	1.8	4	0
Ca (g kg ⁻¹)	†	-	-	0.1	0.0	0.1	0.0	0.2	0.1	0.8	0.1	52	0
Co (mg kg ⁻¹)	0.40	0.82	37	5.2	1.3	4.4	1.1	4.7	1.8	9.3	2.4	7	0
Cr (mg kg ⁻¹)	11.90	<0.01	68	16.0	11.1	29.8	6.2	20.8	7.8	23.1	6.3	11	0
Cu (mg kg ⁻¹)	5.90	0.05	58	17.7	5.5	11.9	2.6	8.2	2.4	23.1	6.7	11	0
Fe (g kg ⁻¹)	4.10	0.13	42	14.7	1.9	15.5	2.4	11.5	3.9	27.2	4.3	30	0
K (g kg ⁻¹)	†	-	-	9.4	3.0	6.0	1.2	3.2	1.5	9.7	1.6	44	0
Li (mg kg ⁻¹)	6.40	0.04	63	35.5	9.4	29.6	8.6	18.6	8.9	40.3	10.0	7	0
Mg (g kg ⁻¹)	12.10	<0.01	58	2.9	0.9	1.5	0.3	1.2	0.5	0.8	0.1	19	0
Mn (g kg ⁻¹)	8.10	0.02	63	0.3	0.1	0.2	0.1	0.3	0.1	1.0	1.6	37	0
Ni (mg kg ⁻¹)	4.40	0.11	47	7.4	3.5	11.7	3.3	7.7	3.0	12.0	4.7	0	0
Sr (mg kg ⁻¹)	†	-	-	14.4	5.3	12.6	2.5	11.6	4.0	74.5	67.8	48	0
Ti (g kg ⁻¹)	8.80	0.01	63	1.1	0.3	1.6	0.6	1.9	0.6	2.4	3.9	19	0
V (mg kg ⁻¹)	8.60	0.01	63	28.9	7.2	58.1	13.5	38.7	13.7	39.8	9.2	4	0
Zn (mg kg ⁻¹)	12.40	<0.01	79	35.7	8.6	18.5	3.6	13.1	2.6	44.9	18.3	4	0
<i>Radionuclides (R)</i>				(n=6)		(n=6)		(n=7)		(n=5)		Higher	Lower
¹³⁷ Cs (Bq kg ⁻¹)	6.00	0.05	63	0.8	0.5	1.8	1.2	2.0	0.6	0.8	0.7	0	0
²¹⁰ Pb _{xs} (Bq kg ⁻¹)	9.90	0.01	58	21.1	16.9	41.8	30.0	93.0	34.4	115.6	51.1	13	0
²²⁸ Ra (Bq kg ⁻¹)	†	-	-	110.0	20.8	95.9	28.4	84.2	17.2	184.3	39.2	63	0
²³⁴ Th (Bq kg ⁻¹)	12.20	<0.01	79	199.4	89.3	79.0	14.2	70.7	12.0	193.4	36.3	0	0
<i>Stable isotopes and organic matter properties (S)</i>				(n=21)		(n=9)		(n=11)		(n=23)		Higher	Lower
δ ¹³ C (‰)	5.80	0.05	74	-23.8	2.1	-19.5	1.2	-22.7	1.1	-24.3	1.6	0	0
δ ¹⁵ N (‰)	5.60	0.06	63	8.3	1.9	8.2	0.6	6.6	0.8	3.9	0.3	25	0
C (%)	10.60	<0.01	47	0.5	0.5	1.8	0.9	2.7	0.9	3.6	1.6	13	0
N (%)	10.10	0.01	47	0.0	0.0	0.2	0.1	0.2	0.1	0.3	0.2	13	0
<i>VIS-based-color parameters (V)</i>				(n=21)		(n=9)		(n=11)		(n=23)		Higher	Lower
L*	77.30	<0.01	68	59.0	8.4	39.9	3.3	42.7	4.4	49.3	2.9	0	0
a*	30.50	<0.01	63	7.2	1.3	12.2	4.5	7.9	2.4	6.1	0.5	0	0
b*	22.90	<0.01	55	21.2	2.8	22.0	4.3	18.1	2.1	14.4	1.4	0	0
C*	21.00	<0.01	57	22.4	2.9	25.3	6.0	19.8	2.9	15.6	1.4	0	0
h	68.00	<0.01	68	71.3	2.1	62.3	4.5	67.1	3.5	66.2	1.5	0	0
x	40.50	<0.01	66	0.4	0.0	0.4	0.0	0.4	0.0	0.4	0.0	0	0
y	24.30	<0.01	56	0.4	0.0	0.4	0.0	0.4	0.0	0.4	0.0	0	0
Z	34.90	<0.01	64	0.2	0.0	0.2	0.0	0.2	0.0	0.3	0.0	0	0
L	77.30	<0.01	65	52.3	8.5	33.5	3.0	36.1	4.0	42.4	2.8	0	0
a	24.20	<0.01	56	6.1	1.1	9.2	3.5	5.9	1.6	4.8	0.3	0	0

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2	b	45.00	<0.01	65	14.4	2. 1	11.9	1.6	10. 6	0.5	9.1	0.7	0	0
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4	u*	23.60	<0.01	54	21.8	3. 1	27.1	8.2	19. 5	3.7	15. 8	1.1	0	0
5														
6	v*	42.80	<0.01	64	25.5	3. 4	21.6	2.8	19. 4	1.0	16. 4	1.4	0	0
7														
8	u'	48.30	<0.01	66	0.2	0. 0	0.3	0.0	0.2	0.0	0.2	0.0	0	0
9														
9	v'	30.70	<0.01	64	0.5	0. 0	0.5	0.0	0.5	0.0	0.5	0.0	0	0

†: variable removed with range test; bold values indicate significant difference between sediment sources at $p < 0.10$ by the Kruskal–Wallis H -test; -, not significant, “n” = number of samples.

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Table 5.

Discriminant analysis results for the different groups of tracing parameter combinations in the eucalyptus catchment.

DFA output	GSRV	GSV	GS	G	V
Wilks' Lambda	0.0036	0.0008	0.0028	0.1514	0.1863
Variability explained by variables (%)	99.6	99.9	99.7	84.9	81.4
Selected Variables	²³⁴ Th	a*	a*	Be	b
	a*	Be	b	Mg	u'
	Cu	C	L	Zn	
	L*	Cr	L*		
	Li	h	Mg		
	Mg	L*	Mn		
	N	Li	V		
	Ti	Mg	v*		
	z	Mn	x		
		Ti	z		
		V	Zn		
		x			
<i>Squared Mahalanobis distances</i>					
Channel vs. Unpaved roads	168	1,29	154	15	16
Channel vs. Eucalyptus	46	919	48	12	14
Unpaved roads vs. Eucalyptus	117	57	206	5	2
Average	107	729	135	10	10
<i>Source type classified correctly (%)</i>					
Channel	100	100	100	83	83
Unpaved road	100	100	100	83	67
Eucalyptus	100	100	100	86	71
Total	100	100	100	84	74
<i>Uncertainty associated with the discrimination of the source (%)</i>					
Channel	<0.1	<0.1	<0.1	17	16
Unpaved road	<0.1	<0.1	<0.1	28	41
Eucalyptus	<0.1	<0.1	<0.1	21	37
Average	<0.1	<0.1	<0.1	22	32

Table 6.

Sediment source contributions predicted by the different approaches in the grassland catchment.

Sediment sample	GSRV	GSV	GS	G	V	VIS-PLSR	GSRV	GSV	GS	G	V	VIS-PLSR	GSRV	GSV	GS	G	V	VIS-PLSR	GSRV	GSV	GS	G	V	VIS-PLSR
(yy/mm/dd)	Oats pasture fields contribution (%)						Natural grassland contribution (%)						Stream channel contribution (%)						RME (%)					Sum (%)
Event 14.06.29	-	-	0	36	33	-	-	-	100	0	32	-	-	-	0	64	35	-	-	111*	194*	54*	0	-
Event 14.07.04 -1	-	-	42	0	36	-	-	-	58	0	32	-	-	-	0	100	31	-	-	13	17	18	5	-
Event 14.07.04 -2	42	24	47	4	35	-	58	76	53	0	32	-	0	0	0	96	33	-	10	15	26*	30*	1	-
Event 14.10.30	-	-	36	59	35	-	-	-	64	0	33	-	-	-	0	41	33	-	-	32*	53*	65*	4	-
Event 14.12.21	0	0	0	51	30	-	100	100	100	0	33	-	0	0	0	49	37	-	37*	44*	76*	77*	1	-
Event 15.10.07	0	0	0	0	0	-	100	100	100	0	31	-	0	0	0	100	69	-	976*	1107*	1937*	57*	0	-
Event 15.10.08	-	-	0	38	31	-	-	-	100	0	33	-	-	-	0	62	36	-	-	3636*	6362*	45*	0	-
Event 16.03.31	0	0	0	36	35	17	100	100	100	0	32	21	0	0	0	64	33	68	-	76*	133*	46*	1	106
Lag deposit 14.08.20	-	-	34	0	36	-	-	-	66	0	32	-	-	-	0	100	32	-	-	9	15	16	0	-
Lag deposit 15.11.24	0	0	7	0	34	-	100	100	93	0	32	-	0	0	0	100	34	-	105*	142*	248*	317*	0	-
Lag deposit 16.02.03	33	5	13	0	30	-	48	94	87	0	34	-	19	1	0	100	36	-	68*	52*	87*	103*	4	-
Lag deposit 16.05.11	0	0	0	48	0	32	100	100	100	0	0	5	0	0	0	52	100	68	116*	19	27*	28*	8	106
Trap 14.05.05	-	-	37	37	0	0	-	-	63	0	36	85	-	-	0	63	64	45	-	23*	33*	34*	11	130
Trap 14.08.14	-	-	0	38	0	35	-	-	100	0	31	98	-	-	0	62	69	2	-	105*	183*	221*	0	135
Trap 15.03.19	-	-	0	38	100	-	-	-	100	0	0	-	-	-	0	62	0	-	-	42*	63*	44*	8	-
Trap 15.07.17	-	-	0	38	32	57	-	-	100	0	34	50	-	-	0	62	35	2	-	27*	40*	38*	10	109
Trap 16.03.31	7	0	0	0	36	-	93	100	100	0	32	-	0	0	0	100	32	-	15	14	21*	17	3	-
Trap 16.10.12	44	0	36	40	35	-	56	100	64	0	33	-	0	0	0	60	33	-	15	18	28*	29*	3	-
Overall mean	14	3	14	26	30	59	84	97	86	0	29	52	2	0	0	74	41	37	4	4	7	18	3	117
Mean for RME <20%	14	3	14	26	30	40	84	97	86	0	29	26	2	0	0	74	41	60	4	4	7	18	3	126

RME = relative mean error; "-" = no sample analyzed; "*" = values with relative mean error more than 20%:

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Table 7.
Sediment source contributions predicted by the different approaches in the eucalyptus catchment.

Sediment sample (yy/mm/dd)	GSRV	GSV	GS	G	V	VIS-PLSR	GSRV	GSV	GS	G	V	VIS-PLSR	GSRV	GSV	GS	G	V	VIS-PLSR	GSRV	GSV	GS	G	V	VIS-PLSR
	Stream channel contribution (%)						Unpaved road contribution (%)						Eucalyptus contribution (%)						RME (%)					Sum (%)
Event 14.09.10	-	81	10	100	0	8	-	0	0	0	0	58	-	19	90	0	100	91	-	19	24*	11	9	157
Event 14.10.30	56	-	53	100	0	-	6	-	0	0	35	-	38	-	47	0	65	-	19	-	15	27*	1	-
Event 16.01.08	82	66	49	75	0	-	0	1	0	19	0	-	18	33	51	6	100	-	28*	9	15	12	2	-
Event 16.07.06	-	69	54	89	0	60	-	0	0	6	35	13	-	31	46	4	65	60	-	35*	5	0	1	132
Event 16.10.18	-	88	40	100	0	-	-	0	0	0	0	-	-	12	60	0	100	-	-	13	15	19	2	-
Lag deposit 14.07.05	-	78	38	65	0	-	-	0	0	23	35	-	-	22	62	13	65	-	-	3	21*	34*	1	-
Lag deposit 14.08.20	67	34	26	41	0	-	0	0	0	59	0	-	33	66	74	0	100	-	10	27*	23*	34*	2	-
Lag deposit 14.09.20	-	77	44	97	0	-	-	0	0	3	0	-	-	23	56	0	100	-	-	3	7	1	2	-
Lag deposit 14.12.20	-	-	35	60	0	-	-	-	0	25	34	-	-	-	65	15	66	-	-	-	21*	34*	1	-
Lag deposit 15.03.12	-	-	47	68	0	-	-	-	0	18	0	-	-	-	53	14	100	-	-	-	7	1	1	-
Lag deposit 15.06.18	-	-	53	71	0	-	-	-	0	16	34	-	-	-	47	13	66	-	-	-	5	2	1	-
Lag deposit 15.09.15	-	77	36	88	0	-	-	0	0	8	0	-	-	23	64	4	100	-	-	3	8	1	3	-
Lag deposit 15.11.24	-	44	42	100	0	-	-	0	7	0	0	-	-	56	51	0	100	-	-	26*	7	3	0	-
Lag deposit 16.02.03	-	41	36	60	0	-	-	0	0	25	34	-	-	59	64	15	66	-	-	28*	21*	34*	1	-
Lag deposit 16.05.11	62	57	26	82	0	-	0	0	0	11	0	-	38	43	74	7	100	-	5	4	10	1	5	-
Lag deposit 16.06.23	62	53	-	-	-	58	0	0	-	-	-	0	38	47	-	-	-	40	5	4	-	-	-	99
Lag deposit 16.11.15	-	31	22	45	0	-	-	0	0	55	0	-	-	69	78	0	100	-	-	30*	25*	34*	2	-
Trap 14.02.12	-	66	28	100	0	34	-	0	0	0	0	35	-	34	72	0	100	49	-	28*	22*	25*	2	117
Trap 14.05.05	58	77	76	100	0	-	42	0	0	0	38	-	0	23	24	0	62	-	17	15	8	8	0	-
Trap 15.03.19	71	92	84	-	-	-	0	0	0	-	-	-	29	8	16	-	-	-	18	21*	15			-
Trap 15.07.17	-	73	18	100	0	-	-	0	0	0	0	-	-	27	82	0	100	-	-	16	17	5	6	-
Trap 16.03.31	-	-	-	89	0	-	-	-	-	8	33	-	-	-	-	3	67	-	-	-	-	12	1	-
Trap 16.03.18	69	51	45	98	0	-	0	0	0	2	33	-	31	49	55	0	67	-	13	10	7	4	1	-
Overall mean	66	64	41	82	0	40	6	0	0	13	15	27	28	36	59	5	85	60	12	11	10	6	2	126
Mean for RME <20%	63	71	48	90	0	40	7	0	0	6	15	26	30	29	52	4	85	60	12	11	10	6	2	126

RME = relative mean error; "-" = no sample analyzed; "*" = values with relative mean error more than 20%.

Table 8.

Weighted sediment yield for each dominant land use in both grassland and eucalyptus catchments.

Catchment	Source	Monitoring year	SY total (ton) (min±max)	Source (%)*	SY source mean (ton yr ⁻¹)	Area source (ha)	SY Source (ton ha ⁻¹ yr ⁻¹)
Grassland	Oats pasture fields	Year 1 (mar/14-fev/15)	67.6 (0.6±29.1)	14	9.5	34.2	0.3
		Year 2 (mar/15-fev/16)	84.7 (0.5±25.7)	14	11.9	34.2	0.3
		Year 3 (mar/16-fev/17)	88.1 (0.5±37.9)	14	12.3	34.2	0.4
		Mean	80.1 (0.5±30.9)	14	11.2	34.2	0.3
	Natural Grassland	Year 1 (mar/14-fev/15)	67.6 (0.6±29.1)	84	56.8	67.9	0.8
		Year 2 (mar/15-fev/16)	84.7 (0.5±25.7)	84	71.1	67.9	1.0
		Year 3 (mar/16-fev/17)	88.1 (0.5±37.9)	84	74.0	67.9	1.1
		Mean	80.1 (0.5±30.9)	84	67.3	67.9	1.0
	Stream channel**	Year 1 (mar/14-fev/15)	67.6 (0.6±29.1)	2	1.4	-	-
		Year 2 (mar/15-fev/16)	84.7 (0.5±25.7)	2	1.7	-	-
		Year 3 (mar/16-fev/17)	88.1 (0.5±37.9)	2	1.8	-	-
		Mean	80.1 (0.5±30.9)	2	1.6	-	-
Eucalyptus	Eucalyptus plantations	Year 1 (mar/14-fev/15)	23.2 (0.1±9.5)	30	7.0	50.6	0.1
		Year 2 (mar/15-fev/16)	29.6 (0.2±10.2)	30	8.9	50.6	0.2
		Year 3 (mar/16-fev/17)	15.7 (0.2±6.8)	30	4.7	50.6	0.1
		Mean	22.8 (0.2±8.8)	30	6.8	50.6	0.1
	Unpaved roads**	Year 1 (mar/14-fev/15)	23.2 (0.1±9.5)	7	1.6	-	-
		Year 2 (mar/15-fev/16)	29.6 (0.2±10.2)	7	2.1	-	-
		Year 3 (mar/16-fev/17)	15.7 (0.2±6.8)	7	1.1	-	-
		Mean	22.8 (0.2±8.8)	7	1.6	-	-
	Stream channel**	Year 1 (mar/14-fev/15)	23.2 (0.1±9.5)	63	14.6	-	-
		Year 2 (mar/15-fev/16)	29.6 (0.2±10.2)	63	18.6	-	-
		Year 3 (mar/16-fev/17)	15.7 (0.2±6.8)	63	9.9	-	-
		Mean	22.8 (0.2±8.8)	63	14.4	-	-

*Mean of all approaches analysis according Tables 5 and 6. ** Estimation of SY for channel and unpaved roads is given only in ton yr⁻¹.

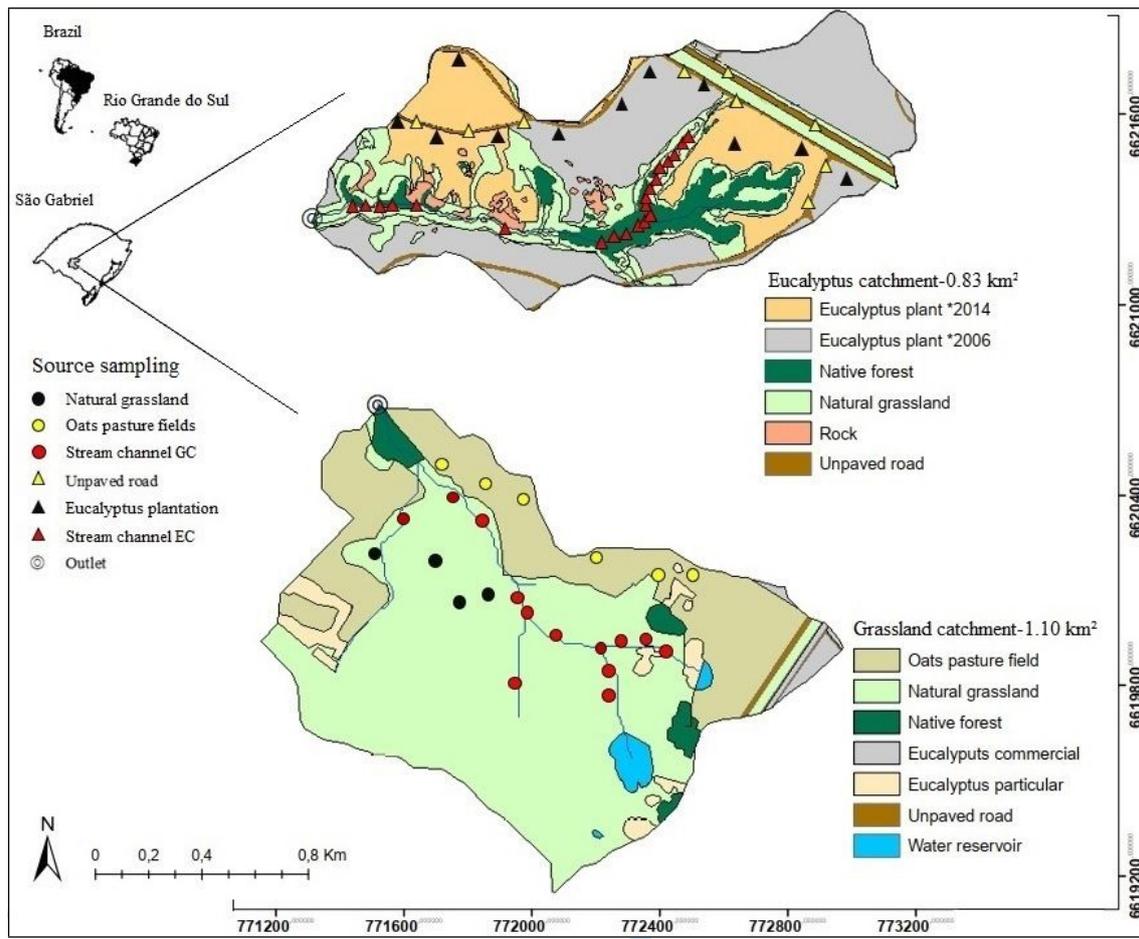


Fig. 1.

Location of the sampling sites and distribution of the main land uses in the investigated eucalyptus and grassland catchments, in Southern Brazil.

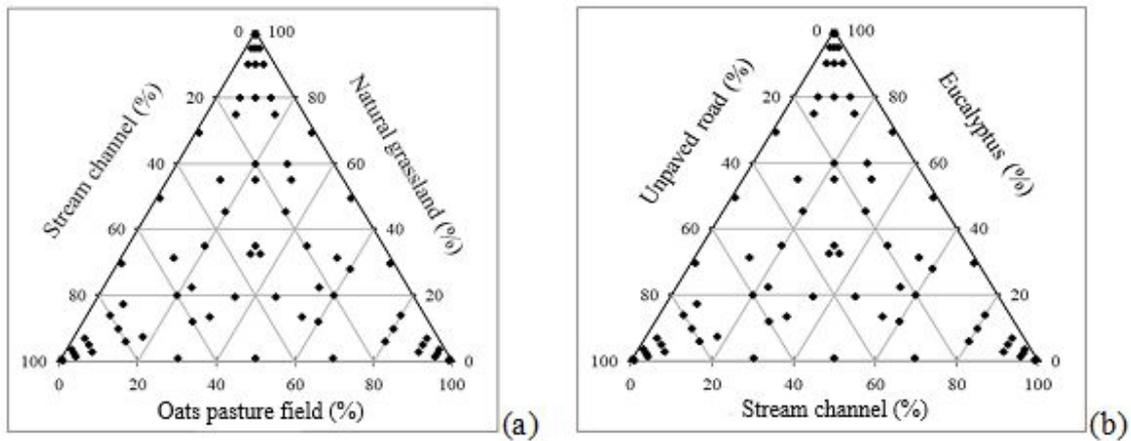
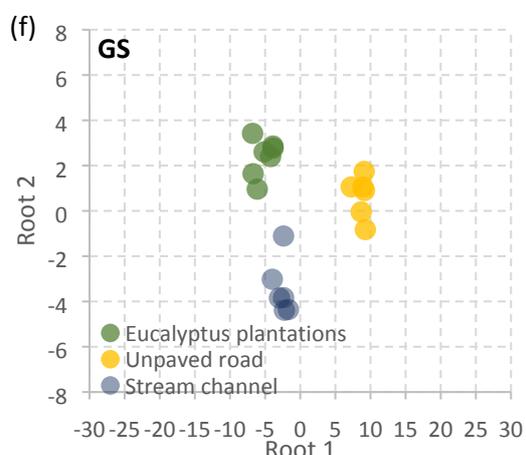
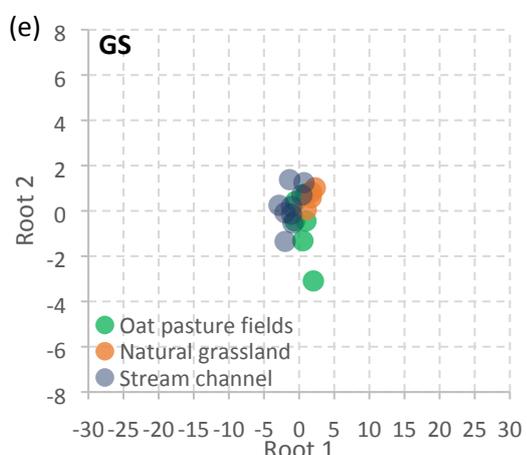
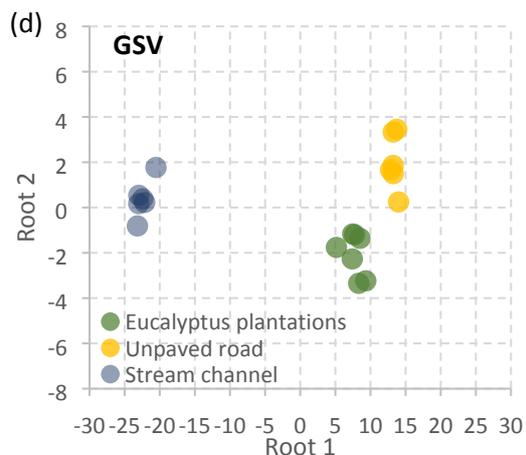
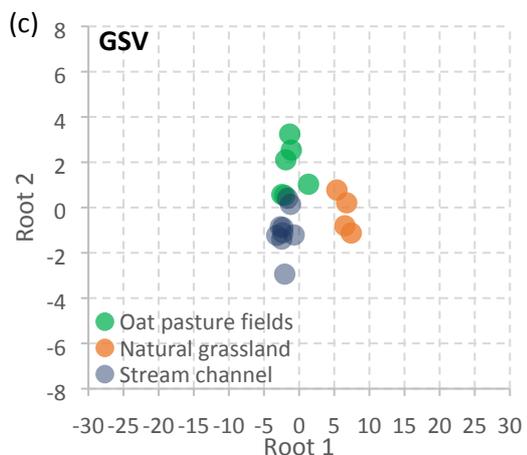
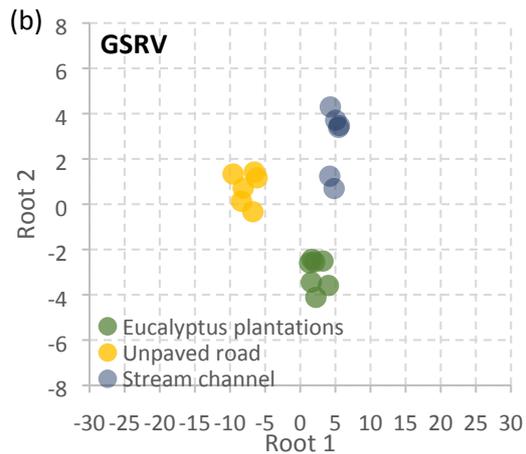
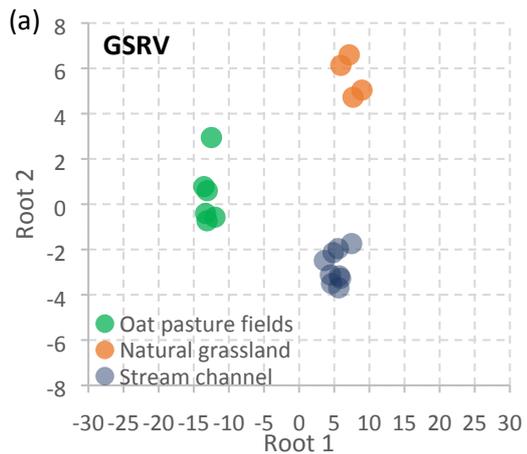


Fig. 2.

Ternary diagram with the location of the experimental mixtures prepared for the VIS-PLSR model calibration for the grassland catchment (a) and the eucalyptus catchment (b).



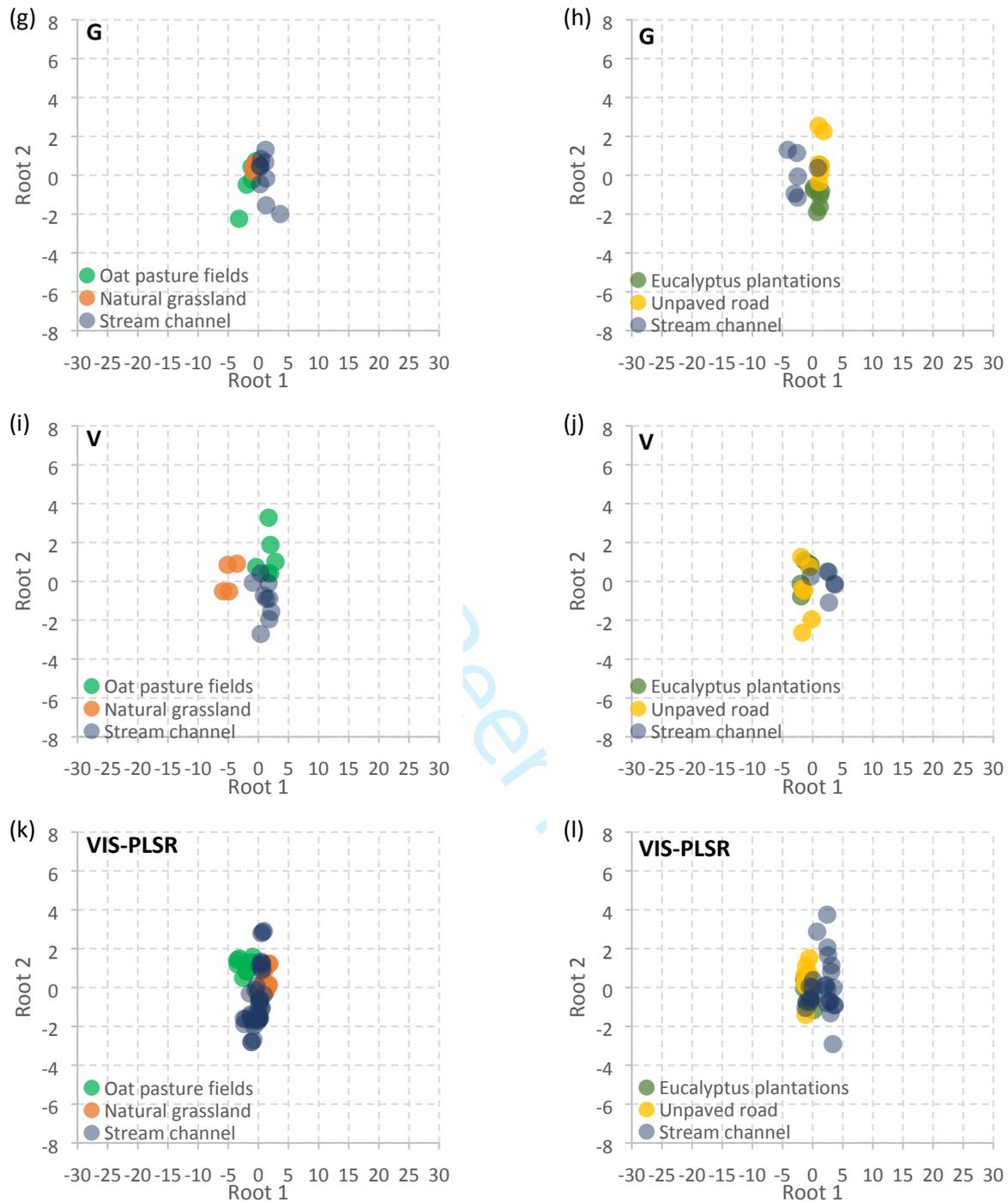


Fig. 3.

Scatterplot of the first and second discriminant functions derived from stepwise discriminant function analysis for GSRV in the grassland catchment (a) and the eucalyptus catchment (b), GSV (c, d), GS (e, f), G (g, h), V (i, j) and VIS-PLSR (k, l), respectively.

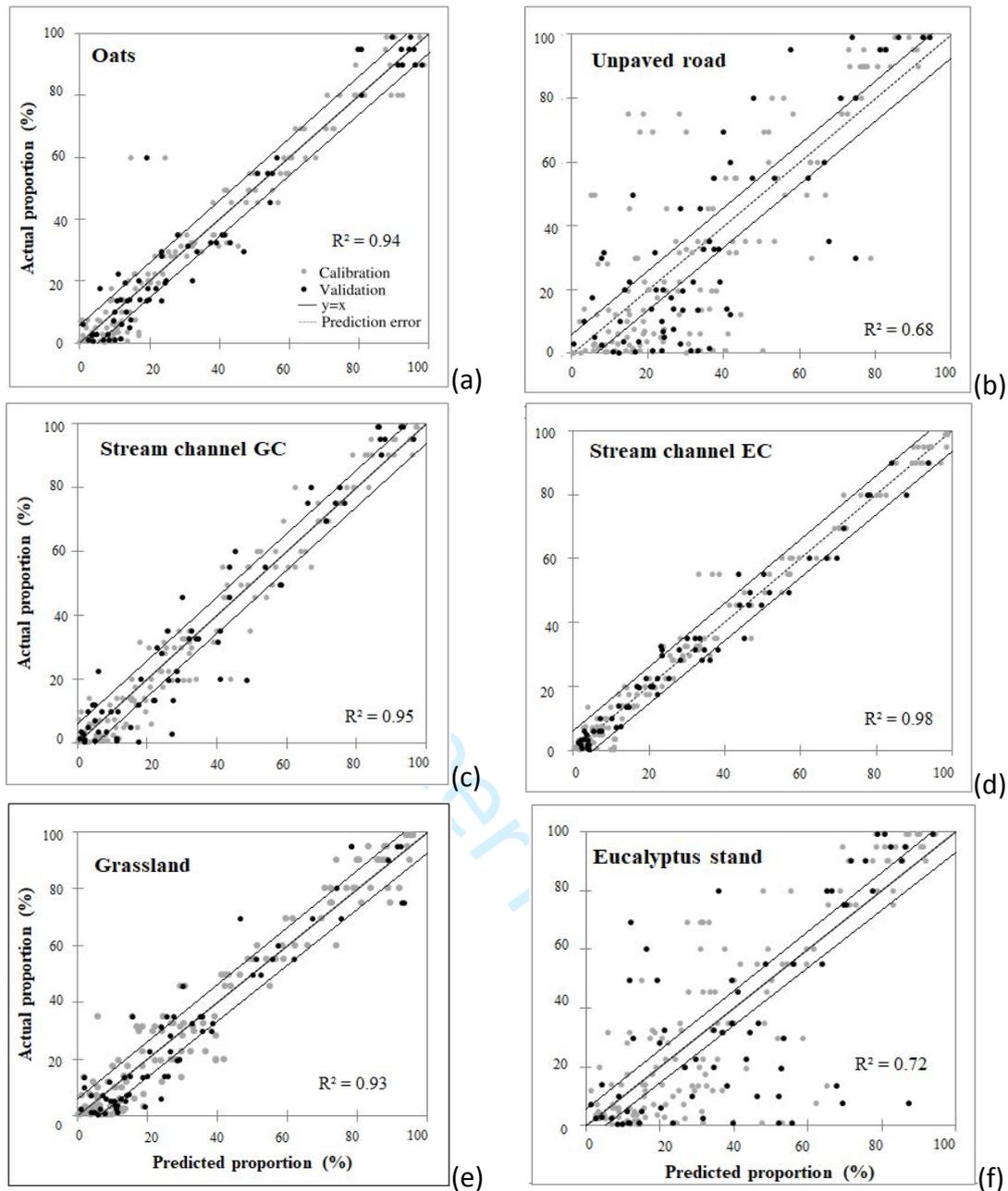


Fig. 4.

The relationship between actual and calculated source contributions predicted by VIS-PLSR models in the grassland catchment: oats pasture fields (a), stream channel GC (c) and natural grassland (e); and to the eucalyptus catchment: unpaved roads (b), stream channel EC (d) and eucalyptus stand (f). Dotted lines indicate the confidence limits (95%).

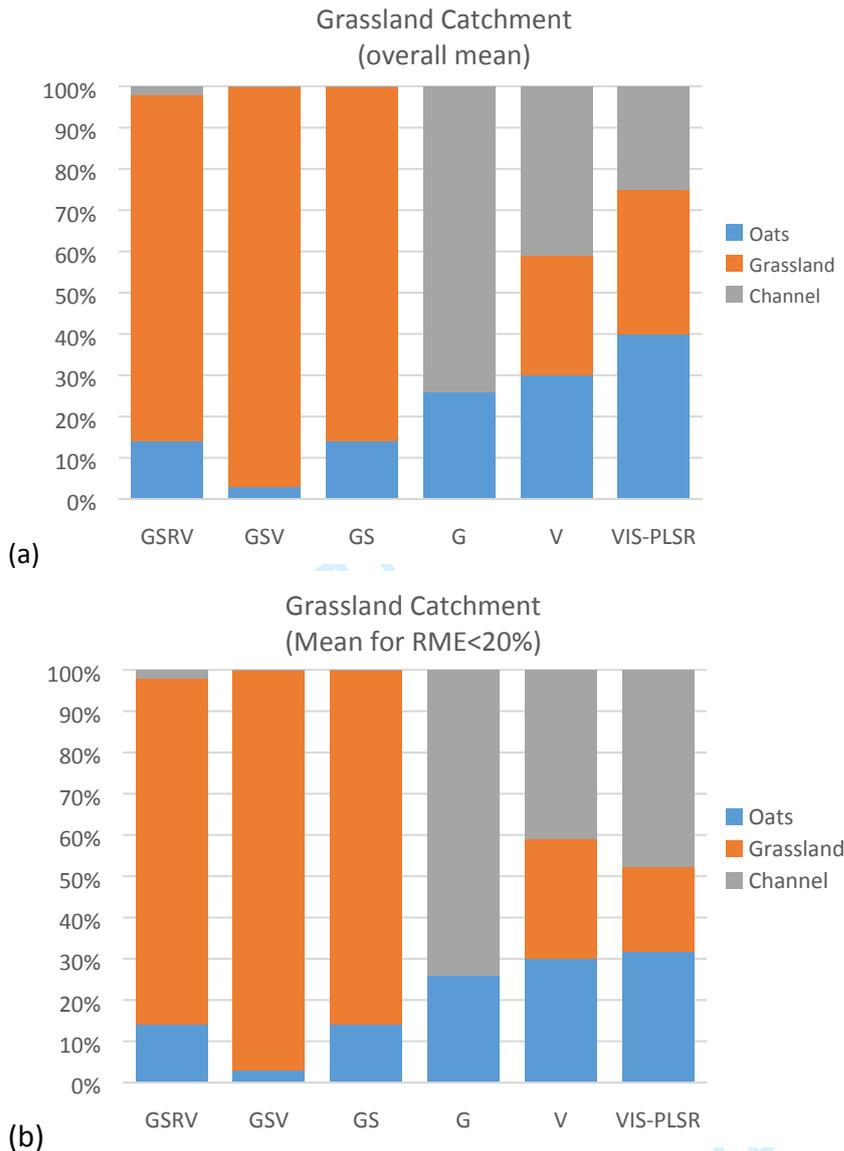
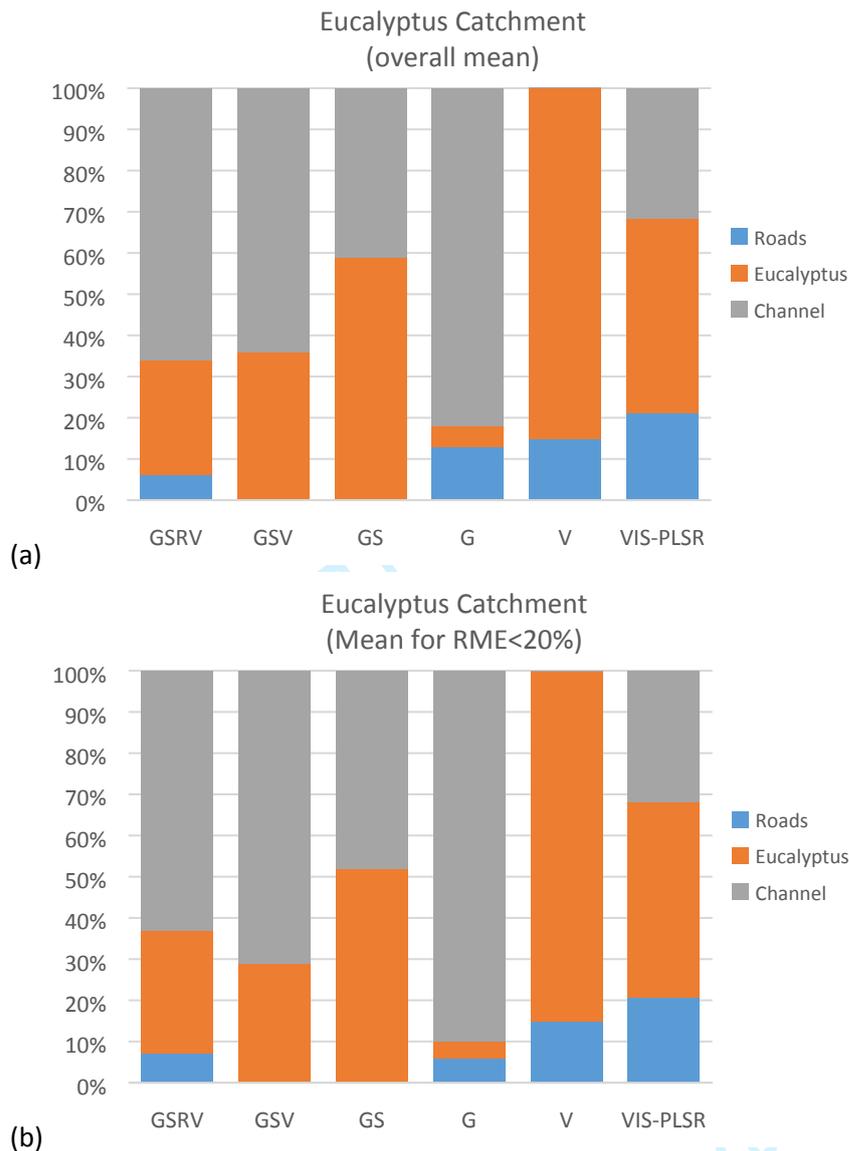


Fig. 5. General sediment source contributions in the grassland catchment as obtained with different combinations of conventional and alternative fingerprinting properties (a) and with values for relative mean error less than 20% (b).

**Fig. 6.**

General sediment source contributions in the eucalyptus catchment as obtained with different combinations of conventional and alternative fingerprinting properties (a) and with values for relative mean error less than 20% (b).