

2021-06

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<http://hdl.handle.net/10026.1/19139>

10.1007/s11368-021-02943-w

Journal of Soils and Sediments

Springer Science and Business Media LLC

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1 **Sediment source apportionment following wildfire in an upland commercial forest catchment,**

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10 **Acknowledgments**

11 We would like to dedicate this study to all people that lost their possessions during this wildfire that
12 affected a vast territory in central Chile. The authors also want to acknowledge the financial and logistic support
13 of Bioforest to perform the study in the Quivolgo catchment. Finally, we would like to acknowledge the valuable
14 comments from the reviewers that greatly improved the quality of this manuscript.

15

16 **Abstract**

17 **Purpose**

18 Wildfires can have major impacts on water scarcity and water quality linked to off-site transfer of
19 polluting ash and nutrients. Understanding sediment sources in burnt landscapes can help to develop mitigation
20 strategies, especially in catchments planted with introduced species that are prone to fire. We investigated
21 sediment sources activated by post-fire rainfall in a small-forested catchment that was impacted by a severe
22 wildfire. The aim was to use environmental radionuclides and elemental geochemistry as tracers to apportion
23 sediment sources within burnt plantation systems.

24 **Methods**

25 Surficial (0 – 2 cm) topsoil (n = 9), sub-surficial (2 – 4 cm) topsoil (*i.e.* below the burnt layer; n = 8)
26 samples from burnt hillslopes as well as forest roads (n = 5) and stream banks (n = 5) soil samples were taken
27 in the Quivolgo catchment, El Maule region, Chile. Sediment samples (n = 9) were collected from behind a v-
28 notched weir on three dates after the fire: May 2017, July 2017, and October 2017. Soil and sediment samples
29 were analysed by gamma spectrometry and Wave-length Dispersive X-Ray Fluorescence (WD-XRF) used to
30 obtain tracer properties. These were evaluated visually and statistically to identify potential non-conservative
31 tracers. Sediment apportionment was undertaken using the MixSIAR mixing model.

32 **Results**

33 The tracer selection procedure resulted in ten tracers being used for sediment apportionment. Tracer
34 suitability was based on: i) weak and non-significant linear relationship between tracer concentrations and
35 Specific Surface Area (SSA) and Soil Organic Matter (SOM), and ii) conservative behaviour supported by the
36 inclusion of sediment samples within source convex hull. Sediments from sub-surface layer (2 – 4 cm) were
37 the dominant source during the first two periods contributing 55 ± 11 and 78 ± 10 % respectively, whereas
38 roads contribution was only important in the last period (71 ± 14 %). Apportionment showed a shift in sediment
39 source (*i.e.* from forest roads to hillslopes) compared to a previous study in the same catchment before wildfire.
40 The main driver of erosion was attributed to overland flow convergence and consequent rill erosion across burnt
41 hillslopes.

42 **Conclusion**

43 The study demonstrated combined use of environmental radionuclides with elemental geochemistry
44 for sediment apportionment within burnt forest plantations and highlighted a switch in predominant source (*e.g.*
45 sub-surface burnt soil) activated by post-fire rainfall events. The findings in this research will help forest
46 companies to develop strategies to reduce off-site impacts of sediment release after wildfire in forest plantations.

47 **Keywords:** Sediment fingerprinting, wildfires, forest plantations, post-fire sediment sources,
48 MixSIAR.

49

50 **Declarations**

51 **Funding**

52 This research in the Quivolgo catchment was supported and financed by Bioforest, Arauco.

53 **Conflicts of interest**

54 The authors declare that there is no conflict of interest in this research and results presented in this
55 manuscript.

56 **Availability of data and material**

57 Supplementary information can be seen in the Electronic Supplementary Material of this article.

58 **Code availability**

59 Not Applicable

60 **Author's contribution**

61 Enrique Muñoz-Arcos, Claudio Bravo-Linares, Ramón Bustamante-Ortega and William H. Blake
62 contributed to the study conception and design. Material preparation, sampling, data collection and analysis
63 were performed by Enrique Muñoz-Arcos, Claudio Bravo-Linares, Luis Ovando-Fuentealba, Ramón
64 Bustamante-Ortega, Alejandra Castillo-Santana, Alicia Cuevas-Aedo and Alex Taylor. The first draft of the
65 manuscript was written by Enrique Muñoz-Arcos and all authors commented on previous versions of the
66 manuscript. All authors read and approved the final version of this manuscript.

67 **1 Introduction**

68 An estimated 7.20 billion ha of land were burnt between 2001 and 2018 at an average rate of more
69 than 400 million ha per year (FAO 2020). Wildfires pose a threat to life and property; particularly at the
70 urban/forest interface. In addition, wildfires can assert pressure on water resource availability and quality
71 (Martin 2016) linked to offsite transfer of polluting ash and nutrients (Blake et al. 2010). This underpins the
72 need for a process understanding of hillslope and channel connectivity in burnt landscapes (Wilkinson et al.
73 2009).

74 Wildfires have been recognised as an important cause of hydrological and geomorphological changes
75 over short and long timescales (Shakesby and Doerr 2006). Depending on its severity, wildfires can remove
76 some or all of the vegetation and litter cover, thereby affecting transpiration, interception, surface storage
77 capacity for rain and obstacles to overland flow (Shakesby and Doerr 2006). In parallel, remaining ashes can
78 have substantial effects on soil physical and chemical properties, nutrient cycles, carbon (C) cycle, hydrological
79 processes, water quality, microbial activity and plant growth (Bodi et al. 2014). Moreover, there are a number
80 of additional factors that can affect post-fire erosion such as slope, fire severity and vegetation status after fire
81 that can cause divergent responses in erosion rates and sediment yields (Shakesby and Doerr 2006). In the post-
82 fire context, rainfall ranks as the most important driver of runoff and erosion caused mainly by changes in soil
83 moisture, structure and infiltration which can accelerate sediment transport and deposition (Ice et al. 2004;
84 Moody et al. 2013; Shakesby et al. 2016). Hence, increased erosion rates and changes to runoff generation can
85 greatly augment the amount of sediment released, especially fine-grained particles, nutrients and other
86 constituents that can be delivered to streams and reservoirs affecting water quality in aquatic ecosystems (Ice
87 et al. 2004; Blake et al. 2010; Smith et al. 2011a; Martin 2016; Rust et al. 2019; Robinne et al. 2020).

88 Understanding soil and sediment process dynamics in fire-affected catchments can support
89 management efforts in facing postfire soil erosion effects and its downstream impacts. Specifically, the
90 assessment of sediment sources in downstream deposits can support targeted mitigation actions within primary
91 runoff contributing and sediment source areas to reduce the loss of ash and topsoil from hillslopes. Sediment
92 fingerprinting has emerged as a valuable tool in this regard and has been applied in a variety of landscapes and
93 settings (Walling 2013; Owens et al. 2016; Collins et al. 2017, 2020). However, its use in fire-affected

94 landscapes has been relatively limited to applications in Australia (Wilkinson et al. 2009; Smith et al. 2011b),
95 Canada (Owens et al. 2012; Stone et al. 2014) and Spain (Estrany et al. 2016; Garcia-Comendador et al. 2020).
96 While Fallout Radionuclides (FRNs) have been used to apportion postfire sources, other tracer properties such
97 as organic compounds (Oros et al. 2002), mineral magnetism (Blake et al. 2006a), colour parameters (Garcia-
98 Comendador et al. 2020) and geochemical elements (Blake et al. 2006b; Owens et al. 2006) have received less
99 attention (Smith et al. 2013). The latter, has shown greater potential for source discrimination between fire
100 severities due to wildfire modifications on surface soil geochemistry (Owens et al. 2006; Blake et al. 2006a;
101 Smith et al. 2013) wherein variability in fire severity has been shown to influence the concentration of trace
102 elements in ash and burnt topsoil (Blake et al. 2006b). Herein, important challenges with variability of
103 geochemical signatures between burnt soil and ash, as well as between burnt and unburnt catchments have been
104 identified (Smith et al. 2013; Stone et al. 2014).

105 Plantation forests cover about 131 million ha (3% of the global forest area) and 44% of this area is
106 composed mainly of introduced species. This is particularly relevant in South America where 97% of species
107 are non-native (FAO 2020). Chile has 2.3 million ha of forest plantations, where *P. radiata* represent 56%
108 followed by eucalyptus plantations (mainly *Eucalyptus globulus* and *Eucalyptus nitens*) covering 37% of the
109 planted area (INFOR 2020). In Chile, thousands of hectares of forests are lost every year due to the increase in
110 the number of wildfires during dry seasons enhanced by global change, rural depopulation and abandonment of
111 traditional land use practices (Carmona et al. 2012; Urrutia-Jalabert et al. 2018; Gonzalez et al. 2018; McWethy
112 et al. 2018; Gomez-Gonzalez et al. 2019). It has been estimated that fire occurrence in central and south-central
113 Chile has increased by around 50% in the last 40 years (González et al. 2011). Moreover, it has been described
114 that intensive transformation of native forests, shrublands and grasslands into massive exotic pyrophyte forest
115 plantations (*e.g.* *P. radiata* and *Eucalyptus* spp.) could have a major influence on the magnitude and recurrence
116 of wildfires by changing the fuel structure and flammability of the landscape (Taylor et al. 2017; McWethy et
117 al. 2018; de la Barrera et al. 2018; Mazzorana et al. 2019). Here, the potential of forest plantations to be affected
118 by wildfires requires consideration in terms of the off-site impacts that sediment release from burnt soils areas
119 can have on waterbodies, especially those that are used for drinking water supply (Smith et al. 2011a; Martin
120 2016).

121 The increase in the number and severity of wildfire events in Chile (McWethy et al. 2018; de la Barrera
122 et al. 2018), and the potential of forest plantations to be affected (Taylor et al. 2017), means there is an urgent
123 need to evaluate postfire catchment responses especially within catchments planted with exotic species and
124 hence a higher fire risk. In this context, the aims of this research were i) to assess dominant sediment sources
125 activated by post-fire rainfall events in a small-forested catchment impacted by a severe wildfire and, ii) to
126 explore the use of environmental radionuclides combined with elemental geochemistry as tracers to apportion
127 sediment sources within burnt plantation systems.

128 **2 Material and Methods**

129 **2.1 Catchment description**

130 Quivolgo catchment is located in “del Maule Region”, (Central Chile; Fig. 1). This region is
131 characterised by a temperate and semi-oceanic climate. The main soil type (WRB 1994) is leptosol and haplic
132 luvisol. The annual precipitation ranges are between 860 and 1130 mm (inter-annual coefficient of variation
133 ranges between 0.30 – 0.34%; data not published), and the period of maximum rainfall is from May to
134 September. El Maule region comprises 384,690 ha of land covered with forest plantations, being the third
135 administrative region of Chile with the largest planted area (17% of the total planted land in the country; INFOR,
136 2020). Quivolgo catchment has a total surface area of 40 ha and the stream length is around 0.8 km. The mean
137 altitude is 427 m a.s.l. and the mean slope is 44% (reaching a maximum slope of 121% in some areas). The
138 catchment flow regime is ephemeral. It depends mainly on precipitation, with maximum flow discharge during
139 winter. During summer the mean annual flow decreases markedly to around a 30 - 60%. The Quivolgo
140 catchment was planted with *Pinus radiata* in 2001 (65.1% of the total surface). Some areas were left intact with
141 native forest species (*Nothofagus glauca*, *Drimys winteri* and *Nothofagus oblicua*; 30.8% of the total catchment
142 area) to provide a riparian buffer strip as a biological corridor to protect watercourses from sediments released
143 during harvesting operations and, at the same time, to keep water physicochemical variables stabilised
144 (temperature, pH and turbidity). Additionally, forest roads covered 4.2% of total catchment area at the time of
145 sampling.

146 During the austral summer of 2017, the central-southern region of Chile suffered one of the biggest
147 forest fire on record. More than 500,000 ha of land were burnt between January 11th and February 18th, where

148 commercial forest plantations were one of the most affected ecosystems (Urrutia-Jalabert et al. 2018; de la
149 Barrera et al. 2018). Furthermore, “El Maule” was the most affected region with 287,027 ha of land being burnt
150 and a total amount of 145,000 ha of commercial forest plantations affected by the fire (de la Barrera et al. 2018).
151 During this wildfire, the entire Quivolgo catchment area was burnt.

152 **2.2 Source and sediment sampling**

153 After forest fire, four potential sediment sources were considered for sediment fingerprinting based on
154 previously reported impacts of wildfire on hillslope hydrological processes. Samples from forest hillslopes
155 comprised soil from surface (first 2 cm of soil surface) and from shallow sub-surface (underneath soil layer of
156 surface soil > 2 cm depth) as defined by the depth of charring. Additionally, surface soil areas deemed to be
157 prone to erosion due direct connection with the stream network (*e.g.* channel banks) or exposed to heavy
158 machinery operations within the catchment (*e.g.* roads) were sampled.

159 A total of 27 composite source samples were collected within the burnt catchment (3 months after the
160 fire). Forest hillslope composite samples (a mix of five ca. 100 g samples taken from the central point and edges
161 of a 10 × 10 m²) were collected to represent the soil surface (0 – 2 cm, n = 9) and the shallow sub-surface
162 material (2 – 4 cm, n = 8) (Fig. 2a). Channel bank soil samples (n = 5) were collected next to slopes at the edge
163 of the stream where active erosion was evident (Fig. 2b). Road samples (n = 5) were taken from verges, owing
164 to the central trackways being covered by coarse rock aggregate material, and at the opposite side of the forest
165 hillslopes to avoid the influence of sediment that had run-on from the upslope area (Fig. 2c). Source material
166 were collected using stainless steel spatulas, placed in double plastic bags, labelled accordingly and geo-
167 referenced by GPS.

168 Bulk sediment samples from the outlet of the Quivolgo catchment were collected from the weir pool,
169 which is a thin plate weir with a 90° shaped notch coupled with a datalogger pressure sensor (TrueBlue series
170 555). Weir and data logger installation followed World Meteorological Organisation (WMO) recommendations
171 (WMO 1994), and the sensor was configured to record data every 5 minutes (Fig. 2d). The weir was emptied
172 and washed after each sampling (to obtain the sediments corresponding to the specific studied periods). Weir
173 samples were collected in May 2017 (Weir 1), July 2017 (Weir 2) and October 2017 (Weir 3). Sediment samples
174 were placed in plastic jars, labelled accordingly, and stored for further analysis.

175 2.3 Sample preparation and analysis

176 The samples were oven-dried at 60°C in aluminium trays covered with Kraft paper (to avoid direct
177 contact of the samples with the tray) and sieved through a 2 mm mesh, obtaining approximately 100 g per
178 sample. Further, samples were sieved to 63 µm mesh. A 1.0 g sub sample was taken for particle size analysis
179 (PSA) and the remaining material (~10 g) milled for 20 minutes at 300 rpm in a Pulverisette 5 planetary ball
180 mill (Fritsch, Germany) using agate milling bowls and balls to achieve homogenous particle size for gamma
181 spectrometry and WD-X-Ray Fluorescence analyses. Additionally, Soil Organic Matter (% SOM) content was
182 estimated by Loss on Ignition (LOI). Approximately 5 g subsamples of < 2 mm soil fraction were weighed (in
183 previously cleaned and dried crucibles) and then ignited at 475°C for 3 h in a muffle furnace.

184 Particle size analysis was performed on the < 63 µm sample material. Prior to particle size analysis by
185 laser granulometry, wet oxidation was carried out to remove organic matter by using 6% H₂O₂ in a heated water
186 bath (all samples were prepared in triplicate). Particle size analysis was undertaken using a Malvern Mastersizer
187 2000 (Malvern, UK) with Hydro-G in compliance with ISO 13320. Optimal particle dispersion was achieved
188 using sodium hexametaphosphate (0.2%) in solution and 90 seconds of ultrasonication during analysis. Pump
189 and stirrer speeds were 2250 and 800 rpm respectively. Each sub-sample was analysed for 30 seconds using red
190 laser, and this was repeated to produce five data sets for each sub-sample. The Software uses an enhanced
191 general model for non-spherical particles with an assumed refractive index of 1.53 and initial light adsorption
192 value of 0.01.

193 Fallout (¹³⁷Cs and ²¹⁰Pb_{ex}) and geogenic (²²⁶Ra, ²³²Th, ²³⁸U and ⁴⁰K) radionuclides were analysed in <
194 63 µm soil fraction following the methodology described in detail by Appleby (2001). All samples were packed
195 and sealed in gas tight containers. Activity concentrations of the target radionuclides were measured in plastic
196 vials using an ORTEC well detector system (GWL-170-15-S, an N-type detector). The HPGe gamma
197 spectrometry system was built to ultra-low background specification for ²¹⁰Pb detection for gamma
198 spectrometry. The instrument was calibrated and operated according to the standard operating procedures
199 contained within the instrument record manual. In brief, the instrument was calibrated using soil material spiked
200 with a certified, traceable mixed radioactive standard 80717-669 (supplied by Eckert & Ziegler Analytics,
201 Georgia, USA). All calibration relationships were derived using ORTEC GammaVision software and verified

202 by inter-laboratory comparison tests with materials supplied by the International Atomic Energy Agency
203 (IAEA), in particular the world-wide proficiency test using radioactive moss soil (IAEA-CU-2009-03). The
204 isotopes ^{210}Pb , ^{214}Pb and ^{137}Cs were determined by their gamma emissions at 46.52, 295.34 (& 351.99) and
205 661.6 keV, respectively. Total ^{210}Pb was measured and its unsupported component calculated by the subtraction
206 of the ^{226}Ra activity, which in turn was measured by the gamma emissions of ^{214}Pb . To produce activity
207 concentrations, samples were counted for at least 100,000 seconds. All activity concentration data were decay
208 corrected to the date of sampling. Owing to low activity concentrations and sample mass, 1-sigma counting
209 uncertainties for ^{137}Cs ranged 12 to 30% and for ^{210}Pb ranged 5 to 18% noting that uncertainties propagated
210 through the mixing model are based on the mean and standard deviation of source group distributions, i.e. the
211 environmental variability and not counting statistics.

212 Sample analysis for minor and major elements (Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, Cr, Mn, Fe, Co,
213 Ni, Cu, Zn, Ga, Br, Rb, Sr, Y, Zr, Nb, Ba, Ce and Pb) was undertaken by Wavelength Dispersive X-Ray
214 Fluorescence (WD-XRF) spectrometry. The milled sample material was mixed with a polypropylene wax
215 binding agent at a ratio of 1:4 (binder:sample), prior to being pressed into sample pellets. Samples were pressed
216 in 40 mm diameter aluminium sample cups under 150 kN of pressure using a Herzog TP20 manual press. Pellets
217 were analysed for a full suite of elements under vacuum using an Axios max (PANalytical, Netherlands). The
218 instrument operated a Rh target X-ray tube at 4kW power with sequential detection of elements undertaken
219 with Ar gas flow and scintillation detectors. Measurement conditions were optimised using PANalytical SuperQ
220 software and the semi-quantitative Omnia analysis application. Instrument drift was assessed following
221 laboratory quality control procedures using a multi element glass sample (glass monitor C3, Breitlander).
222 Particle size analysis, FRNs and geogenic radionuclides and XRF analyses were performed at the Consolidated
223 Radioisotope Facility (CoRiF) at University of Plymouth, UK.

224 **2.4 Statistical analysis and source apportionment**

225 Source and sediment samples values distribution were visualised by using boxplots. Specific
226 comparisons between source groups were statistically tested via a two-sample t-test or two-sample Wilcoxon
227 rank sum test (also known as Mann-Whitney U test) after checking for test assumptions (*e.g.* normality and
228 homogeneity of variance).

229 Tracer selection for source apportionment is a key aspect of the sediment fingerprinting methodology.
230 It has been demonstrated that different tracer selection procedures can lead to substantial variability to the
231 mixing model outputs (Palazon and Navas 2017; Smith et al. 2018). Here a tracer selection procedure was
232 adopted based on the exclusion of apparently non-conservative tracers developed by Smith et al. (2018).
233 Exploratory analysis of sources and mixtures data was undertaken to observe the general geochemical character
234 of the samples. In this case, Principal Component Analysis (PCA) was performed to evaluate source and mixture
235 groupings according to geochemical properties using *FactoMineR* and *factoextra* packages from R (Lê et al.
236 2008; Kassambara and Fabian 2019). Soil and sediment particle size, (via Specific Surface Area, SSA) and Soil
237 Organic Matter (SOM) were tested for significant differences at 95% level of confidence between source groups
238 and between soil and sediment samples after checking tests assumptions (*e.g.* normality and homogeneity of
239 variance). When normality and homogeneity of variance assumptions were met a two-sample t-test (source and
240 mixture groups) and ANOVA (between source groups) were applied, otherwise a Wilcoxon rank sum test or
241 Kruskal-Wallis test was carried out. Moreover, the potential non-conservative behaviour in terms of particles
242 sorting effects and/or organic enrichment of tracer concentrations was examined by computing Pearson's
243 correlation with geochemical properties. When assumptions for both Pearson correlation and linear regression
244 were not met (*e.g.* variables normally distributed and normality of the errors, respectively) a Box-Cox power
245 transformation on tracer values was performed using MASS package in R (Venables and Ripley 2002). When
246 significant correlations ($p < 0.05$) between tracer concentrations and the SSA or SOM was observed, the 95%
247 prediction interval (PI) was computed by simple linear regression to see if sediment mixtures values fell within
248 the PI. In this sense, tracers that showed significant correlation with SSA and SOM and where the sediment
249 values fell outside the PI area were discarded from further mixing model analyses. Therefore, tracer properties
250 that remained after correlation/regression analyses were compared between source and mixture materials
251 through visualisation of source minimum bounding polygons (convex hulls). Where mixtures fell outside the
252 source polygon an assumption of non-conservative behaviour was made. Consequently, tracers were excluded
253 from further analysis when mixtures values lay outside source polygons in most tracer combinations. Finally,
254 source group normality of the remaining tracers was evaluated using Shapiro-Wilks test before the unmixing
255 process.

256 Sediment source apportionment was carried out using the MixSIAR mixing model package (Stock et
257 al. 2018). MixSIAR is a Bayesian framework that uses tracer data to estimate source probability distributions
258 to a mixture. The fundamental mixing equation followed by MixSIAR and, basically any mixing system, is as
259 follow:

$$260 \quad Y_j = \sum_k p_k \mu_{jk}^s, \text{ Eq. 1.}$$

261 where the mixture tracer value, Y_j for each j tracer is equal to the sum of the k source tracer means,
262 μ_{jk}^s , multiplied by their proportional contribution to the mixture, p_k (Stock et al. 2018). The model was run with
263 uninformative priors, using weir pool sampling dates as fixed effects. The error structure was set with the
264 residual error which accounts for unknown sources of variability in weir pool sediment samples apportionment
265 (Parnell et al. 2010). Markov Chain Monte Carlo (MCMC) parameters were set as follow: chain length =
266 3.000.000; burn-in = 1.500.000; thin = 500 and number of chains = 3. Convergence of the model was evaluated
267 using the Gelman-Rubin diagnostic, in which case all variables were below 1.01 (Stock and Semmens 2018).
268 All results are provided as the mean and standard deviations from the posterior distributions obtained by
269 MixSIAR. The matrix plot of correlation between global posterior source probability distributions was used to
270 evaluate the quality of source discrimination. Finally, source apportionment results were compared with results
271 obtained using tracers that passed the classical tracer selection procedure (*e.g.* range test, Kruskal-Wallis test
272 and Discrimination Function Analysis, DFA) first introduced by Collins and Walling (2002). The above
273 described methodology was applied via FingerPro package in R (Lizaga et al. 2020). All statistics and unmixing
274 process were done in R (R Core Team 2018) via R-Studio interface (RStudio Team 2015).

275 **3 Results**

276 **3.1 Catchment hydrological response to wildfire**

277 Accumulated monthly precipitation in the Quivolgo catchment is focussed on winter months (from
278 May to September) accounting for between 200 and 250 mm (Fig. 3). During the study period, pluviometric
279 conditions were similar except for 2016, where precipitation decreased by 46% compared to the mean rainfall
280 during 2014 and 2015. Also, precipitation recorded in 2017 was notably higher compared to previous years
281 (Fig. 3). Flow discharge at the weir was sensitive to rainfall during each season and the minimum flow was

282 registered before the wildfire event. After the fire, discharge ($\text{m}^3 \text{s}^{-1}$) increased almost two-fold compared to
283 pre-fire streamflow conditions (Fig. 3).

284 3.2 Radionuclide activity and elemental concentration of tracers after wildfire

285 An increase of 1.5 and 4.5 times in the activity concentration of ^{137}Cs and $^{210}\text{Pb}_{\text{ex}}$, respectively, was
286 observed in burnt materials of surface layers compared to soil surface values from hillslope samples of a
287 previous study in the same catchment before wildfire (Table 1; data not published). In this sense, ^{137}Cs and
288 $^{210}\text{Pb}_{\text{ex}}$ mean activity concentration (Bq kg^{-1}) in surface soil samples of the burnt catchment were significantly
289 higher than surface soil samples before fire (^{137}Cs = one-sided two-sample t-test, $t(19) = 3.2834$, $p < 0.01$; ^{210}Pb
290 = Welch one-sided t-test, $t(7.58) = 5.57$, $p < 0.0001$).

291 Moreover, $^{210}\text{Pb}_{\text{ex}}$ sub-surface values were significantly lower than surface values after wildfire (Welch
292 one-sided t-test, $t(9.07) = -3.217$, $p < 0.01$; see $^{210}\text{Pb}_{\text{ex}}$ values distribution at Fig. 4) whereas ^{137}Cs did not show
293 significant differences between these two sources (one-sided two-sample t-test, $t(14) = -0.543$, $p > 0.05$). It is
294 noteworthy that $^{210}\text{Pb}_{\text{ex}}$ mean activity for surface samples ($77 \pm 30 \text{ Bq kg}^{-1}$) was almost two times higher than
295 sub-surface samples ($41 \pm 12 \text{ Bq kg}^{-1}$; see Table 1). Furthermore, ^{137}Cs values for channel bank samples after
296 wildfire were not statistically different to surface samples (Welch two-sided t-test, $t(7.01) = -0.20$, $p > 0.05$),
297 where mean activities were similar: $5.8 \pm 0.0 \text{ Bq kg}^{-1}$ and $5.9 \pm 2.1 \text{ Bq kg}^{-1}$, respectively (see Supplementary
298 table 1). However, differences in $^{210}\text{Pb}_{\text{ex}}$ values between these two sources were significant (Welch two-sided
299 t-test, $t(8.21) = -3.43$, $p < 0.05$). Additionally, significant differences between channel banks and sub-surface
300 samples were not observed for either tracers (^{137}Cs : Welch two-sided t-test, $t(7.02) = 0.616$, $p > 0.05$; $^{210}\text{Pb}_{\text{ex}}$:
301 two-sided two-sample t-test, $t(12) = -0.2411$, $p > 0.05$).

302 Elemental concentrations of Na, Mg, Al, P, K, Ca, Mn, Fe, Co, Zn, Sr and Ba in surface material were
303 significantly higher than sub-surface (Na, Mg, Al, P, K, Mn, Fe, Co, Sr and Ba: one-sided two-sample t-test,
304 $t(15) < -1.88$, $p < 0.05$; Ca and Zn: one-sided two-sample Wilcoxon rank sum test, $W > 6$, $p < 0.05$; see for
305 example, Mg, K and Mn values distribution at Fig. 4). The opposite was true for Cl which showed a mean
306 concentration value for surface soil samples significantly lower than sub-surface (one-sided two-sample t-test,
307 $t(15) = 2.25$, $p < 0.05$). Additionally, Na, Mg, P, K, Zn, Rb, Sr and Pb showed significant differences between
308 channel bank and sub-surface sample values (Na, K, Zn, Rb, Sr and Pb: two-sided two-sample t-test, $t(12) >$

309 2.4, $p < 0.05$; Mg: Welch two-sided t-test, $t(10.2) = 5.53$, $p < 0.001$; P: Wilcoxon rank sum test, $W = 45$, $p <$
310 0.001), while Al, Si, S, Cl, Ca, Ti, Cr, Mn, Fe, Co, Ni, Cu, Ga, Y, Zr, Nb, Ba and Ce did not present significant
311 differences between these source values (Si, S, Cl, Ti, Cr, Mn, Ni and Zr: two-sided two-sample t-test, $t(12) <$
312 2.07 , $p > 0.05$; Fe, Co and Y: Welch two-sided t-test, $t(\text{Fe}: 4.4; \text{Co}: 3.4 \text{ and } \text{Y}: 10.2) = 1.06, 1.49 \text{ and } 0.52$ for
313 Fe, Co and Y, respectively, $p > 0.05$; Al, Ca, Cu, Ga and Nb: Wilcoxon rank sum test, $W < 21$, $p > 0.05$; see
314 for example, Ti, Mn and Zr values distribution at Fig. 4).

315 **3.3 Tracer selection procedure**

316 The SSA of sampled soil materials from the Quivolgo catchment presented significant differences in
317 their distribution (Kruskal-Wallis test, $H = 19.6$, $p < 0.001$; see SSA distribution at Fig. 5). For example, sub-
318 surface materials presented SSA values significantly lower than those of surface (Wilcoxon rank sum test, $W =$
319 10 , $p < 0.01$). Additionally, RO samples showed the lowest mean of SSA among sources ($0.17 \pm 0.03 \text{ m}^2 \text{ g}^{-1}$;
320 Supplementary Table 1). Therefore, source and sediment sample groups showed significant differences in SSA
321 values (Wilcoxon rank sum test, $W = 60$, $p < 0.05$).

322 In contrast to SSA, % SOM did not present significant differences between source and sediment
323 samples (two-sided two-sample t-test, $t(34) = -0.57$, $p > 0.05$). Differences between source samples, however,
324 were significant (one-way ANOVA, $F(3, 23) = 12.11$, $p < 0.001$; see % SOM distribution at Fig. 5).
325 Additionally, roads samples showed the lowest mean organic content (7.0 ± 2.2 , see Supplementary Table 1)
326 whereas the highest mean in soil organic matter was observed in sub-surface samples (16.2 ± 3.2 , see Table 1
327 and Supplementary Table 1). Furthermore, % SOM between surface and channel banks were similar ($12.2 \pm$
328 2.9 and 13.1 ± 2.0 , respectively. See Supplementary table 1).

329 Due to the above differences between source groups and sediment mixtures, SOM and SSA linear
330 relationships with tracer properties were evaluated. In the present study, tracers that showed significant
331 correlation ($p < 0.05$) between SSA and elemental concentrations were ^{226}Ra , Si, P, S, Cl, Ca, Mn, Cu, Zn, Sr
332 and Ba (see Supplementary Table 2). Nevertheless, the 95% linear regression PI (Fig. 6) showed that only S,
333 Cl and Ca mixtures values plotted outside PI and hence were considered to be exhibiting a non-conservative
334 behaviour relative to sediment samples (Smith et al. 2018). These three tracers were consequently removed
335 from further analysis.

336 Soil organic matter presented significant correlations ($p < 0.05$) with Si, S, Cl, K, Ca and Nb
337 (Supplementary Table 2) but only S and Cl mixture values fitted outside the PI of the combined sources (Fig.
338 7). It is noteworthy that these tracers showed a highly significant relationship with SOM ($p < 0.001$) and the
339 strong positive association between S and Cl with SOM ($r = 0.8$, see Supplementary Table 2) suggests an
340 enrichment of these tracers with increased soil organic content. Therefore, S and Cl interaction with SOM
341 demonstrated their inability to be used as sediment tracers in catchments affected by wildfire and consequently,
342 they were removed for sediment apportionment calculations.

343 The source convex hull of the remaining tracers (28) showed that for ten tracers sediment mixtures
344 plotted inside the bounding polygon in most of tracer combinations (half number of tracers + 1) (see
345 Supplementary Table 3). Following this process, $^{210}\text{Pb}_{\text{ex}}$, Mg, Si, K, Ti, Mn, Y, Zr, Nb and Pb were selected as
346 good performers and they were evaluated to be included in subsequent source apportionment calculations. The
347 remaining tracers (*e.g.* ^{226}Ra , ^{232}Th , ^{238}U , ^{40}K , Na, Al, P, Cr, Fe, Co, Ni, Cu, Ga, Rb, Sr, Ba and Ce) were
348 excluded. In addition, we noticed that extreme outliers affected the source polygon area considerably which
349 could lead to the potentially erroneous inclusion of sediment samples within the polygon boundary. When
350 outliers were one or more orders of magnitude greater than the mean source tracer values distribution, they were
351 removed only from the convex hull computation. An example of convex hull polygon criteria can be found in
352 the Supplementary Figure 1.

353 In addition, the normality of source group tracers was evaluated. Tracers that fitted normal distribution
354 were Mg, Si, K, Ti, Y, Zr and Nb ($W > 0.93$, $p > 0.05$) while $^{210}\text{Pb}_{\text{ex}}$, Mn and Pb did not ($W < 0.92$, $p < 0.05$).
355 Nevertheless, these tracers were kept in further steps owing to relaxation of the source normality assumption in
356 MixSIAR, supported by Smith et al. (2018) who showed that the removal of tracers on the basis of non-
357 normality led to a decrease in mixing model accuracy using synthetic mixtures.

358 Additionally, Principal Component Analysis (PCA) of soil and sediment samples was undertaken after
359 the tracer selection procedure to explore the geochemical behaviour of source observations (Fig. 8). PCA of
360 remaining tracers led to a high dispersion of source observations (Fig. 8), which can be seen in the low value
361 of explained variance by the first principal component (33% of explained variance). Confidence source ellipses
362 at 95% level showed a clear distinction between the surface and sub-surface sample groupings mainly explained

363 by the influence of $^{210}\text{Pb}_{\text{ex}}$, Mn and Pb tracer values. Also, separation between sub-surface and channel banks
364 groupings is noteworthy. However, overlap between road samples and other source samples confidence ellipses
365 were observed which evidence low discriminatory capacity of tracers between some materials. Additionally,
366 sediment sample confidence ellipses of Weir 1 and Weir 2 plotted inside of sub-surface ellipse while Weir 3
367 plotted inside the road source ellipse.

368 **3.4 Post-fire sediment source contributions and tracer selection method comparison**

369 According to mixing model outputs, the main sources of sediment that accumulated in the weir after
370 the wildfire (from December 2016 until May 2017) were shallow sub-surface soil followed by forest roads *i.e.*
371 55 ± 11 and $30 \pm 11\%$, respectively (summary statistics can be found in Supplementary Table 4). During June
372 and July 2017, weir sediment samples had sub-surface contributions in excess of $78 \pm 10\%$. Between August
373 and October 2017, roads were the dominant sediment source contributing $71 \pm 14\%$ with a notably lower
374 proportion of sub-surface ($19 \pm 10\%$). Surface soil and channel banks apportionment were negligible during the
375 whole study period ($< 10\%$, Fig. 9). In addition, important correlation was observed between roads and sub-
376 surface global posterior distributions ($r = -0.71$) whereas association between remaining source posterior
377 distributions were weak ($r < -0.36$, see Supplementary Figure 2).

378 In comparison, the classical tracer selection procedure resulted in five tracers suitable for
379 apportionment calculations, which were selected on the basis of maximised discrimination among sources.
380 Tracers that passed range test, Kruskal-Wallis test and DFA test were ^{226}Ra , P, Cl, K and Sr. It is important to
381 highlight that K was the only tracer that was selected in both tracer selection methods. In addition, Cl was
382 included in this set of tracers even when it was considered as non-conservative since it had strong association
383 with SSA and SOM and plotted sediment samples outside PI ($r = -0.6$, $r = 0.8$ and Fig. 6, Fig. 7, respectively;
384 also see Supplementary Table 2).

385 Sediment source apportionment with classical tracer selection did not show notable difference in
386 results from the more holistic approach in terms of dominant sources. The same dominant sediment sources *i.e.*
387 sub-surface in the first two periods and roads in the last period were identified (Fig. 10) although differences in
388 the estimated % of contribution were seen. For example, at Weir 2 (June – July), sub-surface contribution
389 accounted for 40 ± 14 (see Supplementary table 5) and, at Weir 3 (August – October), roads contribution was

390 56 ± 15 . In both cases, classical statistics-driven tracer selection procedure resulted in the reduction of the
391 apportionment from these sources compared to the more holistic and inclusive tracer selection applied in this
392 study. Conversely, channel banks contribution increased almost 15% whereas surface soil apportionment was
393 negligible at these three periods. Furthermore, it is noteworthy that the classic tracer selection procedure resulted
394 in wider posterior distributions for the main sources in the periods evaluated compared with the method applied
395 in this study (see Fig. 10). For instance, Credible Intervals (CIs) of sub-surface probability distribution in Weir
396 1 and Weir 2 ranged between 8 – 66% and 10 – 70% respectively compared to 32 – 77 and 55 – 95 for Weir 1
397 and 2, respectively. The same is true for road sources at Weir 3, where posterior probability CI ranged from 24
398 to 84 with classic tracer selection compared to the selection method applied in this study, whose CIs ranged
399 from 41 – 83.

400 **4 Discussion**

401 **4.1 Tracer distribution values in soil samples**

402 The increase of FRNs activity in surface of burnt soil compared with surface soil values before wildfire
403 was in line with other studies which have found that wildfires enhance FRNs concentration due to the
404 combustion of organic matter (*e.g.* leaf litter and humic materials) (Johansen et al. 2003; Wilkinson et al. 2009;
405 Owens et al. 2012; Estrany et al. 2016). Fallout $^{210}\text{Pb}_{\text{ex}}$ would have been deposited on pine needles duff layer
406 and on native trees canopy prior to the fire and when they were reduced to ash, the $^{210}\text{Pb}_{\text{ex}}$ was therefore
407 concentrated on the surface (Owens et al. 2012; Smith et al. 2013). On the other hand, large spatial variability
408 was observed for FRNs in surficial samples after forest fire (see ^{137}Cs and $^{210}\text{Pb}_{\text{ex}}$ boxplots at Fig. 4). In surface
409 material, the within-source variability of both ^{137}Cs and $^{210}\text{Pb}_{\text{ex}}$, may be due to the following reasons: 1) Spatial
410 variation in the intensity of the wildfire and the degree of combustion of organic matter, 2) variations in ash
411 deposition and 3) the extent of ash infiltration into surface soil layers (Owens et al. 2012; Estrany et al. 2016).
412 The latter is attributed to a low intensity rain that occurred a few days before sampling that may have caused
413 the incorporation of part of the ash deposits into the surface soil layer through infiltration leading to sealing of
414 pores and macropores.

415 The similarity in terms of FRN composition between channel banks and sub-surface samples can be
416 attributed to the sample depth in the soil profile, where sub-surface soil corresponds to the 2 – 4 cm soil layer

417 and stream channels incisions are relatively shallow (as can be seen at Fig. 2-b). Furthermore, the discrepancy
418 between surface and sub-surface statistical differences in ^{137}Cs and $^{210}\text{Pb}_{\text{ex}}$ values can be associated to the
419 tendency for $^{210}\text{Pb}_{\text{ex}}$ to present higher concentration in the ash due to its continuous production, residence in the
420 surface litter and delivery; whereas ^{137}Cs tends to have greater association with deeper soil material following
421 the cessation of ^{137}Cs fallout (Blake et al. 2009b; Owens et al. 2012; Smith et al. 2013). Additionally, the absence
422 of ^{137}Cs activity in forest roads samples is a consequence of surface soil being removed during road
423 construction; in contrast to $^{210}\text{Pb}_{\text{ex}}$, which has been deposited continuously from the atmosphere.

424 The increase in concentration of Na, Mg, Al, P, K, Ca, Mn, Fe, Co, Zn, Sr and Ba in surface soil
425 samples can be attributed to the mineralization of soil organic matter with further inputs from canopy due to
426 combustion (Certini 2005; Owens et al. 2006; Smith et al. 2013). On the contrary, decrease in Cl concentration
427 may be due to volatilization from surface layers associated to soil heating or leaching to sub-surface layers
428 (Owens et al. 2006; Smith et al. 2013). On the other hand, the similarities in element composition between sub-
429 surface and channel banks (*e.g.* Al, Si, S, Cl, Ca, Ti, Cr, Mn, Fe, Co, Ni, Cu, Ga, Y, Zr, Nb, Ba and Ce) support
430 that channel banks presents sub-surface material.

431 **4.2 Tracer selection**

432 It is important to give consideration to the potential influence of particle size selectivity in erosion
433 processes *e.g.* where fine particles have greater potential to be transported and may become geochemically
434 enriched in certain properties (Smith and Blake 2014; Laceby et al. 2017). SSA can control some elemental
435 concentration where interactions are due to adsorption or where there is a compositional control on size fraction
436 *e.g.* silica and sand particles. While relationships vary in complexity, it has been shown that below $1.0 \text{ m}^2 \text{ g}^{-1}$
437 the relationship between these interactions is approximately linear (Smith and Blake 2014; Laceby et al. 2017).
438 The impacts of wildfire on hydrological and morphological processes are commonly associated with a change
439 in soil aggregate stability and enhanced or decreased water repellency; which could have implications for
440 infiltration, overland flow, and rainsplash detachment of soil particles (Shakesby and Doerr 2006; Blake et al.
441 2007). Furthermore, it has been demonstrated that soil burning can significantly reduce the clay content with
442 corresponding increase in sand-sized particles (Dyrness and Youngberg 1957) implying aggregation of fine
443 particles into robust coarser composite particles. Conversely, some other studies have shown no significant

444 change in soil aggregate size distribution following a burning episode (Garcia-Corona et al. 2004). In this sense,
445 surface materials presented SSA values significantly higher than those of sub-surface (see Fig. 5) indicating
446 that sub-surface soil was coarser than surface soil whose structure and aggregates stability could have been
447 affected by wildfire (Blake et al. 2009a), mainly by the influence of ash particles that may have been
448 incorporated into the soil surface. Also, it is important to highlight that roads presented the lowest SSA mean,
449 indicating that road material was composed of coarser particles than other soils within the catchment.

450 Burning will substantially modify SOM properties (Ice et al. 2004; Certini 2005) thereby affecting
451 tracer concentration properties (*e.g.* enrichment of trace elements in ash). In some prior studies, researchers
452 have incorporated correction factors to deal with this issue, while others authors have decided to leave
453 concentration properties without modification to avoid over-correction (Smith et al. 2011b). Some other studies
454 have raised concern about using correction factors (Koiter et al. 2018), while Smith and Blake (2014) found
455 that applying combined SSA and total organic content correction completely altered source tracer content (*e.g.*
456 $^{210}\text{Pb}_{\text{ex}}$ activity). In the Quivolgo catchment, surface SOM decreased two-fold compared to unburnt surface
457 SOM (see Table 1), and it was also lower compared to sub-surface samples after wildfire (see Fig. 5), suggesting
458 a depletion in surface SOM caused by burning. Wildfire should not affect or otherwise have a minor impact on
459 road material organic content. Therefore, low values are attributed to the intrinsic composition of forest road
460 cover (mainly influenced by the input of fallen pine needles and branches).

461 The tracers that passed the selection process were: $^{210}\text{Pb}_{\text{ex}}$, Mg, Si, K, Ti, Mn, Y, Zr, Nb and Pb. These
462 fingerprinting properties were selected based on weak and non-significant linear relationship between tracer
463 concentrations and SSA and SOM, thereby negligible alteration of tracer properties due to enrichment or
464 depletion, and conservative behaviour supported by the inclusion of sediment samples within source convex
465 hull boundaries for all tracer combinations. Furthermore, Si, Ti, Y and Zr have been described as conservative
466 due to their preference to be matrix-bound to sediments (Collins et al. 2020) and $^{210}\text{Pb}_{\text{ex}}$ have enough half-live
467 to consider it as conservative in contemporary sediment dynamics (Koiter et al. 2013). In contrast, the use of K
468 and Mg as tracers has been challenged in sediment fingerprinting investigations due to its water solubility
469 potential (Kraushaar et al. 2015). Nevertheless, these two tracers were kept in mixing modelling calculations

470 due to their discriminatory potential between surface and sub-surface sources in a forest fire context (Blake et
471 al. 2006b; Owens et al. 2006; Smith et al. 2013).

472 Principal Component Analysis of selected tracers showed an important overlap between surface and
473 channel banks ellipses. This could be attributed to the influence of surface material inputs into some parts of
474 stream banks due to ash export downslope where soil colour denotes the influence of burnt surface in some
475 areas (see Fig.2b). It is important to highlight the influence of $^{210}\text{Pb}_{\text{ex}}$, Mn and Pb elemental concentration on
476 these sources (see Fig. 4 and Fig. 8). Moreover, overlap between roads and sub-surface ellipses was also
477 observed, indicating the similar geochemistry between these two sources (mainly explained by the influence of
478 Zr and Y concentrations).

479 **4.3 Post-fire sediment source apportionment and its implications.**

480 **4.3.1 Catchment hydrological response to wildfire**

481 The increase in streamflow after fire (Fig. 3) is attributed not only to rainfall, it is also linked to reduced
482 infiltration that may be compounded by soil water repellency and reduction in evapotranspiration (Ice et al.
483 2004; Certini 2005; Shakesby and Doerr 2006) and also the loss of rainfall interception by the canopy (up to
484 65% reduction in the rainfall interception according to Williams et al. 2019). These changes may have generated
485 an increase in runoff and subsequently an increase in flow discharge (Fig. 3).

486 **4.3.2 Shifts in post-fire sediment sources.**

487 The dominance of shallow sub-surface material as a sediment source during the first two periods can
488 be attributed to rill erosion that took place in the catchment hillslopes after the wildfire due to convergence of
489 overland flow from the first rainfall (as seen in Fig. 11 a-b). The generation of surface runoff and rill erosion
490 mechanisms in burnt catchments have been reported because of reduced infiltration capacity due to clogging of
491 pore space by ash and/or soil water repellency distribution in the soil profile during wildfire (Ice et al. 2004;
492 Shakesby and Doerr 2006). Under the “extreme” soil water repellency model, rainfall saturates a wettable burnt
493 surface soil layer which slides downslope over an enhanced water repellent sub-surface carrying substantial
494 quantities of ashes and sediments (*e.g.* surface soil in this study) (DeBano 2000; Shakesby and Doerr 2006;
495 Smith et al. 2013). While overland flow processes were clearly enhanced in the Quivolgo catchment due to the
496 rainfall received during winter months (maximum rainfall peak reached during July 2017, see Fig. 3), a post-

497 fire water repellency-driven sheet-wash/mass wasting model does not support the observation of dominant sub-
498 surface soil release by rill erosion (2 – 4 cm). Here, it seems more likely that the low-infiltration capacity of
499 ash-covered surface has remained relatively cohesive and the overland flow has only achieved sufficient
500 competence to entrain surficial material along flow convergence lines which are rapidly incised leading to
501 dominant sub-surface soil erosion through rilling.

502 Uppermost surface material (0 – 2 cm) was not an important sediment source in the three periods
503 evaluated (Fig. 9). The burnt surface material was observed in inter-rill zones in situ during the sampling
504 campaign (May 2017, three months after the wildfire finished, Fig. 3a) when the rainy period had recently
505 started. This finding contrasts with other studies that observed burnt hillslopes surface as the major sediment
506 source in catchments affected by wildfires (Blake et al. 2009b; Wilkinson et al. 2009; Smith et al. 2011b) where
507 arguably soil water repellency dynamics are notably enhanced due to local vegetation type (e.g. eucalyptus).
508 Furthermore, post-fire erosion from channel banks was not an important sediment source in the three periods
509 evaluated, which contrasts with observations by Owens et al. (2012) who attributed that sub-surface/channel
510 bank erosion were key factors in sediment production in forested catchments after fire. This discrepancy may
511 be due to the following factors: i) buffer zone stabilisation due to riparian vegetation before the fire specially at
512 the first sampling period, in contrast to Owens et al. (2012) where tree death led to channel-side tree falls and
513 loss of root strength, and ii) water level and streamflow power during the rainy periods was not sufficient to
514 erode banks. Although, differences in study design and source sampling should be considered.

515 Forest roads have been reported as a major sediment source in commercial forest catchments without
516 wildfire impacts (Motha et al. 2003; Schuller et al. 2013; Brandt et al. 2018; Bravo-Linares et al. 2020).
517 Additionally, a study was performed in the Quivolgo catchment before the present wildfire condition (2014)
518 using the carbon isotopic composition of fatty acids (CSSI fingerprint) to apportion sediment sources coming
519 from roads and soils under pine plantations, native forest and riparian vegetation or buffer zone (Bravo-Linares
520 et al. 2018). This study showed that unpaved roads were a major sediment source throughout the hydrological
521 year. In the post-wildfire situation, road source contribution to sediments, as a proportion, was only important
522 at the end of the rainy period (October 2017), indicating that fire switched on a new sediment source
523 immediately after the rainy period started, *i.e.* rill erosion, which dominated and overprinted the “background”

524 contribution from roads. This implies a substantially greater volume of sediment mobilised from sub-surface
525 compared to roads during the rainy period post-fire.

526 The mixing model apportionment showed a strong negative correlation between sub-surface and roads
527 global posterior distributions ($r = -0.71$, Supplementary Figure 2) indicating that contribution of one source
528 could be trading off against the other (Parnell et al. 2010, 2013). The reason for this has been attributed to the
529 similarities between sources being evaluated. In this case, it is possible to link the similarity between these two
530 sources to mobilisation of sub-surface material into roads. Nevertheless, this mixing process was avoided by
531 taking samples at the opposite side of forest slopes. When there is evidence that can support similarities between
532 two or more sources (*e.g.* field observations, physical processes, etc.), data can be combined either *a priori* or
533 *a posteriori* (Phillips et al. 2014; Stock et al. 2018; Upadhyay et al. 2018). However, the posterior source
534 distribution aggregation ability of MixSIAR mixing model requires careful consideration when using it in
535 sediment apportionment. In our case, we did not find sufficient plausible reasons to support the sub-surface and
536 roads posterior source distribution aggregation, even when it was possible relying on frequentist statistical
537 testing that showed no significant differences between these two sources in nine of ten tracers used in source
538 apportionment (Stock et al. 2018). Other studies under coniferous tree cover have reported subsoil as the
539 dominant sediment source after wildfire using ^{137}Cs and $^{210}\text{Pb}_{\text{ex}}$ as tracers, including channel banks as part of
540 subsoil source group because of its similar radionuclide composition (Owens et al. 2012). Here, the inclusion
541 of geochemical tracers in the unmixing process allowed sub-surface soil material to be distinguished from
542 channel bank material. This was despite similarities in terms of $^{210}\text{Pb}_{\text{ex}}$ and geochemical composition, with low
543 global posterior distribution correlation between them ($r = -0.2$, Supplementary Figure 2) demonstrating that,
544 for both sources, posterior distributions from the mixing model output are distinguishable (Parnell et al. 2010;
545 Phillips et al. 2014).

546 **4.3.3 Post-fire apportionment considerations and limitations**

547 The application of sediment fingerprinting technology in burnt commercial forest catchments is a
548 valuable tool for forest managers to reveal sources that could contribute sediments to waterbodies and processes
549 therein. To our knowledge, this is the first study that explores the tracer selection procedure based on the
550 exclusion of non-conservative tracers in sediment source apportionment immediately after a wildfire in a

551 catchment with forest plantations. However, there are some limitations in our study that should be addressed in
552 the future: 1) The number of samples per source category should be increased in order to account adequately
553 spatial variability, especially at surface and channel banks source categories, where we found substantial
554 variability in FRNs activity and in geochemical elements concentrations. 2) As a consequence of above,
555 discrimination capacity among sources could be reduced (as shown by PCA plot, Fig. 8) when selecting tracers
556 via exclusion of non-conservative tracers (Smith et al. 2018). Although, comparison with the classical tracer
557 selection procedure (which maximise differences between source categories) did not show differences in terms
558 of the dominant sources contributing to the weir. In fact, this process included one tracer, Cl, that showed
559 important correlation with SSA and SOM content and wider posterior probabilities distributions than the
560 method applied in this study, and 3) We encourage the application of tracer selection procedure based on the
561 exclusion of non-conservative tracers in burnt landscapes knowing the implication that SOM and SSA
562 alterations could have in tracer properties. Additionally, testing model and tracer accuracy and precision with
563 artificial mixtures is advised in order to improve source discrimination capacity and the quality of mixing model
564 outputs.

565 **5 Conclusions**

566 Sediment sources within a forest catchment affected by wildfire were assessed using the MixSIAR
567 mixing model. The exploration of environmental radionuclides together with geochemical elements, through a
568 selection procedure based on the examination of tracer conservative behaviour, led to ten fingerprinting
569 properties (1 FRN and 9 geochemical elements) being used in sediment apportionment within a wildfire forest
570 catchment context. The tracers that passed selection criteria demonstrated their suitability to be used due to
571 following reasons: i) weak and non-significant linear relationship between tracer concentrations and SSA and
572 SOM, thereby negligible alteration of tracer properties due to enrichment or depletion, and ii) conservative
573 behaviour supported by the inclusion of sediment samples within source convex hull boundaries for all tracer
574 combinations.

575 The dominant sediment source during the first two periods evaluated was shallow sub-surface soil (2
576 – 4 cm), demonstrating a shift in the dominant sediment source in the forest catchment after being affected by
577 a wildfire, and the main drivers of sediment release were the overland flow and consequent rill erosion that

578 occurred immediately after the rainy period started. In addition, forest roads contribution was also important,
579 especially during the last period evaluated, which has been reported by other studies in non-fire affected forested
580 catchments. However, considerations should be given to 1) the strong negative correlation between the sub-
581 surface soil and forest roads global posterior distributions found in the mixing model apportionment, 2) the
582 sample size of some source categories *e.g.* channel banks and 3) the low discrimination capacity of the selected
583 tracers.

584 Finally, climate change and land use transformation into massive plantations of exotic tree species can
585 increase wildfire frequency which in turn creates landscapes that are more vulnerable to erosion processes. The
586 increase in the number of wildfires in Chile will generate a sustained source of sediment release into
587 waterbodies, especially during rainy events with consequences for water supply and hydropower production.
588 Early post-fire targeting and management of the potential sediment sources in forested catchment can reduce
589 the off-site impacts of ashes and sediments released after rainy episodes. Herein, sediment fingerprinting can
590 become an affordable tool for forest companies to assess and develop focalised strategies when attempting to
591 face soil erosion problems and its downstream impacts that are risen after a wildfire event.

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763

764 **7 Tables**

765 **Table 1** Percentage of Soil Organic Matter and activity concentration (Bq kg⁻¹) of ¹³⁷Cs and ²¹⁰Pb_{ex} of
 766 hillslope surface soil samples from pre- and post-fire situation in the Quivolgo catchment. Soil samples from
 767 hillslopes were collected from the topsoil first centimetre in 2014 (data not published).

	Pre-fire						
	SOM (%)		¹³⁷ Cs (Bq kg ⁻¹)		²¹⁰ Pb _{ex} (Bq kg ⁻¹)		n
	Mean	SD	Mean	SD	Mean	SD	
Forest Slopes (0 – 1 cm)	25.1	10.1	3.5	1.2	16.5	7.0	
	Post-fire						
	SOM (%)		¹³⁷ Cs (Bq kg ⁻¹)		²¹⁰ Pb _{ex} (Bq kg ⁻¹)		n
	Mean	SD	Mean	SD	Mean	SD	
Surface soil (0 – 2 cm)	12.2	2.9	5.9	2.1	77.4	30.0	
Sub-surface soil (2 – 4 cm)	16.2	3.2	5.4	1.7	40.8	12.3	8

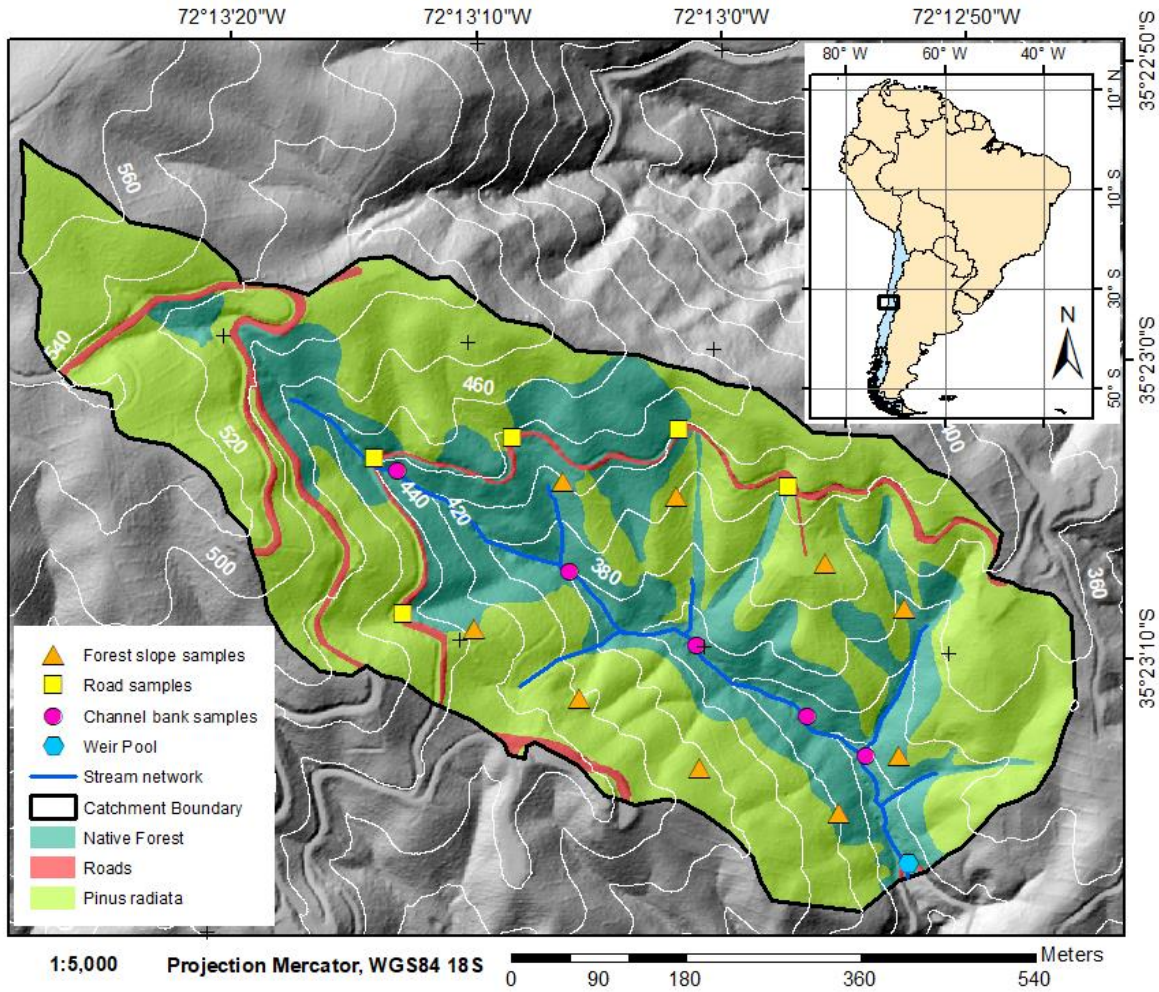
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770 **8 Figure captions**

771 **8.1 Fig. 1**

772 Location map of the Quivolgo catchment in Central Chile (35°23' S, 72°13' W) showing land uses
773 before wildfire and source and sediment sample locations.

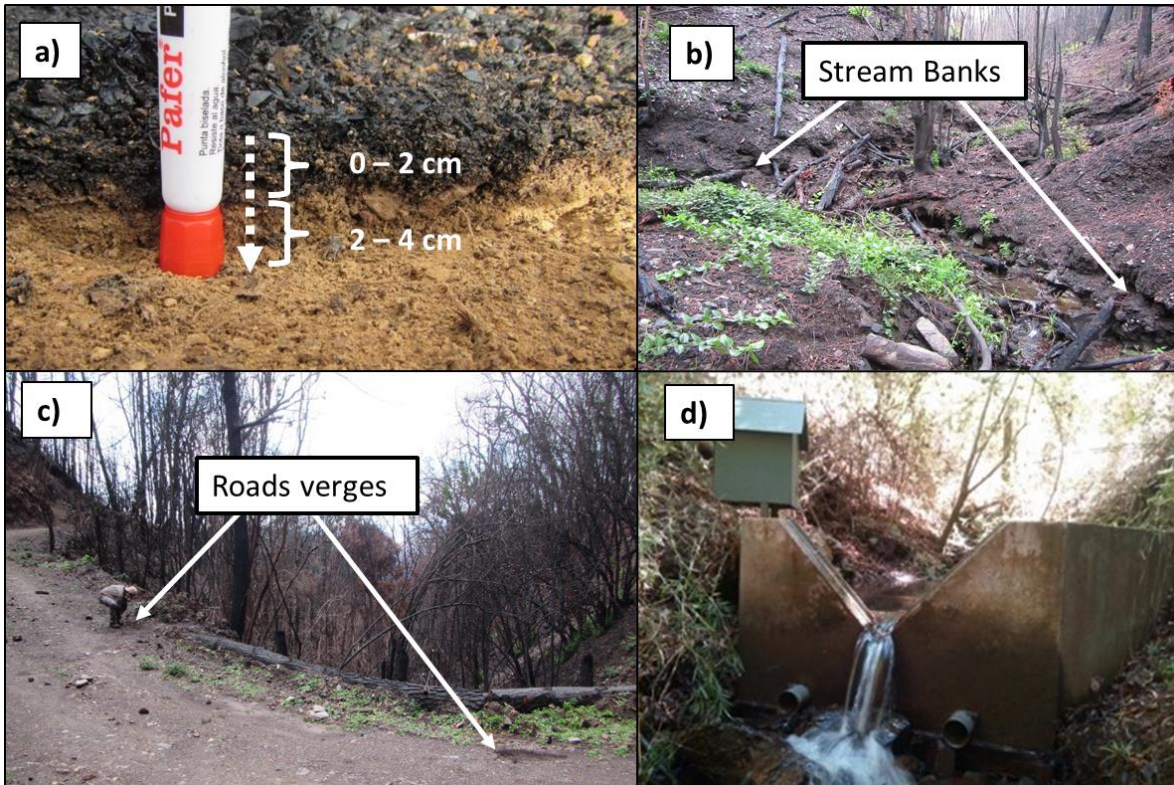


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775

776 **8.2 Fig. 2**

777 a) Surface and sub-surface soil layers of the burnt catchment, b) catchment stream/channel banks, c)
778 roads samples taken from the verges and d) V-notched weir pool that receipts sediments transported by the
779 stream channel.

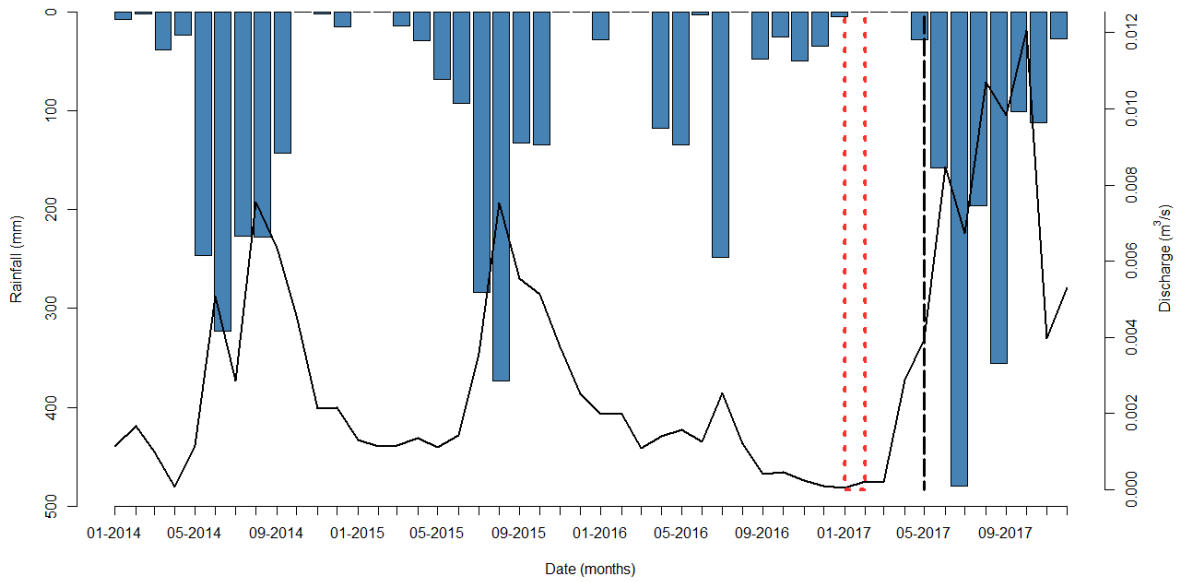


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782 **8.3 Fig. 3**

783 Accumulated monthly rainfall (mm) and flow discharge (m^3/s) at weir between 2014 and 2017 in
784 Quivolgo catchment. The period when the wildfire occurred is delimited with a red dotted line, the sources
785 sampling campaign is indicated by a black vertical dashed line. Note that the rainy period starts in April – May
786 and ends in October (southern hemisphere).

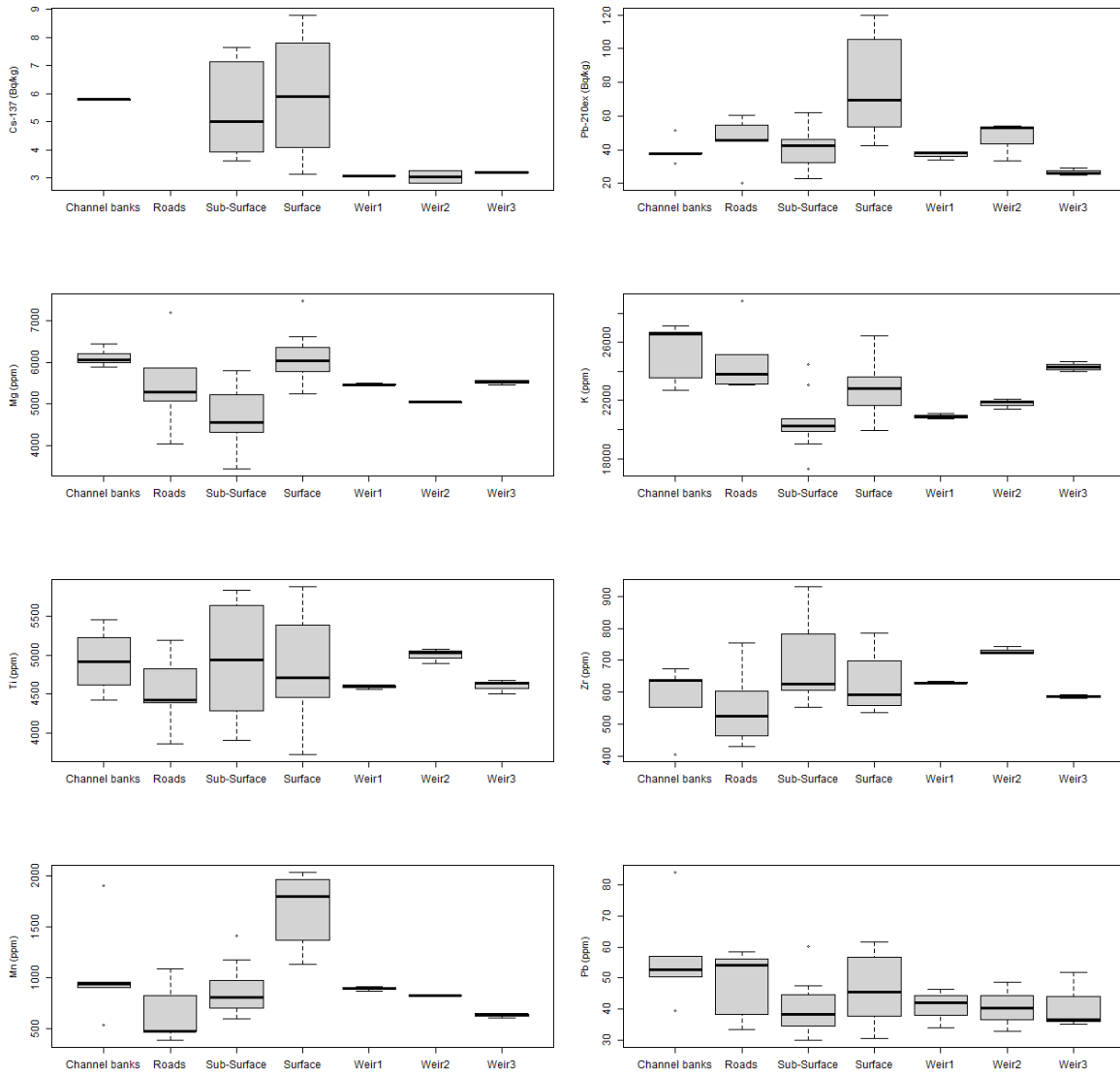


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789 **8.4 Fig. 4**

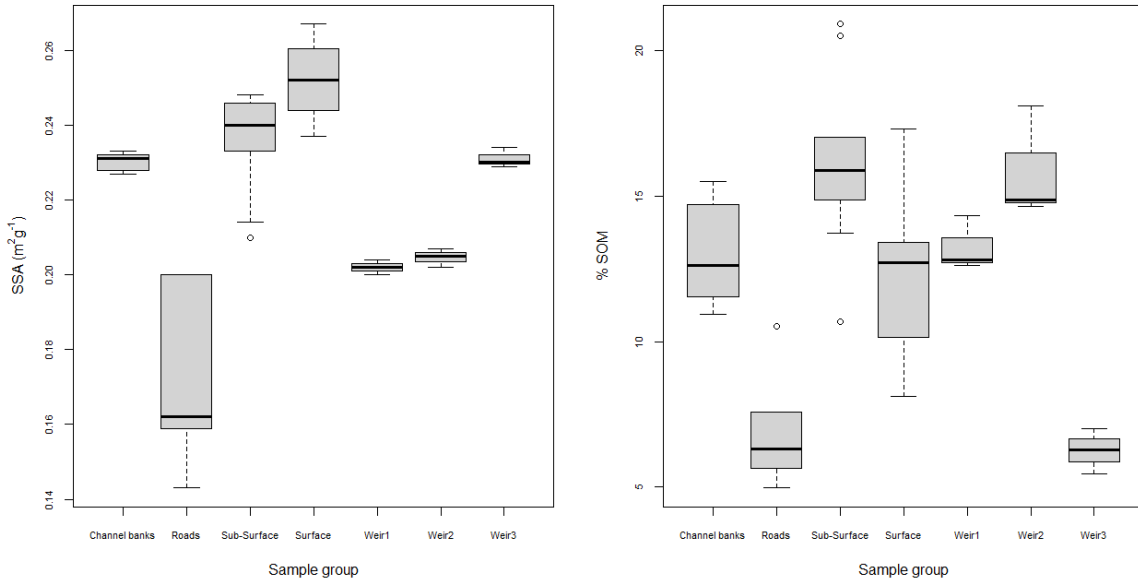
790 Boxplots of soil and sediment samples values distribution of FRNs (^{137}Cs and $^{210}\text{Pb}_{\text{ex}}$) and some
 791 geochemical elements (*i.e.* Mg, K, Ti, Zr, Mn and Pb). ^{137}Cs activity in roads samples were all below minimum
 792 detectable activity (< MDA).



793

794 **8.5 Fig. 5**

795 Boxplots of SSA ($\text{m}^2 \text{g}^{-1}$) and SOM (% Loss-on-Ignition) of samples distribution for source soils and
796 weir pool sediment samples ($< 63 \mu\text{m}$ fraction).



797

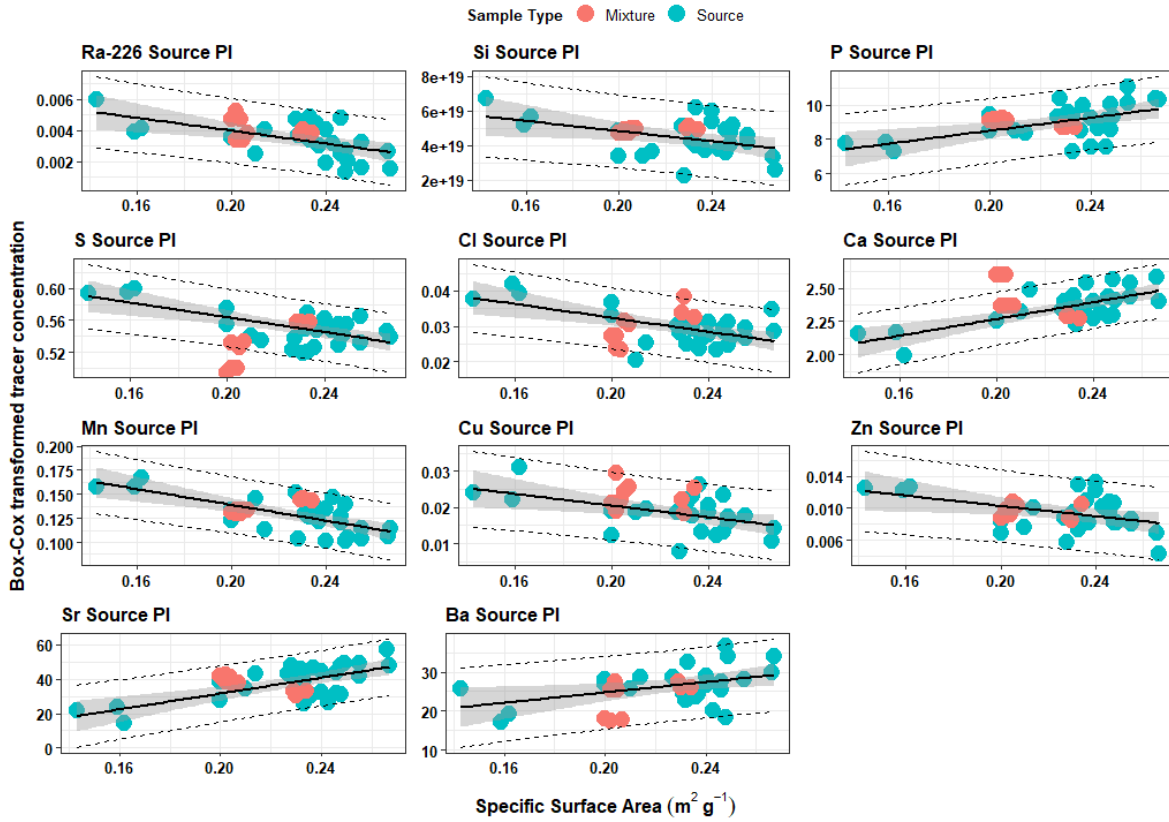
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801 **8.6 Fig. 6**

802 95% Prediction Intervals (dashed lines) and 95% confidence interval (shaded area) of sources linear
803 regression between the Specific Surface Area ($\text{m}^2 \text{g}^{-1}$) and Box-Cox transformed tracer concentration.

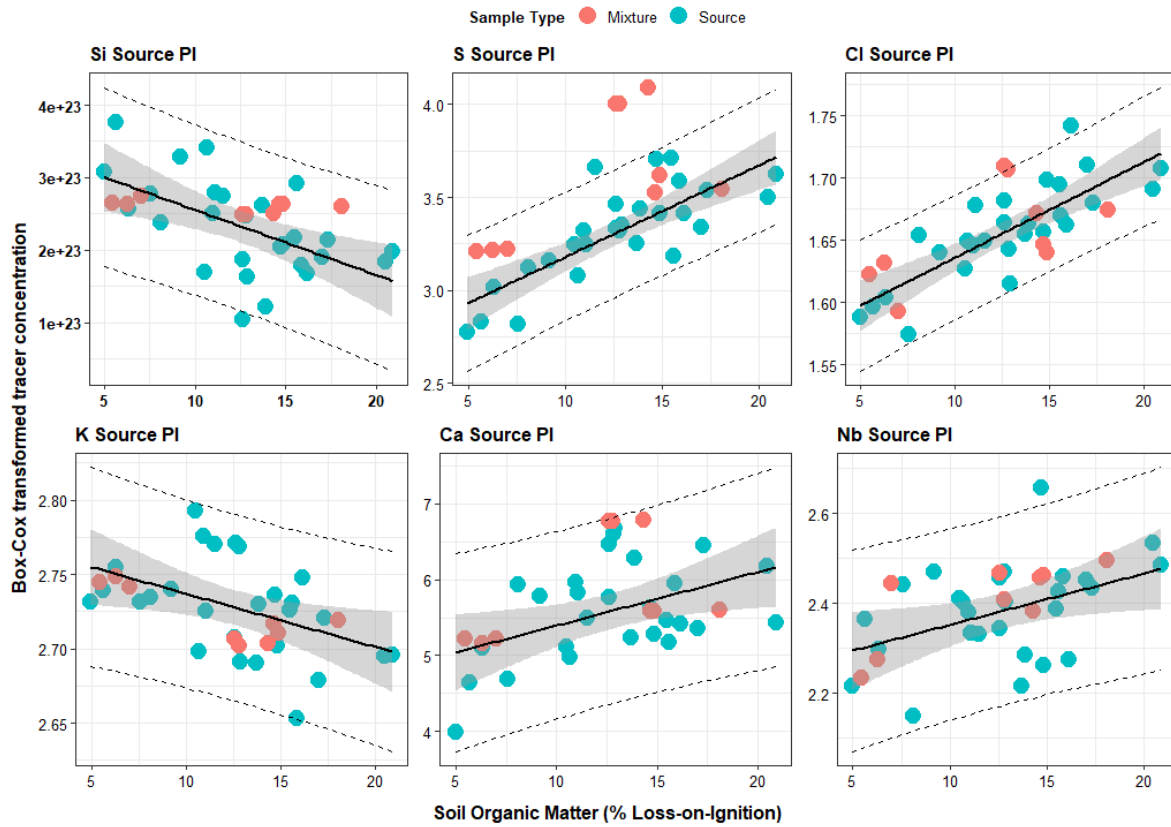


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806 **8.7 Fig. 7**

807 95% Prediction Intervals (dashed lines) and 95% confidence interval (shaded area) of sources linear
808 regression between the Soil Organic Matter (% Loss-on-Ignition) and Box-Cox transformed tracer
809 concentration.

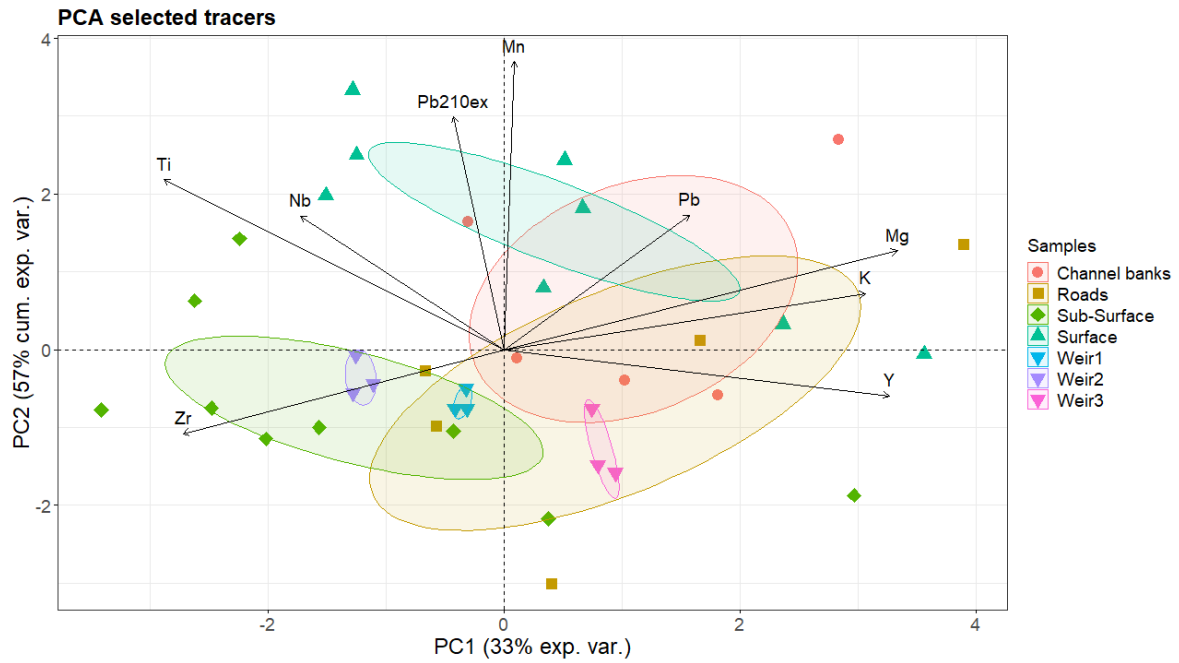


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812 **8.8 Fig. 8**

813 Principal Component Analysis of tracers selected by the criteria applied for burnt catchment sediment
814 apportionment. Ellipses represents 95% confidence level of groups distributions in the hyper-space. In the case
815 of missing values, the mean tracer value was included for visualisation purposes.

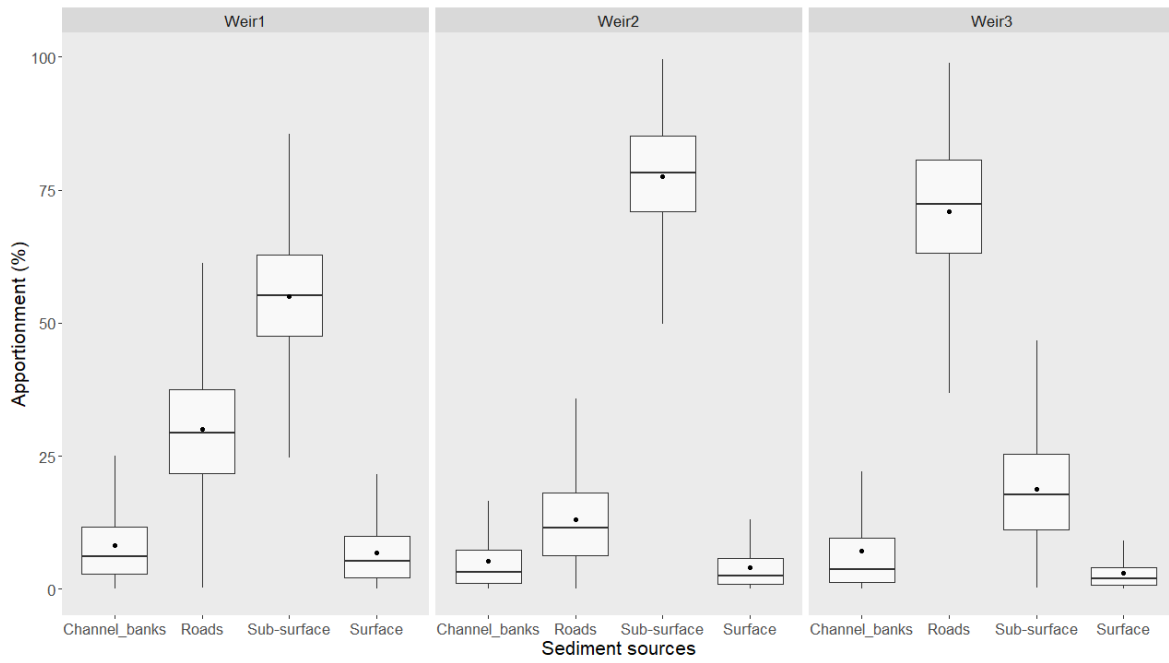


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818 **8.9 Fig. 9**

819 Boxplots of posterior source probability distributions (%) for post-fire sediment samples during May
820 (Weir1), July (Weir2) and October (Weir3) 2017.

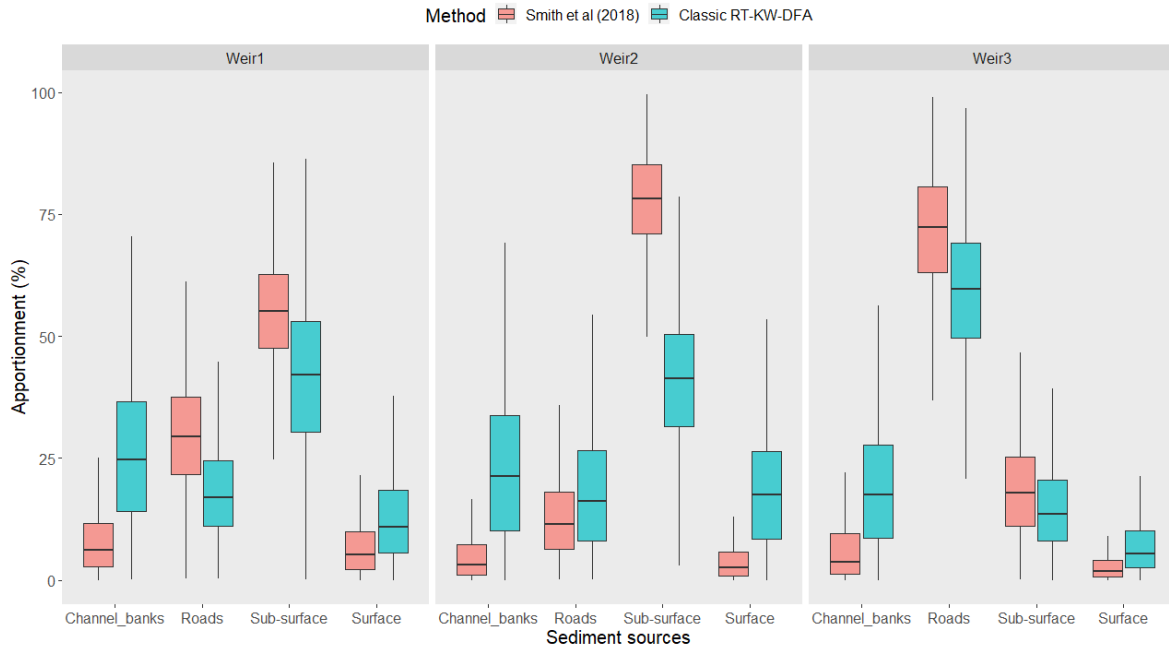


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822

823 **8.10 Fig. 10**

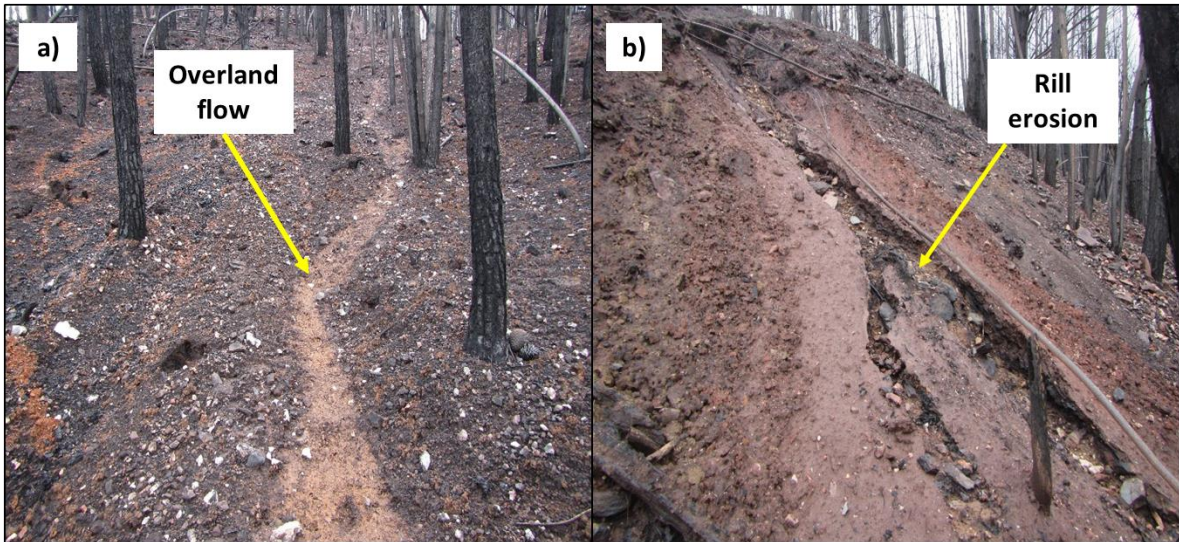
824 Boxplots for posterior source probability distributions (%) obtained from the two tracer selection
825 methods: Classical range test, Kruskal-Wallis test and Discriminant Function Analysis and the method
826 described by Smith et al. (2018). RT, KW and DFA refer to range test, Kruskal-Wallis test and Discriminant
827 Function Analysis, respectively.



828

829 **8.11 Fig. 11**

830 Steep slopes in Quivolgo catchment where overland flow (a) and consequent rill erosion (b) are the
831 main drivers of sediment transport towards the stream channel.



832