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1 Tracing sediment sources in a tropical highland catchment of central Mexico
2 by using conventional and alternative fingerprinting methods
3

4 Olivier Evrard^{a,*}, Jérôme Poulenard^b, Julien Némery^c, Sophie Ayrault^a, Nicolas Gratiot^d,
5 Clément Duvert^d, Christian Prat^d, Irène Lefèvre^a, Philippe Bonté^a, Michel Esteves^d
6

7 ^a Laboratoire des Sciences du Climat et de l'Environnement (LSCE/IPSL) – Unité Mixte de Recherche 8212
8 (CEA, CNRS, UVSQ), 91198-Gif-sur-Yvette Cedex, France

9 ^b Université de Savoie, Environnements Dynamiques et Territoires de Montagne (EDYTEM), Savoie
10 Technolac, 73376-Le Bourget du Lac Cedex, France

11 ^c G-INP/Université Grenoble 1, Laboratoire d'étude des Transferts en Hydrologie et Environnement UMR 5564,
12 Grenoble, France

13 ^d IRD/Université Grenoble 1/CNRS, Laboratoire d'étude des Transferts en Hydrologie et Environnement UMR
14 5564, Grenoble, France

15 *Corresponding author. Address : LSCE – Centre de Recherche du CNRS – Avenue de la Terrasse, bât. 12. F-
16 91198 Gif-sur-Yvette Cedex (France). E-mail address : olivier.evrard@lsce.ipsl.fr. Telephone: +33/1/69 82 35
17 20. Fax: +33/1/69 82 35 68.

18 **Abstract (max. 250 words)**

19 Land degradation is intense in tropical regions where it causes for instance a decline in soil fertility
20 and reservoir siltation. Two fingerprinting approaches (i.e., the conventional approach based on
21 radionuclide and geochemical concentrations and the alternative Diffuse Reflectance Infrared Fourier
22 Transform Spectroscopy method) were conducted independently to outline the sources delivering
23 sediment to the river network draining into the Cointzio reservoir, in Mexican tropical highlands. This
24 study was conducted between May and October in 2009 in subcatchments representative of the
25 different environments supplying sediment to the river network. Overall, Cointzio catchment is
26 characterised by very altered soils and the dominance of Andisols and Acrisols. Both fingerprinting
27 methods provided very similar results regarding the origin of sediment in Huertitas subcatchment
28 (dominated by Acrisols) where the bulk of sediment was supplied by gullies. In contrast, in La Cortina
29 subcatchment dominated by Andisols, the bulk of sediment was supplied by cropland. Sediment
30 originating from Potrerillos subcatchment characterised by a mix of Acrisols and Andisols was
31 supplied in variable proportions by both gullies and rangeland/cropland. In this latter subcatchment,
32 results provided by both fingerprinting methods were very variable. Our results outline the need to
33 take the organic carbon content of soils into account and the difficulty to use geochemical properties to
34 fingerprint sediment in very altered volcanic catchments. However, combining our fingerprinting
35 results with sediment export data provided a way to prioritise the implementation of erosion control
36 measures to mitigate sediment supply to the Cointzio reservoir supplying drinking water to Morelia
37 city.

38 **Keywords**

39 Sediment; fingerprinting; soil types; Mexico; tropical catchment.

1. Introduction

Land degradation is particularly severe in tropical regions, such as in Mexico (Descroix et al., 2008), in southern China (Barton et al., 2004) or in eastern Africa (Nyssen et al., 2004). In Mexico, overgrazing, deforestation, and the intensification of food crop cultivation have led to severe erosion and to a decline in soil fertility (Roldán et al., 2003). Furthermore, once it reaches the river, sediment leads to numerous problems in downstream areas (Owens et al., 2005). It causes for instance an increase in water turbidity and a rapid filling of reservoirs (Syvitski et al., 2005). Sediment is also associated with numerous contaminants (e.g., metals, organic compounds, antibiotics, radionuclides; e.g., Tamtam et al., 2011; Le Cloarec et al., 2011). Their integration into the food chain can lead to public health problems after the consumption of contaminated fish (Sánchez-Chardi et al., 2009; Urban et al., 2009). Sediment also conveys nutrients, and soil erosion and deposition play therefore a significant role in global biogeochemical cycles (Quinton et al., 2010). Furthermore, in mountainous environments, the problems associated with erosion and sedimentation are exacerbated by the large quantities of sediment produced within very short periods (Meybeck et al., 2003; Mano et al., 2009).

Sediment supply to the river needs to be controlled to prevent these problems. However, there is a preliminary need to determine the main erosion sources to implement appropriate and effective erosion mitigation measures. In tropical areas such as the highlands of central Mexico where hydrology is controlled by the succession of a dry and a rainy season, it is generally assumed that the increase in discharge at the beginning of the rainy season can lead to an important resuspension of sediment accumulated in the river channel (e.g. Susperregui et al., 2009). Evrard et al. (2010) showed that the first storms of the year exported the bulk of the sediment stock accumulated in the river channel during the previous rainy season. However, this study also outlined that sediment can also be directly eroded from hillslopes and exported from small (3–12 km²) catchments during individual heavy storms. Furthermore, the contribution of different sediment source areas (e.g., historical gullies vs. cropland) was suspected, but it remained to be quantified in order to prioritise the implementation of erosion control measures.

We propose to use two different sediment fingerprinting techniques to outline the main sources of sediment within those catchments. The fingerprinting method consists in tracing conservative sediment properties or characteristics that can be identified in both catchment sources and sediment delivered downstream (Walling, 2005). So far, very few fingerprinting

75 studies have been conducted in tropical regions (see for instance Collins et al., 2001, in a
76 catchment of Zambia). Fingerprinting generally requires a multi-tracer approach. Besides
77 ‘conventional’ fingerprinting based on the measurement of radionuclides and geochemical
78 elements, alternative fingerprints have been recently used such as sediment colour properties
79 (Martínez-Carreras et al., 2010a, b) and Diffuse Reflectance Infrared Fourier Transform
80 Spectroscopy (DRIFTS; Poulenard et al., 2009; Poulenard et al., in press) to trace the origin of
81 suspended sediment transported by rivers.

82 In this context, we conduct two independent fingerprinting exercises (i.e., the
83 conventional approach based on radionuclide and geochemical concentrations, and the
84 alternative DRIFTS method) to quantify sediment sources in three small tropical catchments
85 of the Mexican Central Highlands. Those three areas cover the range of land use, topographic
86 gradients and soil conditions of the potential areas delivering sediment to a reservoir
87 providing 25% of the water distributed in the region of Morelia city (ca. 1,000,000
88 inhabitants). The implications of the sediment fingerprinting results to control reservoir
89 siltation will also be discussed. Factors controlling sediment fluxes exported from those
90 catchments and sediment transfer times within those areas are discussed elsewhere (Duvert et
91 al., 2010, 2011; Evrard et al., 2010).

92 **2. Materials and methods**

93 **2.1 Study area**

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95
96
97 The Cointzio catchment covers an area of 630 km² located in the transverse volcanic
98 belt of central Mexico (Fig. 1). The catchment bedrock consists of igneous rocks generated by
99 Quaternary volcanic activities. Soils within the catchment are mainly Acrisols on the hillsides,
100 Andisols in headwater areas and Luvisols in the plains (FAO, 2006). The river network is
101 dominated by the Grande de Morelia River. A dam is located at the outlet of the catchment,
102 13 km upstream of Morelia city (ca. 1,000,000 inhabitants). This dam was built in 1940 to
103 supply water for domestic consumption as well as for industrial and agricultural activities.
104 The Cointzio reservoir (4 km²; 65×10⁶ m³) undergoes significant sedimentation, which has
105 led to a severe deterioration of environmental conditions in the lake (Ramirez-Olvera et al.,
106 2004) and to a 20% loss of its water storage capacity since its construction (Susperregui et al.,
107 2009).

108 Three subcatchments representative of the various land use, slope gradients and soil
109 conditions found in the Cointzio catchment were monitored in the framework of this study:

110 Huertitas (3 km²), La Cortina (9 km²) and Potrerillos (12 km²; Fig. 1). Their characteristics
111 are described in Table 1. In addition, we monitored the river discharge and sediment fluxes in
112 Santiago Undameo, at the outlet of the entire catchment, just upstream of Cointzio reservoir
113 (Gratiot et al., 2010; Duvert et al., 2011). It is important to note that villages located within
114 the catchment are not equipped with sanitation systems and that they directly discharge their
115 wastewater into the river network.

116

117 2.2. Field measurements

118

119 *Rainfall and discharge*

120 Rain gauges and river monitoring stations were installed in the three subcatchments (Fig.
121 1). They provided continuous precipitation and water discharge data derived from continuous
122 water level measurements (with a 5-min time step) obtained with a Thalimedde OTT water-
123 level gauge, and discharge calculated using stage-discharge rating curves (see Duvert et al.,
124 2010, for details on this method).

125

126 *Measurement of SSC*

127 At the outlet of each subcatchment, data on Suspended Sediment Concentration (SSC; g
128 L⁻¹) were obtained using an automatic water sampler (ISCO 3700) triggered by water level
129 variations. During floods, water samples were collected after each 5-cm water level variation.
130 This sampling frequency was selected based on the mean characteristics (i.e., flood duration,
131 shape of rising and falling limbs) of the floods recorded previously (i.e., between 2006 –
132 2008; Duvert and Gratiot, unpublished data) in the subcatchments to obtain a trade-off
133 between a satisfactory flood coverage and a reasonable amount of samples to collect in the
134 field (see Duvert et al., 2010 and Duvert et al., 2011 for details).

135 SSC (generally ≥ 2 g L⁻¹) was estimated at the laboratory after drying the entire sample for
136 24 hours at 60°C. For each flood, a composite sample was prepared by mixing all the
137 available individual samples. This provided a mean representative sample of each individual
138 flood with a sufficient quantity of fine sediment (2–50 g) to conduct radionuclide and
139 DRIFTS analyses. Details on the calculation of sediment fluxes at the outlet of the different
140 subcatchments can be found in Duvert et al. (2010). In total, 40 events that occurred between
141 May and November 2009 were sampled throughout the rainy season at the outlet of the three
142 subcatchments.

143

144 *Soil collection*

145 Soil representative of the different land uses (i.e., gullies, cropland, woodland)
146 observed in the three subcatchments was collected. Sampling was concentrated in potential
147 sediment source areas (i.e., sites sensitive to erosion, and potentially connected to the river
148 network). For each potential source (i.e., gullies, cropland, woodland), we collected five
149 samples of surface material potentially submitted to erosion processes and connected to the
150 river (top 0–5 cm; i.e. 0–2 cm at most locations and 0–5 cm where the erosion extent
151 warranted a deeper sampling depth) and mixed them well to provide a homogeneous sample.
152 In total, 17 representative composite samples were collected in the field between June and
153 November 2009 (Fig. 1). This number of sources samples is rather limited, mainly due to
154 practical and logistical reasons, but stress was laid in the field on providing representative
155 samples of each land use class. In ideal conditions, more samples could have been collected.
156 If we were aiming to discriminate the contribution of different lithological sources to
157 suspended sediment, it would not have been enough to capture the within-source variability of
158 fingerprint properties. However, this study aims to discriminate land use sources. We will
159 therefore add a step to the conventional fingerprinting procedure by selecting the most
160 relevant tracers from a physiochemical point of view to achieve this specific objective.

161 Furthermore, riverbed sediment was collected on exposed sites located along the main
162 river channel network, using non-metallic trowels in order to avoid sample contamination.
163 Several subsamples (~ 10) were collected in January 2008 and in June 2009 at each of the 18
164 locations selected along the river network. They were used to prepare composite samples
165 representative of the sediment deposited on the riverbed.

166

167 *2.3 Soil and sediment analysis*

168 *Radionuclide measurements*

169 All suspended sediment and soil samples were dried and sieved (< 250 µm) before
170 analysis. To check that grain size of particles remained similar between soil and sediment
171 samples, grain size distribution was determined with a Malvern® particle size analyzer after
172 being submitted to a 10-min ultrasonic agitation.

173 Fallout and geogenic radionuclides were measured in all the collected samples ($n=55$ with
174 17 composite soil samples, 18 composite riverbed samples and 20 composite suspended
175 sediment samples), whereas the analyses of elemental geochemistry were carried out on a
176 selection of samples ($n=37$, i.e. 17 composite soil samples and 20 composite suspended

177 sediment samples). For the measurement of radionuclides in each sample, soil and sediment
178 were placed in a counting box. Fallout (Am-241, Be-7, Cs-137, Pb-210) and geogenic (K-40,
179 Ra-226, Ra-228, Th-228, Th-234) radionuclide concentrations were determined by gamma-
180 spectrometry using the very low-background coaxial N- and P-types GeHP detectors
181 (Canberra / Ortec) available at the *Laboratoire des Sciences du Climat et de l'Environnement*
182 (Gif-sur-Yvette, France). “Excess” ^{210}Pb ($^{210}\text{Pb}_{\text{xs}}$) was calculated by subtracting the supported
183 activity (determined using two ^{238}U daughters, i.e. ^{214}Pb , by taking the average count number
184 at 295.2 and 351.9 keV, and ^{214}Bi at 609.3 keV) from the total activity of ^{210}Pb (measured at
185 46.5 keV). Efficiencies and background levels of the detectors were periodically controlled
186 with internal and IAEA soil and sediment standards. Radionuclide activities were
187 systematically corrected taking account of the decay after the sampling period.

188

189 *Geochemical measurements*

190 For the measurement of elemental geochemistry, Rare Earth Elements (REE; i.e., Ce, Eu,
191 La, Lu, Sm, Tb, Yb), three major elements (Fe, K, Na) and ten trace elements (As, Ba, Co,
192 Cr, Cs, Hf, Sc, Ta, Th, Zn) were analysed by Instrumental Neutron Activation Analysis
193 (INAA). Dried subsamples (ca. 40–80 mg) were packed into tightly closed plastic bags,
194 without any preliminary digestion. The subsamples were exposed to irradiation at the
195 experimental nuclear reactor Orphée of the *Commissariat à l’Energie Atomique* (CEA;
196 Saclay, France). The subsamples underwent a flux of thermal neutrons of $2.13 \times 10^{13} \text{ n cm}^{-2} \text{ s}^{-1}$
197 during 30 minutes. After a 4-days cooling, four successive measurements of gamma activities
198 were carried out using HPGe detectors. Two reference materials (i.e., IAEA SL-1 and Soil-7)
199 were systematically used to cross-check the results. Uncertainty on these measurements is \leq
200 5%.

201 *Carbon and Nitrogen content measurements*

202 Soil and suspended sediment content in bulk Carbon (C) and Nitrogen (N) was measured
203 by CHN analysis using a CN-analyzer FlashEA 1112 (Thermo Fisher Sci., MA, USA) at the
204 *Laboratoire d’Ecologie Alpine* (LECA; Grenoble, France). About 20 mg of each sample were
205 burnt at high temperature with a catalyser under helium flux, to transform total C into carbon
206 dioxide and total N into N_2 . Obtained CO_2 and N_2 were separated using a gas chromatography
207 column and analysed by thermal conductivity detector. Results are expressed in %C and %N.
208 Soil content in carbonates was analysed at the INRA soil analysis laboratory in Arras (France)
209 using classical calcimetric method (Robertson et al, 1999) according to the normative
210 procedure NF ISO 10693. Results are expressed in %C (CaCO_3).

211

212 *Isotopic analyses*

213 Furthermore, $\delta^{13}\text{C}$ measurements were conducted to outline potentially different
214 contributions of avocado fields and maize fields, based on the difference in the stable carbon
215 isotope signatures between C3 and C4 plants (Balesdent and Wagner, 1988). $\delta^{13}\text{C}$ analyses
216 were conducted on representative samples from (i) maize fields and (ii) avocado fields from
217 La Cortina subcatchment to test the potential discrimination between both sources. Analyses
218 were conducted at LSCE on an EA-IRMS devoted to organic samples (Thermo Fischer
219 Delta+XP).

220

221 *Spectroscopic measurements*

222 A ThermoNicolet 380 spectrometer equipped with a liquid-nitrogen cooled MCT
223 (Mercury – Cadmium – Telluride) detector was used to perform the Fourier Transform
224 Infrared (FT-IR) analysis of soil and suspended sediment. Spectra were obtained using the
225 diffuse reflectance (DRIFTS) measurement technique. The spectra scan range was 4000 –650
226 cm^{-1} at a resolution of 2 cm^{-1} with 32 co-added scans per spectrum. Results were then
227 compiled using the OMNIC© (version 7.3) software provided by the spectrometer
228 manufacturer (ThermoNicolet, USA). This software facilitated measurement of the peak areas
229 that were relevant to determine the DRIFTS signatures.

230

231 *2.4. Statistical analyses and sediment tracing*

232 *Conventional mixing model*

233 The different sediment source types were characterised by their mean concentration
234 and by the standard deviation of each of the 29 radionuclide and geochemical properties
235 measured in the samples. The ability of the 29 potential fingerprinting properties to
236 discriminate between the potential sediment sources was investigated by conducting a
237 Kruskal-Wallis *H*-test as initially proposed by Collins and Walling (2002). Based on the set of
238 discriminating properties retained, an optimum ‘composite fingerprint’ was identified by
239 performing a stepwise selection procedure. This procedure consisted in minimising Wilk’s
240 lambda, as suggested by Collins and Walling (2002). Then, we constructed a Monte Carlo
241 mixing model to quantify the range of contribution of each sediment source to the sediment
242 samples collected at the different stations. Details on this model can be found in Evrard et al.
243 (2011).

244

245 *DRIFTS-PLS model for source determination*

246 In each subcatchment, two main types of potential sediment sources were considered
247 (see the land use classes mentioned in Table 2). Two to six soil samples of each source were
248 mixed in equal proportions to constitute a unique reference sample for the corresponding
249 source type. In subsequent steps, those reference samples were mixed in various weight
250 proportions. A DRIFTS spectrum was obtained for each mixture. The number of mixtures
251 prepared in each case varied between 20 and 30. This choice was dictated by (i) the previous
252 experiments showing that the chemometric models can be built based on the analyses of 20 to
253 30 mixtures (Poulenard et al., 2009 ; Poulenard et al., in press) and (ii) the willingness not to
254 over complicate the procedure when applying the DRIFTS-PLS method to several
255 subcatchments. Relationships between DRIFTS spectra ('x' variate) and the corresponding
256 weight contribution of the sediment source datasets ('y' variate) were analysed using Partial
257 Least Square (PLS) analyses. Approximately 75% of samples were used to develop the
258 calibration models, whereas the remaining 25% were used for validation. In order to get rid of
259 the small differences due to uncontrolled sources of variation, data pre-processing methods
260 were applied to the spectra, such as baseline correction, Savitzky–Golay smoothing, mean
261 centering, variable scaling, multiplicative signal correction (MSC) and standard normal
262 variate (SNV). The procedure followed by Poulenard et al. (2009; in press) was used to
263 determine the number of components providing the best compromise between the description
264 of the calibration set and the model predictive power, i.e. the lowest predictive standard error
265 (PRESS).

266 The predictive performance of the models was evaluated by calculating several
267 standard indicators such as the root-mean-square error of calibration (RMSEC), the root-
268 mean-square error of cross-validation (RMSECV), the root-mean-square error of prediction
269 (RMSEP), and coefficient of determination R^2 of predicted values against reference data.
270 Model validation was performed by both cross-validation and validation. RMSECV and
271 RMSEP values provided the average uncertainty that can be expected for predictions of future
272 samples. This uncertainty is only associated with the use of PLS models. Additional
273 uncertainty associated with the application of this model to suspended sediment could not be
274 taken into account.

275 Two independent PLS models were constructed to estimate the proportion of sediment
276 originating from the main potential sources in each subcatchment. DRIFTS spectra of
277 suspended sediment were then introduced into these PLS models to estimate the contribution
278 of each sediment source and the associated uncertainty (Poulenard et al., in press).

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3. Results

3.1. Quantifying the contribution of sources delivering sediment to the river

Overall, soils of the catchment are characterized by very low concentrations in geochemical elements and carbonates (< 0.01 %) (Table 2). These low levels reflect the strongly weathered character of soils in this highland volcanic region of central Mexico. This strong alteration is confirmed by the low content of soils in elements such as K, C and N in degraded soils and gullies (Table 2) as already reported by Bravo-Espinoza et al. (2009). Furthermore, the bulk of C content in soils is mainly under organic form as previously observed in the same study area (Cavoleda et al, 2011). In this context and to provide a strong physiochemical basis to our conventional fingerprinting exercise, we decided to restrict our fingerprint property selection to the fallout radionuclides and the biogenic elements. Fallout radionuclides such as ^{137}Cs and $^{210}\text{Pb}_{\text{xs}}$ can indeed provide a powerful tracer to discriminate between surface and subsurface (i.e., gully) sources (Wallbrink and Murray, 1993). Biogenic elements (e.g., C, N) can also provide this information, provided they are used to quantify the sources of suspended sediment to ensure that the conservation of those properties is achieved during the rapid transfer of particles to and within the river (Collins and Walling, 2002).

Conservation of particle grain size during their transfer between the sources and the river constitutes another prerequisite of the fingerprinting method to verify. Particle size was constant in 2009 at the outlet of each subcatchment, as demonstrated by the median particle size (d50) of sediment collected in Huertitas ($10\pm 1 \mu\text{m}$), Potrerillos ($12\pm 4 \mu\text{m}$) and La Cortina ($26\pm 6 \mu\text{m}$). Furthermore, those values are very close to the median sizes of particles measured in the soil samples (Tables 2 and 3).

Conventional fingerprint properties retained to discriminate sediment sources in the three subcatchments are provided in Table 4. Performance of predictive models used for the DRIFTS approach are given in Table 5. The PLS model, based on soil mixture analyses, was robust despite the limited number of samples analysed (Table 5). The correlations between actual and predicted proportions are excellent with R^2 close to 1 for all the models. The RMSEP (average difference between prediction and actual value on the set of calibration – i.e., not used to build the model) remains close to 10%. This is acceptable given that our study only aimed at obtaining an order of magnitude of the contribution of different sources delivering sediment to the river.

314 *Huertitas*

315

316 This subcatchment exported large quantities of sediment ($900 - 1500 \text{ t km}^{-2} \text{ y}^{-1}$;
317 Duvert et al., 2010). Active gully networks provided a constant source of sediment. Our
318 fingerprinting analyses showed that sediment collected at the outlet during the 2009 rainy
319 season was mostly supplied by the extended gully network of this subcatchment. This
320 contribution varied between $72 \pm 10\%$ and $100 \pm 12\%$ according to the model based on
321 spectroscopic properties, and between $88 - 98\%$ according to the conventional mixing model
322 (Figure 2). The low content in %C (about 1%) and in %N (about 0.08%) of sediment during
323 floods illustrates that most of the sediment originated from gully soils poor in C and N.
324 Sediment supply by cropland slightly decreased throughout the rainy season, which probably
325 reflects the effect of vegetation growth that protected the soil against rainfall splash effect and
326 erosion. During the most erosive storms (e.g., on 3 September), sediment was almost only
327 supplied by gullies. Results provided by both spectroscopic and “conventional” fingerprinting
328 techniques are consistent.

329

330 *La Cortina*

331

332 This subcatchment exported low quantities of sediment (ca. $30 \text{ t km}^{-2} \text{ y}^{-1}$; Duvert et al.,
333 2010). An attempt was made to differentiate the contribution of different vegetation types
334 based on their stable carbon isotopic signature but we could not outline any different
335 contribution of maize and avocado fields. However, test analyses showed that $\delta^{13}\text{C}$ value in
336 river sediment ($-22.22 \pm 0.15\text{‰}$) was close to cropland value (between $-22.31 \pm 0.10\text{‰}$ and
337 $-21.86 \pm 0.10\text{‰}$) and significantly different from the woodland value ($-26.60 \pm 0.10\text{‰}$). DRIFT-
338 PLS model showed that 70 to $80 \pm 20\%$ of the suspended sediment was provided by cropland
339 without significant differences all throughout the season (Figure 3). The mixing model based
340 on conventional fingerprinting properties similarly showed that $50 - 85\%$ of sediment was
341 supplied from cropland area. This result is consistent with the mean content in carbon (6.5 %) and
342 nitrogen (0.4 %) of sediment during flood that is very close to the mean content measured
343 in soils (%C = 6.31 and %N = 0.43) (Table 2). Sediment export was higher during the first
344 months of the rainy season because vegetation growth has progressively protected the soil
345 against erosion and because the first storm of the rainy season flushed the sediment stock
346 accumulated on the riverbed as demonstrated by Evrard et al. (2010).

347

348 *Potrerosillos*

349

350 Sediment source contributions varied strongly throughout the rainy season. This
351 subcatchment was characterized by large sediment exports ($600 - 800 \text{ t km}^{-2} \text{ y}^{-1}$; Duvert et al.,
352 2010). A rapid succession of several storms was observed in this catchment in 2009 that was
353 characterized by a strong reactivity and a “sawtooth behaviour”. Sediment was delivered by
354 both gullies and rangeland, in variable proportions (Figure 4).

355 Results provided by both fingerprinting techniques differ, mainly for 3 to 4 events
356 (Figure 4). Conventional fingerprinting showed a strongly variable supply of sediment by
357 gullies (5–86%) and rangeland (14–95%) all throughout the season. A very variable sediment
358 supply by gullies (36–97%) was also outlined by the DRIFTS approach. This bias is partially
359 explained by the relationships observed between the contribution of the topsoils in rangeland
360 determined by DRIFTS-PLS model and the organic carbon content of suspended sediment
361 (Figure 5). The mid-infrared signature and thereby the results of DRIFTS-PLS were clearly
362 influenced by the organic carbon content originating from the topsoil horizons. The DRIFTS-
363 PLS model provided in this case discrimination between the relative contribution of topsoils
364 (relatively rich in organic matter; Table 2) and the deep horizons of gullies depleted in organic
365 matter. During the first flood of the season, river sediment sample was characterized by a very
366 high C content (2% vs. 0.3–0.8% during the rest of the season; Fig. 5). This is probably due to
367 the export of cow dung stored on the soil surface by the first heavy storm of the season. This
368 indicates that, at the beginning of the rainy season, an important stock of sediment and
369 organic matter can be easily mobilized. This stock is probably accumulated during the dry
370 season due to the combined effect of cattle grazing and trampling of the soil.

371

372 *3.2. Origin of sediment along the river network*

373

374 Overall, concentrations in geochemical elements and activities in radionuclides are
375 very low in all the riverbed sediment collected along the river network of the entire catchment
376 (data not shown). Various ratios between geochemical elements and radionuclides were
377 calculated but they did not provide a way to outline specific sediment contributions within the
378 catchment. However, farming practices such as application of fertilizers and pesticides can
379 affect the composition of agricultural soils and sediment (Bravo-Espinoza et al., 2009).
380 Phosphate fertilizers contain indeed 10 – 200 times more U than soils, whereas they have a
381 lower Th content than soils (Takeda et al., 2004). The U/Th ratio can therefore provide a way
382 to outline the supply of sediment by agricultural soils along the river network. Figure 6
383 illustrates that U/Th ratio gradually increased from headwaters to the Cointzio reservoir. We

384 hypothesize that this observation reflects the increase in sediment supply by agricultural areas
385 along the river network. This hypothesis is consistent with the more detailed fingerprinting
386 results available for the three subcatchments (Fig. 6): (i) U/Th ratio is high in sediment
387 collected at the outlet of La Cortina where the bulk of material was supplied by cropland; (ii)
388 U/Th ratio was lower at the outlet of Potrerillos where a mixed contribution of gullies and
389 rangeland was outlined and (iii) U/Th ratio was the lowest at the outlet of Huertitas where
390 sediment was almost exclusively provided by the gully network.

391

392 *3.3. Origin of sediment at the catchment outlet*

393

394 Based on the previous work conducted in the subcatchments, we wanted to outline the
395 origin of suspended sediment collected at the river station draining the entire catchment and
396 located just upstream of Cointzio reservoir. We chose to discriminate the sediment delivered
397 by Acrisols (i.e., dominant type in Huertitas and Potrerillos) from sediment supplied by
398 Andisols (i.e., dominant type in La Cortina). Reference Acrisols were taken from Huertitas
399 gullies, whereas reference Andisols consisted of cropland soil (i.e., corn and avocado fields)
400 from La Cortina. Infrared spectrum of Acrisols was characterized by the dominance of
401 kaolinite in the clay fraction with three classical bands in the 3600–3700 cm⁻¹ area. In
402 contrast, Andisol spectrum was associated with gibbsite characteristic bands (Figure 7).

403 When compared to those source spectra, the infrared spectra of suspended sediment
404 collected at Santiago Undameo station were very similar to the Acrisol spectra. The PLS-
405 DRIFTS model confirmed the dominance of the contribution of Acrisols delivering more than
406 70% of the suspended sediment conveyed by the river at that location. This proportion can
407 even reach 90% during most of the rainy season during which 99% of sediment are exported
408 to the lake (Figure 8). In contrast, during the dry season, the PLS-DRIFT model indicates that
409 the sediment delivery from cultivated Andisols is higher and can reach up to 30% of total
410 sediment, but this sediment corresponds to only 1% of the annual export from the entire
411 catchment, which is negligible. Furthermore, those low water periods also coincide with the
412 highest organic carbon concentrations measured in river sediment (Figure 8). This carbon is
413 likely to originate from local villages that are not equipped with sanitation systems. In
414 contrast, during high flow periods, river sediment is characterised by very low carbon
415 contents, which is consistent with a dominant sediment supply by Huertitas or Potrerillos-like
416 gully soils (Table 2). Apparent increase of sediment supply by cultivated Andisols to the river

417 could then reflect a bias of the DRIFT-PLS approach induced by this strong increase in the
418 organic carbon content of sediment during the dry season.

419 420 **4. Discussion**

421 422 *4.1 Sediment tracing methods*

423
424 The low concentrations in geochemical elements made it difficult to outline significant
425 composition differences between different types of sources (e.g., soil types). In contrast, low
426 activities in fallout radionuclides provided an efficient way to discriminate between different
427 types of sources (typically gullies vs. cropland surface sheet erosion) when low-background
428 and efficient gamma spectrometry detectors are available to conduct measurements. The
429 results obtained in this study confirmed the preliminary observations made by Evrard et al.
430 (2010) on potential sediment sources based on the Cs-137 activities in soils and sediment.

431 The DRIFT-PLS method provided results that are very consistent with the
432 conventional geochemical approach in the context where minerals provide a dominant signal
433 to the soil. In these contexts, with very distinct mineralogy, a simple qualitative comparison of
434 infrared spectra provided a fast way to identify the dominant sediment sources. The
435 dependence of the infrared method to the soil content in organic matter has facilitated
436 sediment source apportionment in Potrerillos catchment, where it outlined the relative
437 important contribution of surface (cropland) and depth (gullies) material, which was
438 consistent with the carbon content of exported sediment. However, during the low stage
439 period, the introduction of one source of soluble organic matter delivered by anthropogenic
440 activities that is likely to have sorbed onto suspended sediment led to an overestimation of the
441 contribution of surface soil to river sediment, given that they were the only sources included
442 into the model and containing a large organic carbon content.

443

444

445 *4.2 Soil conservation in highland tropical catchments of central Mexico*

446

447 Our results have clear and significant management implications to control erosion and
448 reservoir siltation in this Mexican catchment. It is important to highlight that high turbidity
449 levels are observed at a regional scale in lakes and reservoirs of the Mexican central plateau
450 (Merino-Ibarra et al., 2007; Bravo-Inclan et al., 2008 ; Severo et al., 2002). However, none of
451 them attenuates light penetration to such a degree as in Cointzio reservoir, where Secchi disk
452 depths rarely exceed 0.2m (Susperregui et al., 2009). Our results showed that gullies

453 developed in Acrisols provide the bulk of fine particles to the reservoir, even if they occupy
454 less than 0.5% of the catchment area (Mendoza and Lopez, 2007). In this context, it is clear
455 that mitigation efforts concentrated in areas sensitive to soil erosion could lead to a rapid
456 improvement of water quality in the catchment and, more importantly, in the Cointzio
457 reservoir.

458 In future, the expected decrease in rain and the increase in temperature should result in
459 an increase in aridity and in surface runoff (Gratiot et al., 2010), which will certainly
460 complicate the implementation of soil conservation measures already tested in the area such
461 as the introduction of a crop rotation and the use of a crop residue cover on the soil (Bravo-
462 Espinoza et al, 2009). In this context, it is urgent to test some complementary mitigating
463 strategies to stabilize specific gullies and to evaluate their effectiveness, before generalizing
464 their installation across the entire Cointzio catchment (Martinez-Palacios et al., 2011).

465

466 **5. Conclusions**

467 Land degradation is intense in tropical regions, such as in volcanic highlands of central
468 Mexico. Two fingerprinting approaches were conducted to outline the main sources
469 delivering sediment leading to the siltation of Cointzio reservoir draining a 630-km²
470 catchment. This study was conducted in three subcatchments (3 – 12 km²) representative of
471 the different environments observed in this area. Both fingerprinting methods provided
472 similar results in Huertitas catchment, covered with Acrisols, where sediment was almost
473 exclusively delivered by gullies. In La Cortina, characterized by Andisols, sediment was
474 supplied by cropland. However, results provided by both methods in Potrerillos, covered with
475 a mix of Andisols and Acrisols, strongly differed. Furthermore, massive export of sediment
476 rich in organic matter produced by cattle activity and trampling during the dry season by the
477 first heavy storm of the year is suspected in this subcatchment. This study thereby outlined
478 several difficulties encountered when conducting fingerprinting studies in this type of
479 volcanic catchments. First, soils are very altered in this region, complicating the choice of
480 relevant geochemical fingerprint properties. Second, the DRIFTS-PLS method proved to be
481 very sensitive to the soil content in organic matter. Nevertheless, in a second step, our study
482 could demonstrate the dominant contribution of Acrisols to the sediment delivered from the
483 entire 630-km² catchment to the Cointzio reservoir. Soil conservation measures should
484 therefore focus on stabilizing gully networks as the ones observed in Huertitas catchment as
485 well as on implementing alternative farming practices in Potrerillos-like areas. In the future,
486

487 both fingerprinting methods could usefully be applied to trace sediment in different
488 environments, and solutions to decrease the potential DRIFTS-PLS method sensitivity to soil
489 organic matter content should be investigated.

490

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492

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496 Restoration) project funded by European Union. The authors are also very grateful to Dr.
497 Christine Hatté for conducting the test $\delta^{13}\text{C}$ measurements.

498

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657 **Figure captions**

658

659 Figure 1. Location of the monitored subcatchments (Huertitas, Potrerillos, La Cortina) and the
 660 Santiago Undameo station within the Cointzio catchment in the tropical highlands of central
 661 Mexico. Areas characterized by different soil types are also delineated, and locations where
 662 composite soil samples were collected are indicated. This study focused on the samples
 663 collected within the boundaries of the monitored subcatchments.

664

665 Figure 2. Sediment sources in Huertitas catchment as defined by conventional and alternative
 666 fingerprinting techniques. Please note that, because of logistical problems to conduct the
 667 sample irradiations, results derived from the conventional mixing model are only available for
 668 the five first composite river sediment samples of the season.

669

670 Figure 3. Sediment sources in La Cortina catchment as defined by conventional and
 671 alternative fingerprinting techniques.

672

673 Figure 4. Sediment sources in Potrerillos catchment as defined by conventional and
674 alternative fingerprinting techniques.

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676 Figure 5. Relationship between contribution of rangeland topsoil and suspended sediment
677 organic carbon content at the outlet of Potrerillos subcatchment. Carbon content of sediment
678 collected during the 1/7/2009 flood constitutes an outlier (probably explained by the massive
679 export of cow dung stored on the soil surface by the first heavy storm of the season).

680

681 Figure 6. Values of U/Th ratio measured in riverbed sediment samples collected along the
682 river network within the entire catchment (U corresponds to activities in Ra-226, and Th to
683 activities in Ra-228).

684

685 Figure 7. Typical spectra of Andisols, Acrisols and suspended sediment collected at Santiago
686 Undameo station.

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688 Figure 8. Contribution of Acrisols to sediment monitored at Santiago Undameo station.
689 Discharge values correspond to mean discharges measured during the sediment collection
690 period by the trap.

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700 **Tables**

701

702 Table 1

703 Altitude, soil, slope, and erosion characteristics of the study sites

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705 Subcatchment	705 Area (km ²)	705 Altitude range (m)	705 Soil types ^a	705 Severely eroded areas ^b
				706 (% of the catchment surface)

707

708 Huertitas	3	2150 – 2450	Acrisols (100%)	6
709 La Cortina	9	2250 – 2700	Andisols (100%)	0
710 Potrerillos	12	2200 – 2700	Acrisols (60%)	1
			Andisols (40%)	
712 Undameo	630	2000 – 3440	Andisols (37%)	
			Luvisols	0.5
			Acrisols (33%)	

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716 ^a According to FAO (2006).717 ^b Derived from the analysis of aerial photographs.

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Table 2
Geochemical (g kg⁻¹), radionuclide (Bq kg⁻¹), organic matter (%) and grain size properties of the sediment source samples.

Huertitas		n	K	Na	As	Th	Yb	Hf	Fe	Tb	Sc	Ta	Cs	Co	Eu	xs-Pb-210	Pb-210	Th-234	Ra-226	Ra-228	Th-228	Cs-137	%C	%N	%C (CaCO3)	% <63µm	d50 (µm)
Gullies	mean	6	0.0	0.0	2.3	6.2	2.5	6.2	6.8	0.5	23.7	1.0	0.9	20.7	0.9	0.0	17.0	23.4	23.7	24.2	24.2	0.0	0.34	0.03	< 0,01	95	12
	variance		0.0	0.0	1.5	1.9	0.2	1.1	0.1	0.1	1.5	0.5	0.8	9.9	0.0	0.0	20.0	23.3	31.3	38.1	34.6	0.0	0.28	0.03		1	2
Cropland	mean	3	0.4	0.2	2.0	7.1	2.9	7.1	6.4	0.9	20.0	0.8	1.8	30.1	1.0	8.0	34.0	32.5	26.1	27.2	27.7	1.3	2.73	0.21	< 0,01	88	17
	variance		0.0	0.0	5.7	3.9	0.0	0.4	0.1	0.0	0.3	0.8	4.3	43.2	0.0	5.1	63.2	19.3	36.7	48.1	65.8	0.9	0.75	0.06		3	2
La Cortina																											
Cropland	Mean	2	0.1	0.1	6.4	9.4	3.0	8.7	7.2	1.0	22.3	2.1	5.0	21.5	1.4	16.2	49.8	38.1	33.7	36.8	37.4	3.7	6.31	0.43	0.02	89	19
	Variance		0.0	0.0	1.1	3.7	0.1	1.3	0.2	0.0	0.2	0.9	0.2	16.2	0.1	5.3	6.6	14.0	23.9	10.8	10.8	0.3	0.14	0.03	0.01	11	10
Woodland	Mean	2	0.2	0.1	5.5	9	2	7.7	7.2	1.2	23.7	1.3	5.1	25	1.6	81.0	37.1	37.8	32.8	36.1	36.8	6.6	9.94	0.50	0.01	81	26
	Variance		0.0	0.0	0.3	0.4	0.1	0.4	0.4	0.1	1.2	0.1	0.3	1.3	0.1	62.0	1.9	1.9	1.6	1.8	1.8	0.3	5.6	0.1	0.01	6	2
Potrerosillos																											
Gullies	Mean	2	0.7	0.9	0.0	8.0	2.4	8.2	6.2	1.6	17.3	1.8	4.5	29.2	1.3	0.0	17.2	32.0	23.1	32.2	32.8	1.3	0.13	0.01	< 0,01	83	25
	Variance		0.0	0.1	0.0	0.0	0.0	1.0	0.1	0.4	1.6	0.0	6.8	15.4	0.0	0.0	4.0	6.3	8.5	1.9	3.5	2.1	0.12	0.02		11	8
Cropland	Mean	2	0.4	0.4	3.2	6.8	2.5	6.0	6.1	0.5	17.3	2.2	1.6	27.0	0.9	7.0	30.0	33.7	23.8	31.6	32.1	1.4	1.43	0.11	< 0,01	82	22
	Variance		0.0	0.0	0.3	0.0	0.0	0.1	2.8	0.4	3.3	0.4	3.4	37.0	0.0	10.0	53.3	3.2	13.2	0.1	0.3	0.9	0.74	0.07		11	12

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Table 3
Geochemical (g kg⁻¹), radionuclide (Bq kg⁻¹), organic matter (%) and grain size properties of the river sediment samples.

Huertitas	Date	K	Na	As	Th	Yb	Hf	Fe	Tb	Sc	Ta	Cs	Co	Eu	xs-Pb-210	Pb-210	Th-234	Ra-226	Ra-228	Th-228	Cs-137	%C	%N	% <63µm	d50 (µm)
	26/06/09	0.2	0.1	2.8	6	2	6.0	6.1	0.0	20.1	0.0	3.3	18	0.7	15.0	34.1	31.4	19.3	29.2	26.4	0.5	1.2	0.08	97	10
	12/07/09	0.2	0.1	3.3	7	3	6.9	6.3	0.0	21.5	1.3	0.0	19	0.9	11.0	33.5	32.3	22.1	28.3	27.5	0.5	1.1	0.08	99	9
	25/07/09	0.1	0.1	0.0	5	2	7.1	6.3	0.0	19.4	0.0	0.0	21	0.6	13.0	34	26.2	21.0	26	27	0.0	1.2	0.08	92	11
	25/07/09	0.2	0.1	2.5	6	3	6.1	6.6	0.7	21.5	1.0	2.4	22	0.8	14.0	34	29.6	20.0	26	27	0.0	1.2	0.08	96	10
	04/08/09	0.2	0.1	3.3	6	2	6.4	6.5	0.0	20.4	0.0	2.4	24	0.6	10.0	25.6	23.8	15.5	24.5	22.9	0.4	0.9	0.05	91	11
La Cortina	Date	K	Na	As	Th	Yb	Hf	Fe	Tb	Sc	Ta	Cs	Co	Eu	xs-Pb-210	Pb-210	Th-234	Ra-226	Ra-228	Th-228	Cs-137	%C	%N	% <63µm	d50 (µm)
	14/07/09	0.3	0.2	3.8	7	3	6.2	6.5	2.3	23.8	1.5	4.2	23	1.9	40.0	65.3	38.1	25.8	29.4	30.9	2.8	7.1	0.48	88	18
	25/07/09	0.2	0.1	4.2	6	3	5.6	6.3	1.0	22.8	1.2	2.6	22	1.4	50.0	68.0	23.1	18.6	21.3	25.4	2.5	6.9	0.45	n/a	n/a
	06/08/09	0.2	0.1	3.4	6	3	5.6	6.5	0.8	21.6	1.1	2.1	25	1.1	24.0	42.0	28.4	17.6	23.1	24.1	2.4	5.4	0.32	76	29
	14/08/09	0.2	0.2	3.3	6	3	5.7	6.4	0.7	22.1	0.0	2.5	22	1.4	48.0	61.2	22.9	13.0	25.5	24.9	2.0	6.1	0.39	81	29
	26/08/09	0.2	0.1	0.0	6	3	6.0	6.4	0.9	21.5	0.0	2.6	23	1.1	39.0	59.3	34.0	20.5	27.8	28.6	2.0	7.1	0.42	75	30
Potrerillos	Date	K	Na	As	Th	Yb	Hf	Fe	Tb	Sc	Ta	Cs	Co	Eu	xs-Pb-210	Pb-210	Th-234	Ra-226	Ra-228	Th-228	Cs-137	%C	%N	% <63µm	d50 (µm)
	01/07/09	0.6	0.4	0.0	9	5	8.1	6.4	1.4	22.6	1.6	3.5	28	1.9	5.0	29.7	38.3	24.7	37.8	36.4	0.5	2.0	0.12	n/a	n/a
	12/07/09	0.5	0.4	0.0	7	3	7.0	5.6	1.1	18.0	0.0	5.6	22	1.3	8.0	29.8	32.1	21.7	35.8	35.4	0.0	0.4	0.02	97	10
	15/07/09	0.6	0.3	2.8	8	3	6.8	6.2	0.0	20.6	0.0	4.2	25	1.4	1.0	25.5	34.9	24.5	32.4	32.2	0.3	0.8	0.05	95	10
	16/07/09	0.6	0.5	3.0	8	4	7.2	6.0	0.0	19.7	1.3	4.6	26	1.3	15.0	29.4	30.1	14.0	31.9	26.8	0.0	0.3	0.01	88	16
	17/07/09	0.6	0.6	3.3	6	3	6.5	5.7	0.0	16.8	0.0	5.6	29	1.2	6.0	19.8	27.4	13.6	27.7	24.3	0.3	0.4	0.02	n/a	n/a
	19/07/09	0.6	0.6	0.0	7	3	6.7	6.5	1.1	19.0	1.5	3.4	36	1.2	5.0	17.9	27.3	12.9	26.0	25.1	0.3	0.6	0.02	88	16
	26/07/09	0.6	0.5	3.3	8	3	5.9	5.6	0.7	17.8	1.3	6.2	22	1.1	14.0	28.0	29.0	15.0	33	33.0	0.0	0.4	0.02	99	7
	28/07/09	0.6	0.4	2.6	8	3	6.8	6.3	0.7	20.2	2.0	6.1	23	1.2	17.0	20.0	32.0	13.0	33	28.0	0.0	0.5	0.03	99	8
	18/08/09	0.6	0.7	0.0	7	3	6.8	6.6	0.9	18.4	1.5	4.9	41	1.1	1.0	26.8	33.5	24.7	31.9	33.1	0.2	0.5	0.02	87	16
	22/08/09	0.5	0.4	2.4	8	3	8.5	6.9	0.8	20.7	1.3	5.9	31	1.0	1.0	23.2	35.9	24.2	33.1	33.7	0.4	0.5	0.03	86	19

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776 Table 4
 777 Discriminant properties in the three study sites (Wilk's lambda and percentage of correctly classified samples)
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780	Fingerprint property	Wilk's lambda	Cumulative % of
781	added		samples classified
782			correctly
783	<hr/>		
784	(a) Huertitas subcatchment		
785	C	0.0192	96
786	xs-Pb-210	0.0096	98
787	Cs-137	0.0048	100
788	(b) La Cortina subcatchment		
789	xs-Pb-210	5.88 E-15	100
790	Cs-137	9.81 E-17	100
791	(c) Potrerillos subcatchment		
792	C	1.07 E-16	100
793	xs-Pb-210	2.18 E-17	100
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808 Table 5
 809 Predictive performance of the PLS (Partial Least Squares) models in the different Mexican subcatchments
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Sub-catchment	Number of mixture samples	Considered sources of sediments	R ²	RMSEC	RMSEP	RMSECV
Huertitas	20	Gullies	0.99	2.38	4.49	6.13
		Cropland	0.99	1.1	4.41	5.88
La Cortina	32	Forest Road	0.99	1.18	2.43	6.64
		Cropland	0.99	2.96	8.66	6.67
Potrerillos	22	Gullies	0.99	0.321	11.1	11.1
		Cropland	0.98	5.04	7.46	6.73
Undameo	22	Acrisols	0.99	4.08	2.24	13.2
		Andisols	0.99	5.21	3.41	13.6

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 812 R²: coefficient of determination; RMSEC: Root-Mean-Square Error of Calibration; RMSEP: Root-Mean-Square Error of Prediction; RMSECV:
 813 Root-Mean-Square Error of Cross-Validation.

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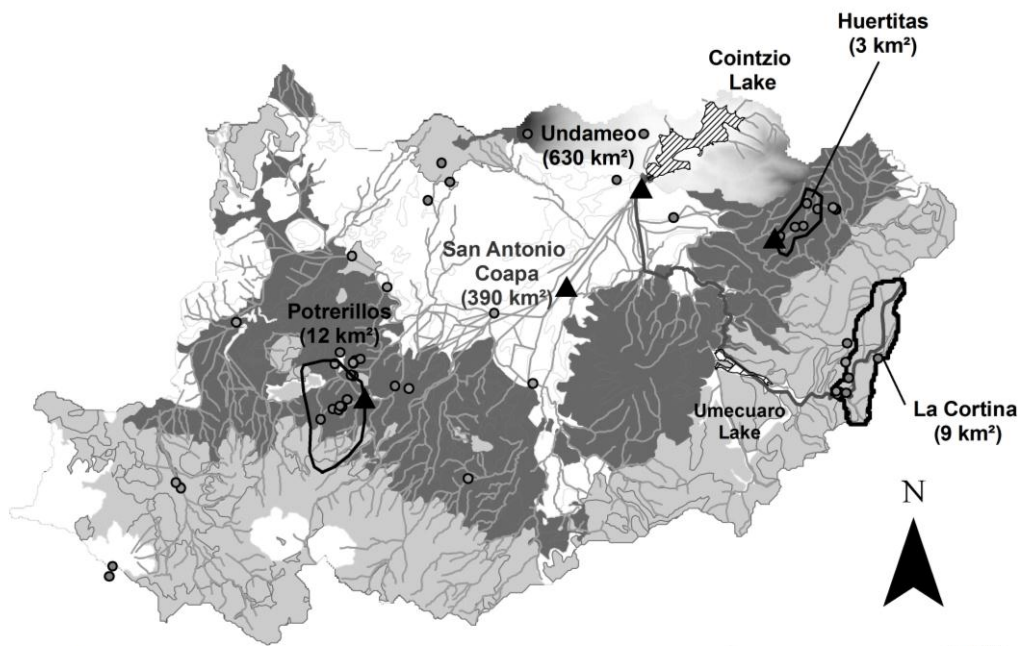
[S180] Tracing sediment sources in a tropical highland catchment of central Mexico by using conventional and alternative fingerprinting methods

Journal:	<i>Hydrological Processes</i>
Manuscript ID:	HYP-11-0762.R1
Wiley - Manuscript type:	Special Issue Paper
Date Submitted by the Author:	n/a
Complete List of Authors:	Evrard, Olivier; CEA, LSCE Poulenard, Jerome Nemery, Julien; G-INP, LTHE Ayrault, Sophie; CEA, LSCE Gratiot, Nicolas; IRD, LTHE Duvert, Clement; IRD, LTHE Prat, Christian; IRD, LTHE Lefèvre, Irène; CEA, LSCE Bonté, Philippe; CEA, LSCE Esteves, Michel; IRD, LTHE
Keywords:	sediment, fingerprinting, soil types, Mexico, tropical catchment

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Manuscripts

Review

Figure 1

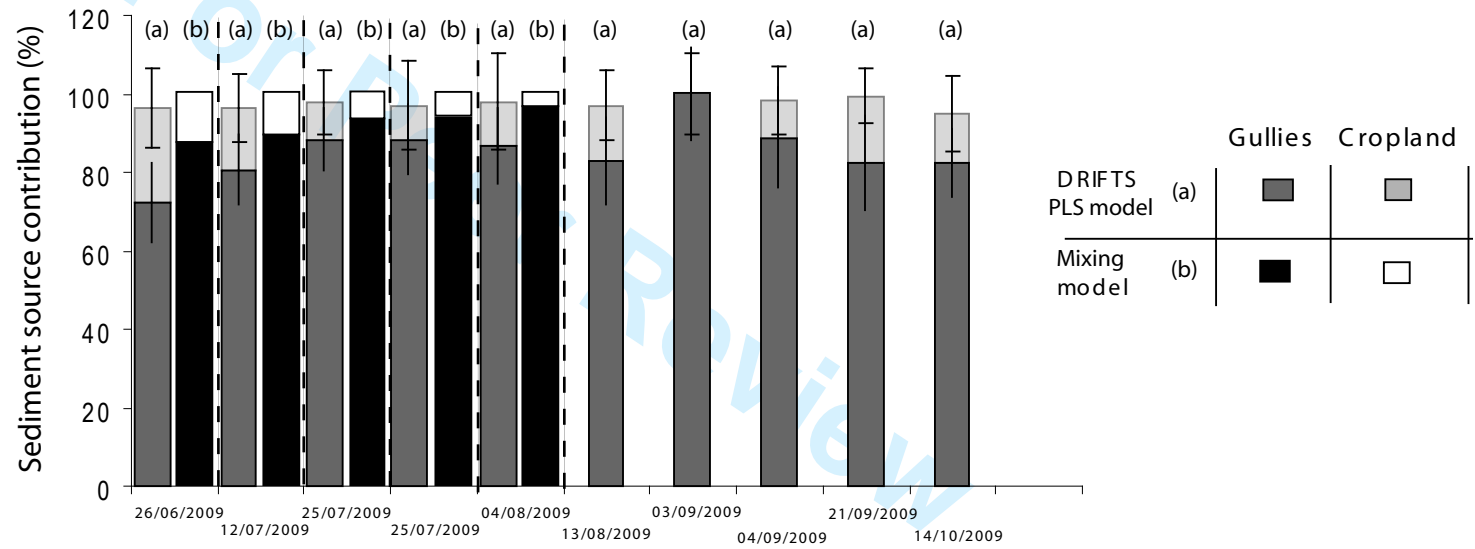


Legend

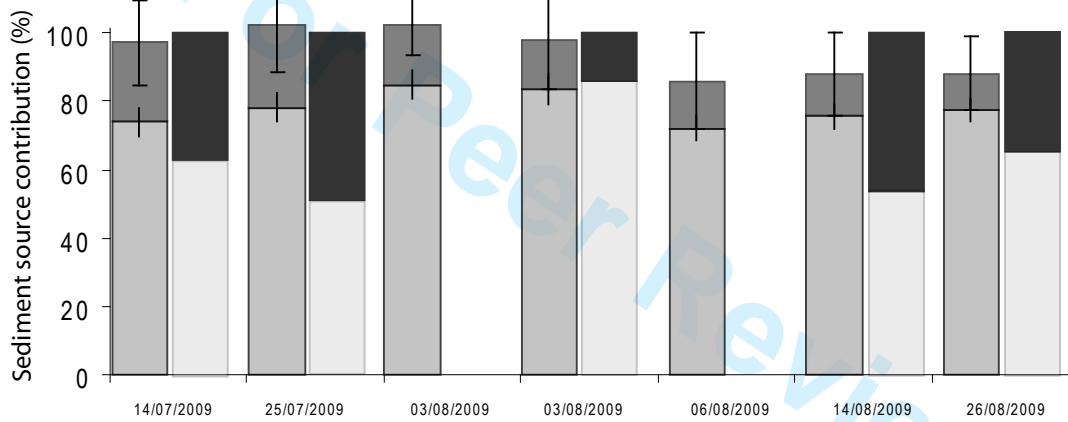
- ▲ River/rainfall monitoring station
- Composite soil samples
- Rio Grande de Morelia
- Other rivers
- ▭ Monitored subcatchment
- Soil types**
- Acrisols
- Andisols
- Other soil types





0 10 KM



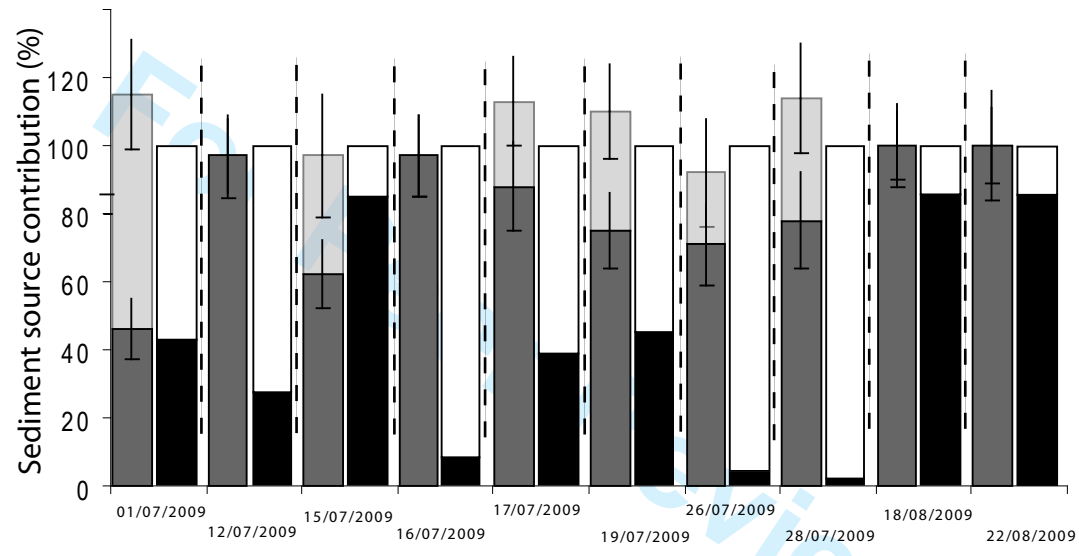


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		Cropland	Woodland
DRIFTS PLS model	(a)		
Mixing model	(b)		

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		Gullies	Rangeland
DRIFTS PLS model	(a)		
Mixing model	(b)		

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Figure 5.

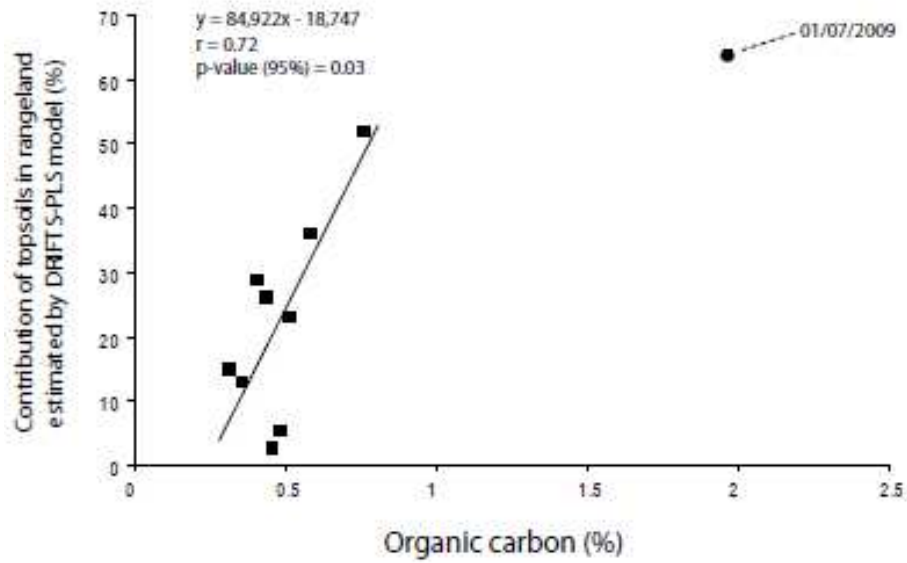
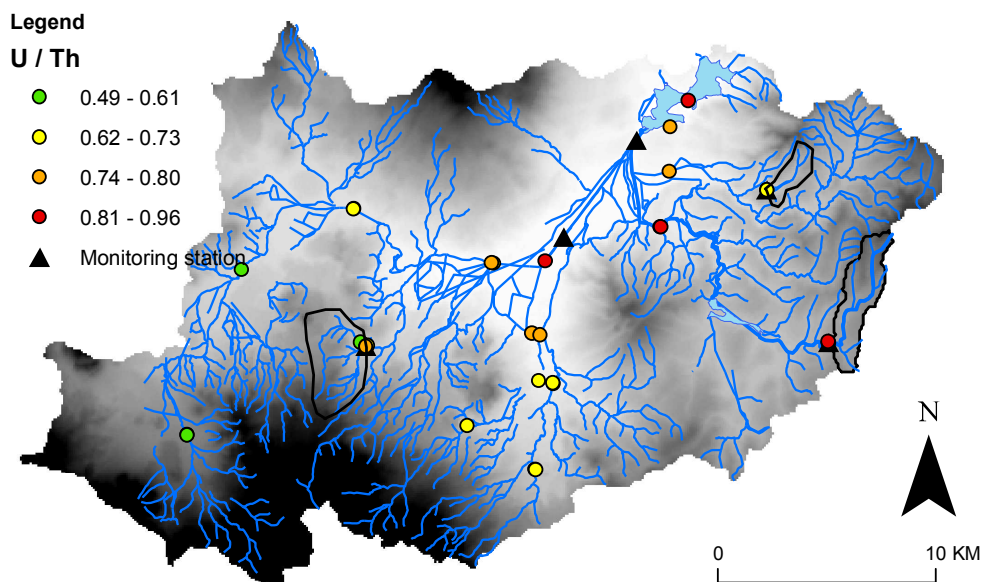


Figure 6. Values of U/Th ratio measured in riverbed sediment samples collected along the river network within the entire catchment (U corresponds to activities in Ra-226, and Th to activities in Ra-228).



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Figure 7.

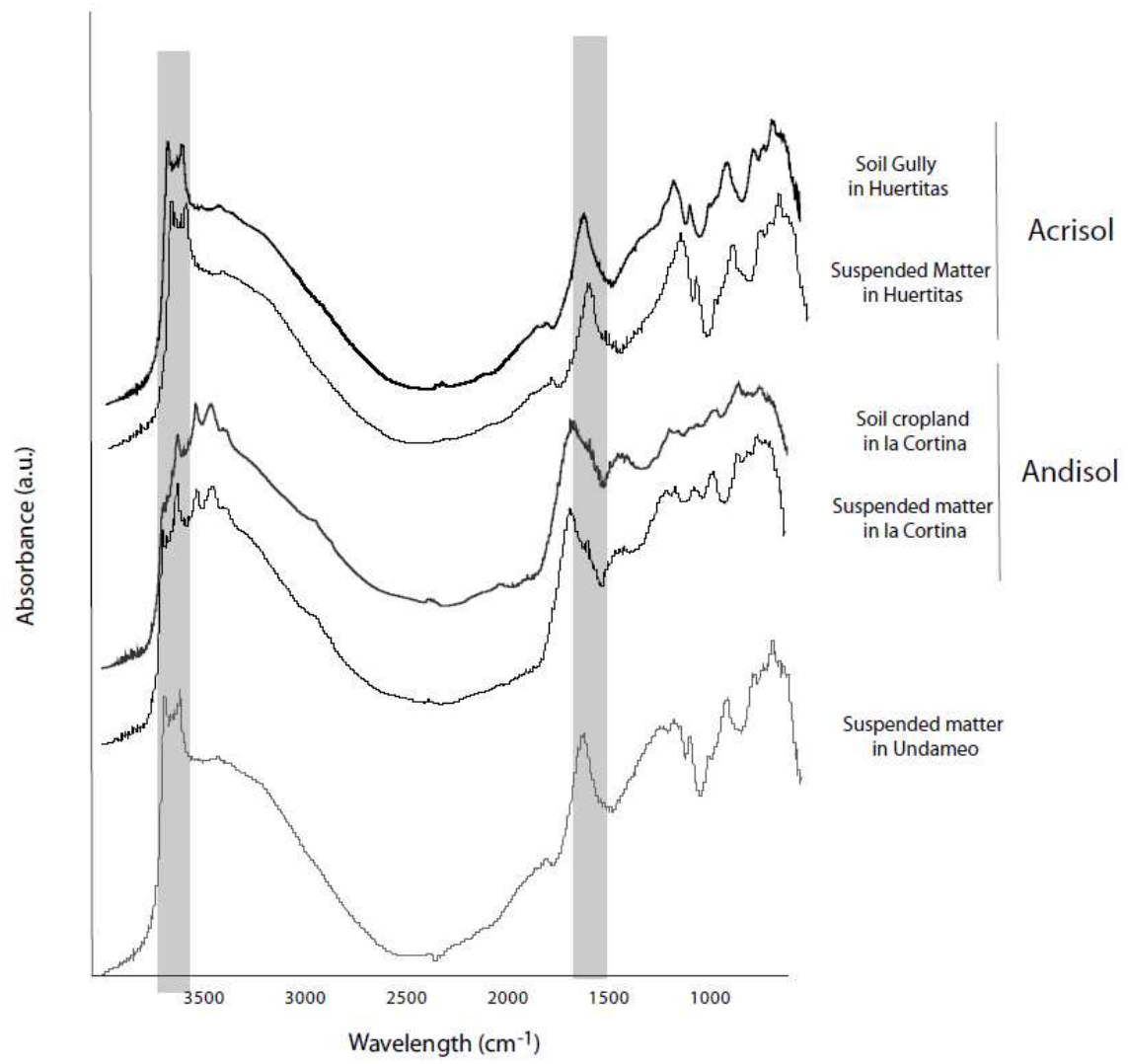


Figure 8.

