



## Luminescence of quartz and feldspar fingerprints provenance and correlates with the source area denudation in the Amazon River basin

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1 Luminescence of quartz and feldspar fingerprints provenance and correlates with the  
2 source area denudation in the Amazon River basin

3

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## Abstract

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The Amazon region hosts the world's largest watershed spanning from high elevation Andean terrains to lowland cratonic shield areas in tropical South America. This study explores variations in optically stimulated luminescence (OSL) and infrared stimulated luminescence (IRSL) signals in suspended silt and riverbed sands retrieved from major Amazon rivers. These rivers drain Pre-Cambrian to Cenozoic source rocks in areas with contrasting denudation rates.

In contrast to the previous studies, we do not observe an increase in the OSL sensitivity of quartz with transport distance; for example, Tapajós and Xingu Rivers show more sensitive quartz than Solimões and Madeira Rivers, even though the latter have a significantly larger catchment area and longer sediment transport distance. Interestingly, high sensitivity quartz is observed in rivers draining relatively stable Central Brazil and Guiana shield areas (denudation rate  $\xi = 0.04 \text{ mm.yr}^{-1}$ ), while low sensitivity quartz occurs in less stable Andean terrains ( $\xi = 0.24 \text{ mm.yr}^{-1}$ ). An apparent linear correlation between quartz OSL sensitivity and denudation rate suggests that OSL sensitivity may be used as a proxy for erosion rates in the Amazon basin. Furthermore, luminescence sensitivity measured in sand or silt arises from the same mineral components (quartz and feldspar) and clearly discriminates between Andean and shield sediments, avoiding the grain size bias in provenance analysis. These results have implications for using luminescence sensitivity as a proxy for Andean and shield contributions in the stratigraphic record, providing a new tool to reconstruct past drainage configurations within the Amazon basin.

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## 1. Introduction

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Large tropical rivers promote the transfer of rock weathering products to oceans (Latrubesse et al. 2005), representing an important component of the Earth surface system. The Amazon River is the world's largest river draining an area of  $6.15 \times 10^6 \text{ km}^2$ , with average annual water discharge of  $200,000 \text{ m}^3.\text{s}^{-1}$  (Meade, 1994). The tributaries of the Amazon River mainly drain the Guiana shield and Central Brazil shield, with the headwaters of some western Amazon rivers flowing through the Andes mountain belt. Gibbs (1967) estimated that 82% of

56 the suspended sediments transported by the modern Amazon River come from Andean zones  
57 representing only 12% of the Amazon River basin. According to Filizola and Guyot (2009),  
58 total suspended sediment yield from Andean areas is more than  $10^9$  t.yr<sup>-1</sup> while the maximum  
59 total suspended sediment yield of shield areas is only  $10^8$  t.yr<sup>-1</sup>. This decoupling between  
60 suspended sediment load and drainage area arises from contrasting erosion rates in catchments  
61 draining the Andes and the cratonic shield areas. This contrast is also observed for the  
62 production of total dissolved solids transported by Amazon rivers. Andean tributaries  
63 contribute with around 64% of total dissolved solids delivered by the Amazon River to the  
64 Atlantic Ocean (Moquet et al., 2016). According to Wittmann et al. (2010) erosion rates in the  
65 Andes (0.5 mm.yr<sup>-1</sup>) are one to two orders of magnitude higher than the erosion rates in Guiana  
66 and Central Brazil shields (0.01 mm.yr<sup>-1</sup>). Most of the studies regarding the origin and transport  
67 of sediments in the Amazon fluvial system focused on fine-grained suspended sediments  
68 (Meade, 1994; Guyot et al., 2007; Viers et al., 2008) or dissolved solids (Moquet et al., 2016)  
69 and little is known about the sources and transport of bedload sands and their coupling to  
70 suspended sediments on a basin-wide scale. Furthermore, the relative contribution of sediments  
71 transported by tributaries of the Solimões River, which has large Andean and lowland  
72 tributaries, is poorly constrained. Suspended and bedload sediments transported and stored  
73 within the Amazon River system support the development of flooding forest substrates and its  
74 specific biodiversity (McClain and Naiman, 2008). Sediments from the Amazon River  
75 accumulated in the equatorial Atlantic Ocean are also important archives to reconstruct past  
76 changes in Amazon precipitation (Govin et al., 2014) and landscape (Dobson et al., 2001).  
77 Thus, understanding sediment sources and transport-storage routes in the Amazon River system  
78 is critical to any reconstruction of past conditions of the Amazon climate and its fluvial system.

79         Diverse properties of terrigenous sediments like major elements geochemistry (e.g.  
80 Govin et al., 2012) and heavy minerals suites (e.g. Morton and Hallsworth, 1999) have been  
81 used in sediment provenance analysis. However, sediment provenance analysis based on  
82 elemental geochemistry can be influenced by grain size (Bouchez et al., 2011) while minor  
83 components like heavy minerals can promote bias toward source rock types rich in specific

84 heavy minerals more resistant to weathering processes (Morton and Hallsworth, 1999). Isotopic  
85 analysis such as neodymium and strontium isotopes (Viers et al., 2008), while being relatively  
86 robust, are rather expensive and not available in most laboratories. Thus, it becomes imperative  
87 to search for other innovative approaches to provenance analysis, which are robust, inexpensive  
88 and easily accessible. It has been demonstrated in the past that the optically stimulated  
89 luminescence (OSL) may be used for provenance fingerprinting (Sawakuchi et al., 2012; Lü et  
90 al., 2014). In particular, the OSL sensitivity (emission intensity per unit mass per unit radiation  
91 dose) of quartz grains may be related to the source and transport history (i.e. deposition-erosion  
92 cycles) of sediments (Preusser et al., 2006; Pietsch et al., 2008; Juyal et al., 2009; Sawakuchi  
93 et al., 2011; Gliganic et al., 2017). In this study, we use OSL signals of quartz and infrared  
94 stimulated luminescence (IRSL) signals of feldspar to characterize the sources of suspended  
95 and riverbed sediments in the major tributaries of the Amazon main stem, namely the Solimões,  
96 Negro, Madeira, Tapajós and Xingu Rivers. The sources of sediments transported by the  
97 Solimões River, which is named as Amazon River after meeting the Negro River, is  
98 distinguished by the analysis of sediments from its major tributaries, including the Içá, Japurá,  
99 Jutaí, Juruá, Tefé, Urucu (Coari) and Purus Rivers. This study has general implications for  
100 provenance reconstructions in tropical settings since quartz and feldspar, which dominate the  
101 luminescence signals of sediments, are major components of terrigenous sediments.

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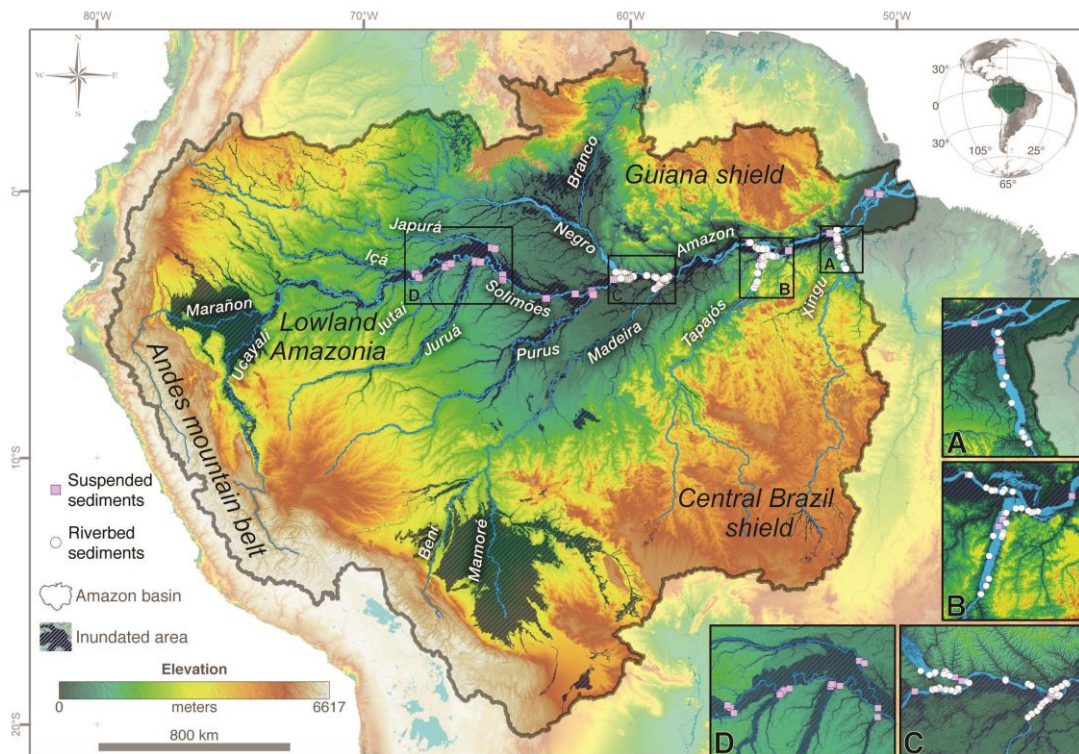
## 103 **2. The Amazon fluvial system**

104 The modern Amazon fluvial system drains the equator between about 5°N to 15°S,  
105 placing the Amazon basin in the wet tropics, a region defined by relatively high precipitation  
106 (>1,500 mm.yr<sup>-1</sup>) and temperature (>20°C) (Silva et al., 2011). Besides covering this large  
107 latitudinal range, the Amazon drainage basin extends longitudinally across 3,000 km,  
108 connecting the Andes mountain belt, Guiana shield and the Central Brazil shield with the  
109 equatorial Atlantic Ocean. This relief configuration and large latitudinal range induce high  
110 spatial variability of rainfall in the Amazon drainage basin (Espinoza Villar et al., 2009). After  
111 reaching the shore of the equatorial Atlantic Ocean, the Amazon River sediments are

112 transported northwestward by the North Brazil Current and are deposited on the northern South  
113 American continental margin giving rise to prominent features like the Guiana mud belt and  
114 the sub-aqueous Amazon delta (Nittrouer et al., 1995). The Amazon rivers drain regions with  
115 contrasting relief and erosion rates (Wittmann et al., 2010) as well as with variable rock types,  
116 including Pre-Cambrian igneous and metamorphic rocks in shield areas, Cenozoic  
117 metasedimentary and volcanic rocks in Andes and Paleozoic-Mesozoic sedimentary and  
118 igneous rocks in lowland Amazonia (Jaillard et al., 2000; Tassinari et al., 2000). This diverse  
119 geological scenario assigns different characteristics to the Amazon rivers, which are classified  
120 regarding the water type in white, black and clear water rivers (Sioli, 1984). White water rivers  
121 have high suspended load and dissolved solids, and neutral to alkaline waters; they are  
122 represented by rivers with headwaters draining the Andes mountains such as the Madeira,  
123 Solimões, and Içá Rivers (Figure 1). Black water rivers have low suspended load, high  
124 concentration of organic dissolved compounds and acidic waters; the Negro River draining the  
125 Guiana shield is the major Amazon black water river. Clear water rivers have low suspended  
126 load and acidic to slightly alkaline waters (Sioli, 1984). The Xingu and Tapajós Rivers, which  
127 drain the Central Brazil shield are the major Amazon clear water rivers.

128         The Solimões River, formed by the confluence of the Marañón and Ucayali Rivers, and  
129 the Madeira River, are the main Andean tributaries of the Amazon River; the upper reaches of  
130 these rivers drain the Andean highlands (> 3,000 m), while their lower reaches flow through  
131 Amazon lowlands (< 200 m). The Solimões and Madeira Rivers drain areas of ~2,150,000 km<sup>2</sup>  
132 and ~1,360,000 km<sup>2</sup>, respectively and contribute around 90% of the total suspended load  
133 transported by the Amazon River (Latrubesse et al., 2005). The Negro River drains an area of  
134 approximately 700,000 km<sup>2</sup>, mainly flowing through the Guiana shield on areas with elevation  
135 lower than 200 m until it reaches the Solimões River to form the Amazon River. The Tapajós  
136 and Xingu Rivers are the main tributaries of the Amazon River in eastern Amazon. The area of  
137 the Tapajós and Xingu drainage basins are around 500,000 km<sup>2</sup>, emplaced on medium altitude  
138 terrains (30-700 m) of the Central Brazil shield. The Negro, Tapajós and Xingu Rivers have

139 very low sediment yield compared to the Solimões and Madeira Rivers (Latrubesse et al.,  
140 2005).



141  
142 Figure 1. Sediment sampling sites in the Amazon fluvial system. Major  
143 geomorphological domains are represented by the Andes mountain belt, Guiana shield, Central  
144 Brazil shield and lowland Amazonia, which define areas with different elevations.

145

### 146 3. Methods

#### 147 3.1. Sediment sampling

148 Riverbed and suspended sediments were collected in downstream sectors of the  
149 Solimões, Negro, Madeira, Tapajós and Xingu Rivers and along the Amazon main stem.  
150 Additionally, suspended sediments samples were also collected in the downstream sectors of  
151 the major tributaries of the Solimões River, which included the Içá, Japurá, Jutai, Juruá, Tefé,  
152 Urucu-Coari and Purus Rivers, as well as in the Solimões-Amazon main stem between the  
153 mouths of the Içá and Xingu Rivers (Figure 1). The sampling surveys were carried out during  
154 periods of low water level in September of 2011 and October-November of 2015 and during a  
155 period of high water level in May of 2012. Riverbed sand samples were retrieved from bar tops

156 exposed during the low water level periods or using a grab sampler in underwater channel  
157 zones; these range from nine (Negro) to 25 (Amazon) samples collected per river. Suspended  
158 sediments were retrieved at 2/3 water column depth or at three different depths of 1 m, 2/3  
159 water column depth and 1-2 m above riverbed. Water collected with a submersible pump was  
160 filtered using acetate cellulose filters (0.2  $\mu\text{m}$ ) for concentration of sediments for inorganic  
161 geochemistry analysis (250 to 1000 ml of water per filter). Filters with suspended sediments  
162 were dried (40°C) immediately after water filtration. For luminescence measurements in  
163 suspended sediments, larger volumes of water (40-60 l) sampled during October-November of  
164 2015 were used to concentrate fine-grained sediments ( $> 0.45 \mu\text{m}$ ) using a membrane  
165 ultrafiltration system coupled to a peristaltic pump. Suspended sediments for inorganic  
166 geochemistry analysis were sampled in three sites per studied river during the low and high  
167 water level periods. The only exception was the Xingu River, with four sampling sites per water  
168 level period.

169         The catchment area upstream sampling sites and channel length from sampling sites to  
170 river springs were estimated using digital elevation models. These data allowed to evaluate  
171 variations in luminescence sensitivity along sediment transport routes and to test the hypothesis  
172 that sediment transport drives the sensitization of quartz in the Amazon fluvial system.

173

### 174 **3.2. Luminescence measurements**

175         The riverbed sand samples were wet sieved to isolate the 180–250  $\mu\text{m}$  grain size fraction,  
176 which is a grain-size fraction representing bed load sediments in the studied samples and it is  
177 suitable for luminescence measurements in multigrain aliquots. Lithium metatungstate solution  
178 (density 2.85  $\text{g}/\text{cm}^3$ ) was used to separate light (feldspar and quartz) from heavy minerals. Tests  
179 using hydrochloric acid (HCl 10%) showed the absence of carbonate minerals. Organic matter  
180 was eliminated using hydrogen peroxide ( $\text{H}_2\text{O}_2$ ). Thus, the preparation of polymineral sand  
181 grains only included HCl and  $\text{H}_2\text{O}_2$  treatments, without application of hydrofluoric (HF) acid  
182 etching. Three to six riverbed samples from each river were selected for preparation of pure  
183 quartz fraction through HF acid treatment (38% HF for 40 min) of the lighter fraction. Infrared



184 stimulation (IR) was performed to confirm the absence of feldspar contamination in the HF  
185 treated quartz fraction. Samples with remaining feldspar were subjected to steps of HF 5%  
186 etching for 24 hours followed by wet sieving (180  $\mu\text{m}$ ), with IR signal checked after each step,  
187 until complete elimination of feldspar grains.

188         The suspended sediment samples were centrifuged to eliminate water excess and increase  
189 sediment concentration. Grain size separation and chemical treatments were not performed with  
190 suspended sediment samples to avoid sediment loss. Grain size analyses were performed to  
191 characterize the silt fractions dominating the suspended sediment samples, using a Mastersizer  
192 2000 laser diffraction particle size analyzer (Malvern Instruments). Sediment samples were  
193 dispersed in deionized water for measurement. Grain size (0.1-1000  $\mu\text{m}$ ) distributions show  
194 that the fine to medium silt (7-30  $\mu\text{m}$ ) dominate the majority of the analyzed suspended  
195 sediments. A small number of samples have modes in the very fine to fine silt fraction (4-15  
196  $\mu\text{m}$ ) or in the medium to coarse silt fraction (15-62  $\mu\text{m}$ ). The amount of sand ranges from 0.4  
197 to 49.9%, but 90% of the suspended sediment samples have less than 16% of sand. Higher  
198 concentrations of sand only occur in samples collected at depths near the riverbed.

199         Luminescence measurements were performed in the Luminescence and Gamma  
200 Spectrometry Laboratory at the Institute of Geosciences of the University of São Paulo using  
201 polymineral (quartz and feldspar) aliquots and quartz aliquots of fine sand (180–250  $\mu\text{m}$ ) or  
202 polymineral aliquots of silt (4-62  $\mu\text{m}$ ). Measurements were performed in a Risø TL/OSL DA-  
203 20 reader equipped with a built-in beta source ( $^{90}\text{Sr}/^{90}\text{Y}$ ; dose rate of 0.088  $\text{Gy}\cdot\text{s}^{-1}$  for cups and  
204 0.108  $\text{Gy}\cdot\text{s}^{-1}$  for discs), a bialkali PM tube (Thorn EMI 9635QB), a sample heater plate, and  
205 blue and IR light emitting diodes (LEDs). Aliquots were measured using the blue LED's and  
206 light detection through a 7.5 mm Hoya U-340 glass filter (290–340 nm). For sand sized grains,  
207 about 12 aliquots per sample were prepared by mounting grains in a steel cup. An acrylic plate  
208 with a microhole of 1470  $\mu\text{m}$  diameter per 1860  $\mu\text{m}$  depth was used to mount aliquots with  
209 similar volume and mass; each coarse-grained (sand) aliquot comprised approximately of 150  
210 to 200 sand grains, as observed under the microscope, with average mass of  $8.1 \pm 0.9$  mg  
211 (n=60). For the suspended silt samples, aliquots were prepared by evaporating four drops of

212 suspended sediment water solution over aluminum discs. Four fine-grained aliquots were  
213 measured per silt sample. X-ray fluorescence (XRF) measurements were used to determine the  
214 concentrations of Na, Ca, K and Si in polymineral sand aliquots; these measurements were  
215 performed in the Center for Nuclear Technologies of the Technical University of Denmark,  
216 using a Risø TL/OSL DA-20 reader equipped with a XRF attachment. Combined XRF and  
217 luminescence measurements were carried out in samples from the Solimões (11), Negro (5),  
218 Madeira (6) and Amazon (2) Rivers. The results of the XRF and luminescence measurements  
219 were correlated to evaluate the use of luminescence signals as a proxy for the concentration of  
220 feldspar relative to quartz grains.

221         The luminescence procedures (Table 1) included the bleaching of aliquots (step 1) to  
222 eliminate residual natural luminescence signals. Afterwards, 10 Gy and 50 Gy beta doses were  
223 given to induce luminescence signals in sand and silt aliquots, respectively. A pre-heat at 190°C  
224 for 10s was applied before luminescence measurements to eliminate unstable signals. Infrared  
225 (IR) stimulation for 300 s (step 4) measured feldspars signal in polymineral aliquots. This step  
226 served the purpose of screening any quartz aliquots that suffer from feldspar contamination;  
227 such aliquots were subsequently rejected from data analysis. OSL using blue light stimulation  
228 (BOSL) at 125°C was measured in step 5; this OSL signal represents quartz in case of pure  
229 quartz aliquots and is dominated by quartz in the polymineral aliquots. Step 6 repeated the OSL  
230 measurement to determine the background underlying the OSL obtained in step 5. The integral  
231 of the first 1s minus the last ten seconds of OSL emission from step 5 was used to estimate the  
232 intensity of the fast component ( $BOSL_F$ ) dominated signal from quartz aliquots (Choi et al.,  
233 2006; Jain et al., 2003). A quartz OSL sensitivity ratio was obtained by dividing  $BOSL_F$  from  
234 step 5 and the total OSL signal ( $BOSL_T$ ) obtained from step 6. This ratio represents the relative  
235 intensity of the fast component compared to the slow and medium components and also  
236 normalizes for the dispersion in sensitivity estimates due to differences in the number of light  
237 emitting grains from aliquot to aliquot within the same sample. The signal from the feldspar  
238 fraction ( $IRSL_f$ ) of the polymineral aliquots was estimated through the integration of the first  
239 1.2 s of IRSL emission in step 4, minus the corresponding average intensity in the last ten

240 seconds. The  $IRSL_I$  signal represents only the initial luminescence emission from feldspar  
 241 grains and should not be confused with the fast OSL component. The  $IRSL_I/BOSL_F$  (Step  
 242 4/Step 5) ratio was used as an index measuring the relative concentration of feldspar with  
 243 respect to quartz; note that this is only an approximate index since  $BOSL_F$  has also contribution  
 244 from feldspar grains because of incomplete resetting of feldspar OSL by IR exposure at 125 °C  
 245 (Jain and Singhvi, 2001). Furthermore, not all quartz grains may be emitting OSL (Duller,  
 246 2008).

Step	Procedure
1	Blue LEDs stimulation at 125°C for 100 s
2	Beta radiation dose of 10 Gy (sand) or 50 Gy (silt)
3	Pre-heat at 190°C for 10 s
4	Infrared stimulation at 60°C for 300 s
5	Blue LEDs stimulation at 125°C for 100 s
6	Blue LEDs stimulation at 125°C for 100 s

247

248 Table 1. The sequence of procedures used to measure luminescence sensitivity of sand  
 249 and silt aliquots.

250

### 251 3.3. Fe and K concentrations in suspended sediments

252 Fe and K data measured in suspended sediments were used as a proxy for the  
 253 provenance of sediments in the Amazon River basin. The concentrations of Fe and K are related  
 254 to the intensity of chemical weathering (Govin et al., 2012) in sediment source areas and  
 255 discriminate between sediments derived from Amazon Andean and Amazon lowland shield  
 256 rivers (Govin et al. 2014). Fe and K in suspended sediments were measured using inductively  
 257 coupled plasma-optical emission spectrometer (ICP-OES, Agilent 720) in the Marum-Center  
 258 for Marine Environmental Sciences of the University of Bremen. Digestion of suspended  
 259 material was performed with a microwave system (MLS, 1200 MEGA). For this purpose, 7 ml  
 260  $HNO_3$  (65%), 0.5 ml HF (40%), 0.5 ml HCl (30%), and 0.5 ml MilliQ was added to about 50  
 261 mg sample material (filter + suspended material) previously placed into Teflon liners. All acids  
 262 were of suprapure quality. See Zhang et al. (2017) for a detailed description of Fe/K data.

263

264 **4. Results**

265 Luminescence sensitivity results (Tables 2 and 3 and Supplementary Table S1) show  
 266 that riverbed sands and suspended silt of rivers with Andean headwaters like the Solimões, Içá,  
 267 Japurá and Madeira Rivers show lower sensitivity ( $BOSL_F$ ) compared to sand and silt from  
 268 shield rivers like the Tapajós and Xingu Rivers. Sediments from the Madeira River have higher  
 269 sensitivity than sediments from the Solimões River. Sand grains from the Negro River have  
 270 intermediate sensitivity (Figure 2). The Tapajós and Xingu Rivers stand out by the presence of  
 271 sand grains with higher variability of sensitivity across samples.

272 Results from pure quartz aliquots (Table 3 and Figure 2) confirmed the higher  
 273 sensitivity of quartz from shield rivers (Xingu and Tapajós). The exception is the similarity  
 274 between the sensitivity of quartz from the Negro and Madeira Rivers. Suspended silt and  
 275 riverbed sands show similar patterns of sensitivity variation among rivers. The variations in the  
 276 luminescence sensitivity are significantly larger than the 11% random uncertainty in aliquot  
 277 mass ( $8.1 \pm 0.9$  mg). This confirms that that our aliquot preparation approach combined with  
 278 luminescence signal normalization does not induce any systematic variations in measured  
 279 sensitivity of the studied sand samples.

River	N	% $BOSL_F/BOSL_T$		IRSL <sub>I</sub> / $BOSL_F$	
		Average	Std. dev.	Average	Std. dev.
Içá	5	9.20	2.64	112.75	29.56
Jutaf	3	23.14	0.84	48.60	22.39
Juruá	5	15.51	1.52	64.52	2.56
Japurá	5	7.89	0.61	112.81	34.93
Tefé	1	10.67	-	104.03	-
Coari	1	11.79	-	82.18	-
Purus	5	14.56	1.90	66.94	9.03
Solimões	10	7.57	0.64	157.44	22.66
Negro	7	19.6	13.08	72.79	17.34
Madeira	5	10.17	1.37	113.03	8.66
Tapajós	4	29.38	16.15	43.80	15.21
Xingu	5	25.16	12.23	51.47	20.30
Amazon	14	9.89	1.17	126.36	23.91

280

281 Table 2 – Summary of luminescence sensitivity results for suspended sediment samples  
 282 (silt grain size). N is the number of samples per river.

283

284

River	Polyminerals aliquots (quartz+feldspar)					Pure quartz aliquots		
	N	%BOSL <sub>F</sub> /BOSL <sub>T</sub>		IRSL <sub>L</sub> /BOSL <sub>F</sub>		%BOSL <sub>F</sub> /BOSL <sub>T</sub>		
		Average	Std. dev.	Average	Std. dev.	N	Average	Std. dev.
Solimões	21	4.27	0.62	83.46	16.22	6	15.37	5.04
Negro	9	14.53	6.82	33.40	22.70	3	25.26	14.86
Madeira	16	6.47	2.33	66.28	26.46	6	31.65	10.99
Tapajós	13	57.41	12.08	2.56	3.19	4	65.73	5.73
Xingu	9	42.08	13.59	2.16	1.68	6	56.26	7.25
Amazon	25	5.76	5.01	81.12	23.74	3	19.22	1.81

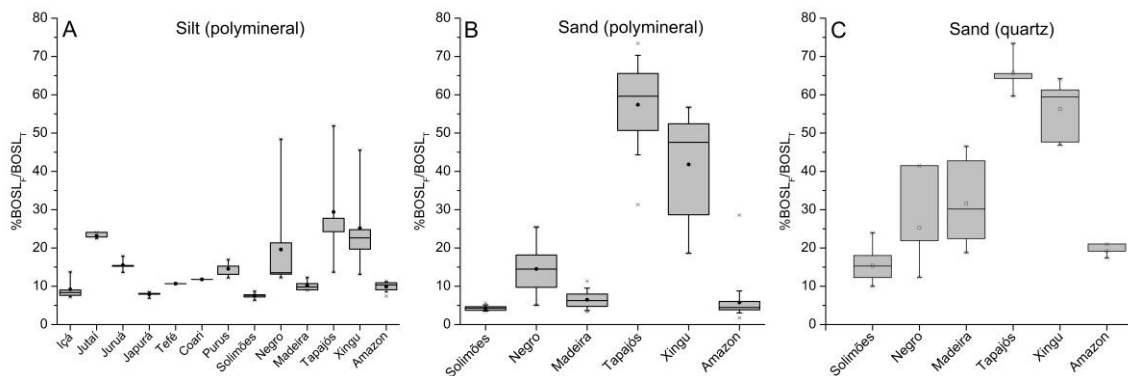
285

286

Table 3 – Summary of luminescence sensitivity results for riverbed sediment samples

287

(sand grain size). N is the number of samples per river.



288

289

Figure 2 – Luminescence sensitivity of samples from the Amazon River and its major

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tributaries. Data from polyminerals suspended silt (A), polyminerals riverbed sand (B), and pure

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quartz riverbed sand (C) are organized from upstream (left) to downstream (right) tributaries

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of the Solimões-Amazon main stem.

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The ratio BOSL<sub>F</sub>/BOSL<sub>T</sub> represents the relative intensity of the quartz fast OSL

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component compared to the slowly bleaching component(s). The slow and medium

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component(s) in the polyminerals aliquots contains an additional contribution from the slowly

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bleaching feldspar signal than in the pure quartz aliquots. Similarly, the intensity of IRSL from

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an aliquot is both a function of the sensitivity and proportion of feldspar in that aliquot. It is

299

expected that for the given sensitivity of quartz, the BOSL<sub>F</sub>/BOSL<sub>T</sub> ratio in polyminerals aliquots

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should decrease with an increase in the feldspar concentration both due to the reduced quantity

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of quartz (dilution effect), as well as an increase in the BOSL<sub>T</sub> due to feldspar contribution

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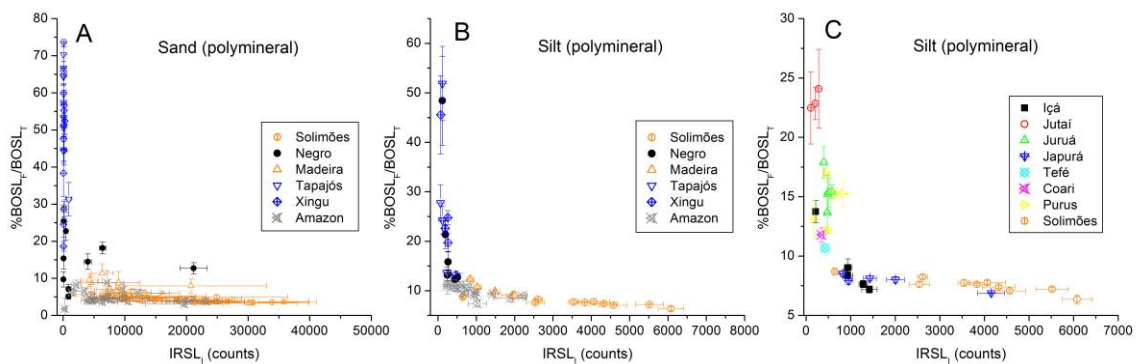
(note that these are both linear effects). This is exactly what we observe in the polyminerals data;

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both suspended silt and riverbed sands show a trend of increasing BOSL<sub>F</sub> sensitivity with

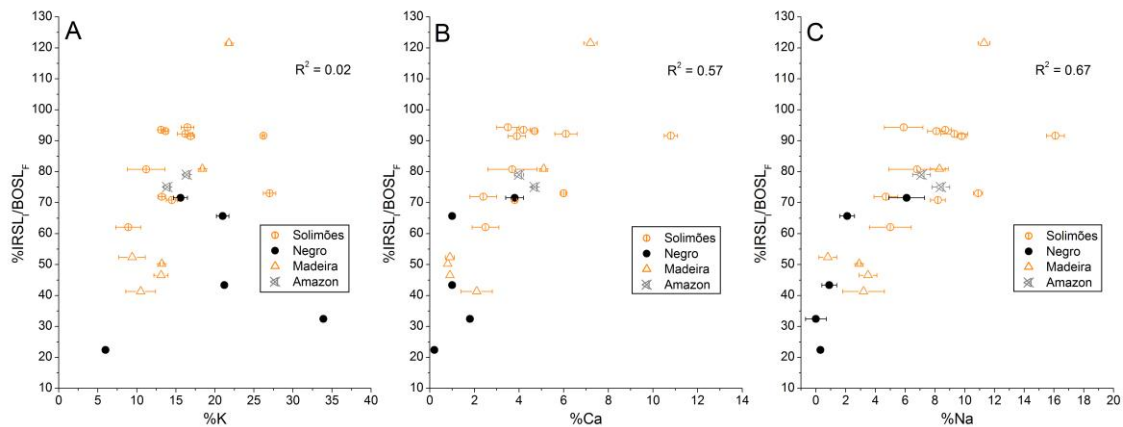
304 decreasing  $IRSL_L$  sensitivity and feldspar content (Figure 3). Note that the near-zero value on  
305 the x axis indicates the pure quartz fractions; the spread in y values here is thus the true variation  
306 in the sensitivity of the quartz OSL component across samples.

307 The  $IRSL_L/BOSL_F$  ratio is positively correlated with the concentrations of Na and Ca,  
308 but a negligible correlation ( $R^2=0.02$ ) was observed between  $IRSL_L/BOSL_F$  ratio and K (Figure  
309 4 and Supplementary Table S2). This suggests that the IRSL intensity variation is controlled  
310 by Na-Ca feldspar concentration. The K content does not show a clear correlation due to the  
311 presence of a few high K bearing aliquots with below average sensitivity. The likely  
312 explanation for this result is that K may be contained in non-feldspar minerals such as biotite  
313 and muscovite. In summary, these results point out that the relative  $BOSL_F$  sensitivity is  
314 decreasing with an increase in the feldspar content, thus suggesting that the amount of feldspar  
315 grains is the dominant source affecting the  $BOSL_F$  sensitivity in the polymineral aliquots.



316

317 Figure 3 – Variation of  $BOSL_F$  and  $IRSL_L$  luminescence sensitivities in polymineral  
318 aliquots of riverbed sand (A) and suspended silt (B) of the major tributaries of the Amazon  
319 River. Luminescence sensitivities of suspended silt from tributaries of the Solimões River are  
320 shown in C. Samples named as “Solimões” correspond to sediments from the Solimões River  
321 main stem. Data are organized from upstream (left) to downstream (right) tributaries of the  
322 Solimões-Amazon main stem.



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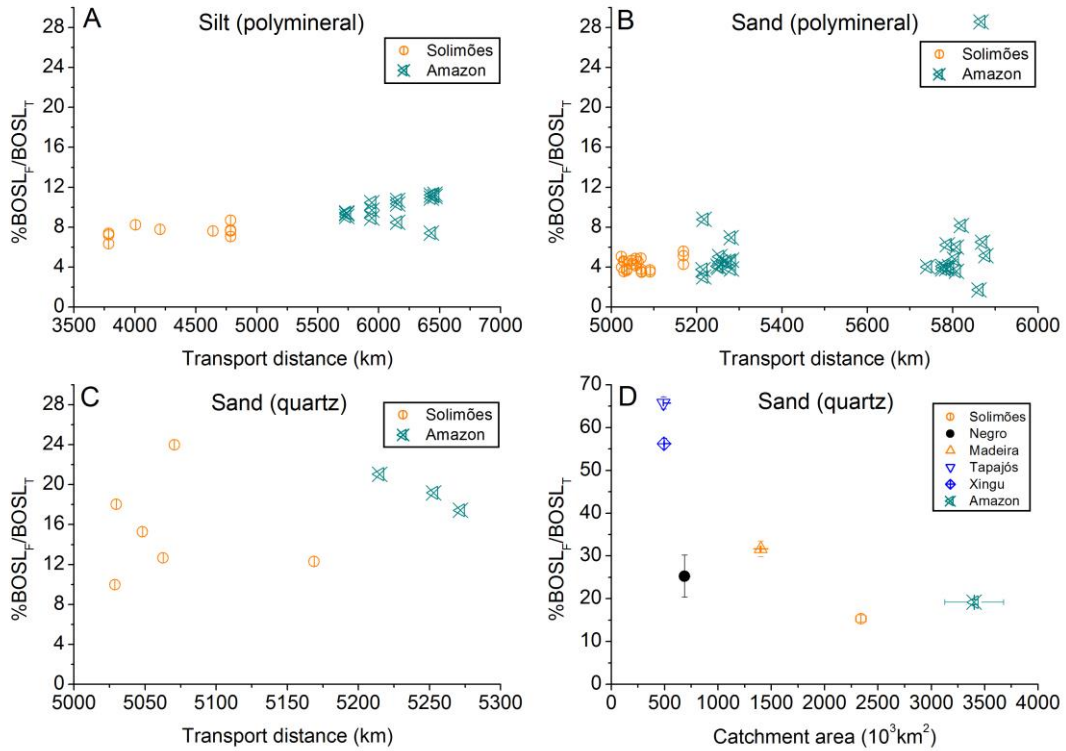
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Figure 4 – Variation of potassium (A), calcium (B) and sodium (C) contents compared to the  $IRSL_I/BOSL_F$  ratio measured in polymineral sand aliquots of the Solimões (11), Negro (5), Madeira (6) and Amazon (2) Rivers.

Sediments from the Solimões, Madeira and Amazon Rivers, which have larger catchments, requiring longer distances of sediment transport from headwaters to the sampling sites, have quartz grains with lower sensitivity (Figure 5 and Table 3). Also, a systematic trend of increase in quartz sensitivity ( $BOSL_F$ ) with distance from the sediment source areas is not observed along the Solimões-Amazon River main stem (Figure 5 and Supplementary Table S3). The Fe/K ratio has been used as a proxy to differentiate between sediments of Andean and lowland shield tributaries of the Amazon River (Govin et al. 2014). In the studied samples, the Solimões, Madeira and Amazon Rivers show suspended sediments enriched in K (low Fe/K), with little variation between the low and high water level periods (Figure 6). The higher concentration of K in sediments of these Rivers fits with their higher  $IRSL_I$  signal, confirming higher feldspar concentration in sediments from these rivers. Suspended sediments transported by the Negro, Tapajós and Xingu Rivers show higher Fe/K values, with significant increase in Fe concentration during the high water level period, when rainfall increases the input of sediments derived from the erosion of heavy weathered soils. Lateritic soil profiles that cover most of the upstream shield areas are enriched in Fe oxides and hydroxides, which are transported to the river channels during the rainy season.



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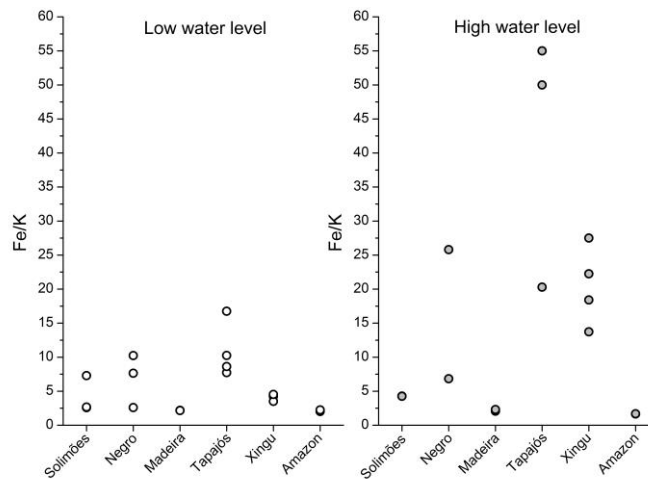
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Figure 5 – Variation in luminescence sensitivities of polym mineral silt (A), polym mineral sand (B) and pure quartz sand (C) along the Solimões-Amazon River main stem. A comparison between the sensitivity of quartz and the catchment area upstream the sampling sites are shown in D. Each river is represented by the average and standard deviation (error bars) values of quartz sensitivity and catchment area.



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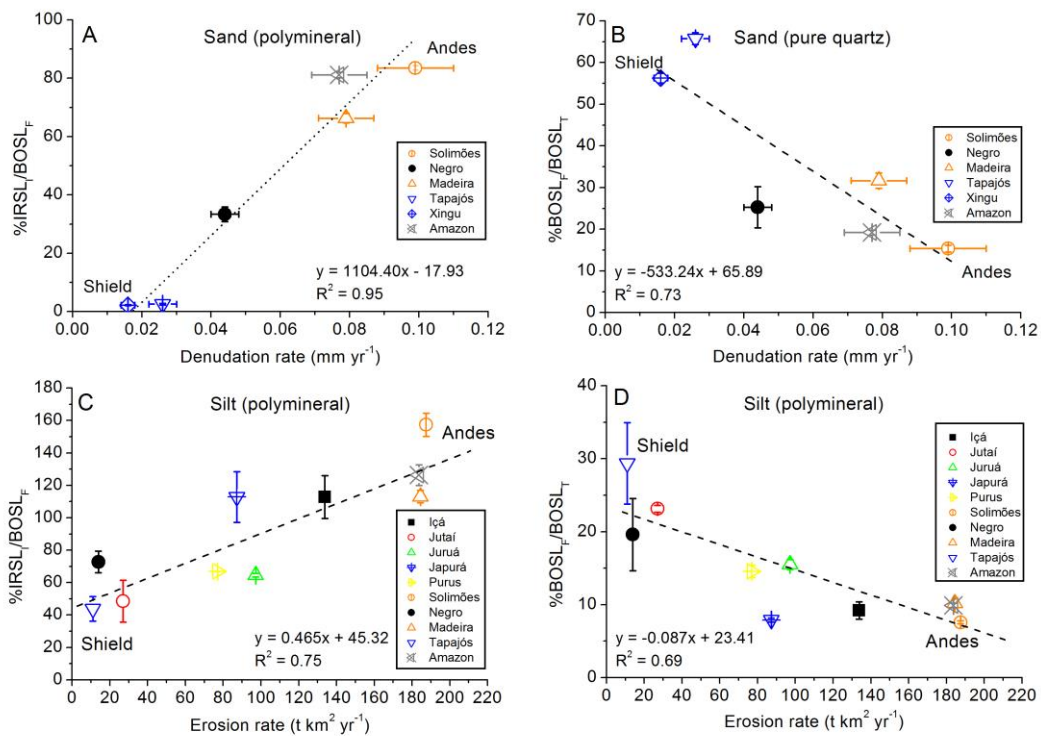
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Figure 6 – Fe/K ratio in suspended sediments sampled during the low (A) and high (B) water level periods. Number of samples: Solimões high=3, low=3; Madeira high=3, low=3; Negro high=3, low=2; Tapajós high=3, low=4; Xingu high=4, low=4.



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355 The IRSL signal from the polymineral aliquots shows a positive correlation with  
356 denudation rate determined through  $^{10}\text{Be}$  concentrations (Wittmann et al., 2010) in sediments  
357 from the Amazon rivers (Figure 7). This correlation suggests that for areas with high denudation  
358 rates there is relatively less time available for chemical weathering of feldspar; thus, the  
359 resulting fine sand fraction has a higher feldspar to quartz ratio. However, interestingly the  
360 BOSL<sub>F</sub> signal from pure quartz aliquots correlates negatively with denudation rates; this is a  
361 new observation, which suggests that landscape denudation may play an important role in  
362 luminescence sensitization of quartz.



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364 Figure 7 – Variation of luminescence signals of feldspar (A) and quartz (B) from  
365 riverbed sands in terms of denudation rates derived from  $^{10}\text{Be}$  concentrations measured in  
366 quartz (125-250  $\mu\text{m}$ ). Feldspar (%IRSL<sub>i</sub>/BOSL<sub>F</sub>) and quartz (%BOSL<sub>F</sub>/BOSL<sub>T</sub>) signals  
367 respectively measured in polymineral and pure quartz aliquots from fine sand. Denudation rates  
368 data compiled from Wittmann et al. (2010). In Wittmann et al. (2010), the samples from the  
369 Tapajós and Xingu Rivers represent sediments from headlands. Samples from Negro, Madeira  
370 and Solimões Rivers are from lower reaches near the sites sampled in this study. Samples from

371 the Amazon River represent sediments from sectors downstream the Madeira River and  
372 upstream the Tapajós River (Parintins and Óbidos). C and D show the ratios  $\%IRSL_L/BOSL_F$   
373 and  $\%BOSL_F/BOSL_T$  measured in polymineral aliquots of suspended silt in terms of erosion  
374 rates calculated through sediment gauging stations. Erosion rates data compiled from Bouchez  
375 et al. (2014).

376

## 377 **5. Discussion**

### 378 **5.1. Sensitization of quartz OSL in nature**

379 OSL sensitivity is a fundamental property of quartz for its use as a luminescence  
380 geochronometer. The relationship between OSL and the radiation dose accumulated since the  
381 last daylight exposure of quartz grains allows estimation of the sediment burial age. There have  
382 been numerous studies in the past to understand OSL characteristics of quartz for its robust  
383 application in geochronometry. However, most of these studies have focused on laboratory-  
384 induced changes in the quartz OSL using a combination of heat, light or ionizing radiation  
385 (Wintle and Adamiec, 2017), and the drivers that induce quartz OSL sensitization in nature are  
386 still poorly understood.

387 Quartz extracted from different types of igneous and metamorphic rocks has a  
388 relatively low sensitivity (Chithambo et al., 2007; Guralnik et al., 2015) compared to quartz  
389 from sediments (Sawakuchi et al., 2011). Heating was the first factor recognized to increase the  
390 luminescence sensitivity of quartz (Bøtter-Jensen et al., 1995). However, significant  
391 sensitization of different OSL components of quartz only occurs after heating to temperatures  
392 above 300°C (Jain et al., 2003), which is not likely in surface sedimentary environments.  
393 Interestingly, the luminescence sensitivity of quartz grains from sediments can vary by several  
394 orders of magnitude (Pietsch et al., 2008). These observations suggest that the sensitization of  
395 quartz occurs somewhere between source to sink in sedimentary environments.

396 Pietsch et al. (2008) and Gliganic et al. (2017) observed a positive correlation between  
397 the OSL sensitivity of quartz and downstream distance of sediment transport along Australian  
398 rivers, proposing that sensitization occurs due to cycles of irradiation under burial and solar

399 exposure during sediment transport. However, the increase in OSL sensitivity with the distance  
400 of sediment transport is not confirmed by our new data from the Amazon rivers. On the  
401 contrary, lower sensitivity quartz occurs in the Solimões, Madeira and Amazon Rivers, which  
402 have longer sediment transport pathways and larger catchment areas (Figure 5). Thus,  
403 luminescence characteristics inherited from sediment source rocks or other surface processes  
404 can drive the sensitization of quartz in the Amazon fluvial system.

405         Quartz from sediments deposited around mountain ranges like the New Zealand Alps  
406 (Preusser et al., 2006), European Alps (Klasen et al., 2007), Scottish Highlands (Lukas et al.,  
407 2007), Andes (Steffen et al., 2009) and Himalayas (Jaiswal et al., 2008) usually has very low  
408 luminescence sensitivity. On the other hand, regions lacking young high relief mountain ranges  
409 like southeastern Australia (Fitzsimmons et al., 2010) and northeastern and southeastern Brazil  
410 (Guedes et al., 2011) have widespread occurrence of sediments with high sensitivity quartz  
411 grains suitable for luminescence dating. This geographical pattern implies that mountain  
412 building and rates of uplift and erosion may play an important role in the sensitization of quartz  
413 in nature. In the studied rivers, quartz with the lowest OSL sensitivity is provided by the  
414 Solimões River (Figures 1 and 2, Tables 2 and 3), where most of its sediments are supplied by  
415 the Andes mountain belt (Gibbs, 1967; Govin et al., 2014). Quartz with higher sensitivity  
416 occurs in the Tapajós and Xingu Rivers, which exclusively drain stable cratonic areas of the  
417 Central Brazil shield. The Madeira and Negro Rivers have quartz with intermediate sensitivity,  
418 indicating a mixture of sediments from Andean and shield sources. This is in agreement with  
419 the area drained by these rivers. Despite its Andean headwaters, the middle and lower Madeira  
420 River receive sediments from tributaries running over the Central Brazil shield. On the other  
421 hand, the Negro River mostly drains the Guiana shield, but has lowland tributaries draining  
422 Quaternary terraces of the Japurá and Solimões Rivers, whose sediments are provided mainly  
423 by Andean sources.

424         The sediment source rocks in Andes, Central Brazil and Guianas are very diverse and  
425 include many types of igneous, sedimentary and metamorphic rocks (Jaillard et al., 2000;  
426 Tassinari et al., 2000; Rossetti et al., 2005). The headwaters of the Madeira and Solimões Rivers

427 are located in the Eastern Cordillera of the Bolivian and Peruvian Andes, an area with  
428 widespread occurrence of Mesozoic and Neogene volcanic and metasedimentary rocks.  
429 However, most of the Andean drainages flowing to the lowland Amazon run over sediments of  
430 the subandean zone and of the eastern Andean lowlands (Jailard et al., 2000). The shield areas  
431 in Central Brazil and Guianas are dominated by Pre-Cambrian metamorphic and intrusive  
432 igneous rocks. Upstream areas of the Xingu and Tapajós Rivers also include Paleozoic and  
433 Mesozoic sedimentary rocks that overlay the metamorphic and igneous rocks of the southern  
434 portion of the Central Brazil shield. Andean and shield areas also have contrasting denudation  
435 rates as indicated by fluvial sediment flux and total dissolved solids data (Bouchez et al., 2014;  
436 Moquet et al., 2016) and cosmogenic nuclide data (Wittman and Blanckendburg, 2009;  
437 Wittmann et al., 2009; 2010). Denudation rates in river catchments from Andes ( $0.5 \text{ mm.yr}^{-1}$ )  
438 and shield Amazon ( $0.01 \text{ mm.yr}^{-1}$ ) catchments differ by one order of magnitude (Wittmann et  
439 al., 2010). The different geological context between Andes and shield areas define two end-  
440 member sediment compositions that are sourced to the Amazon River basin (Basu et al., 1990;  
441 Viers et al., 2008). The higher feldspar content in Andean sediments, as indicated by the higher  
442  $\text{IRSL}_i/\text{BOSL}_F$  ratio in sediments from the Solimões River (Figure 3, Tables 2 and 3), further  
443 confirms that lower sensitivity quartz is derived from rocks located in catchments under higher  
444 denudation rates. In this case, higher denudation rates in Andes are linked to fast erosion of  
445 soils, which allows short time for chemical weathering and, thus, favors the preservation of  
446 feldspar grains in river sediments. On the other hand, geological settings under lower  
447 denudation rates (e.g. Tapajós and Xingu Rivers) promote longer weathering time and thereby  
448 reduced feldspar input (due to weathering), and high sensitivity quartz. Also, the Solimões,  
449 Negro and Madeira Rivers flow through major Quaternary and Neogene sediment accumulation  
450 areas in lowland Amazonia (Rossetti et al., 2005) while the Tapajós and Xingu Rivers comprise  
451 bedrock incised channels with low accommodation space and high sediment bypass. On  
452 average, it is expected that sediment quartz grains have a higher near-surface residence time in  
453 low relief, slowly eroding catchments due to low accommodation space (Jain et al., 2004)  
454 compared to quartz grains derived from rocks in high relief, fast eroding catchments. It is

455 plausible that the average residence time of quartz grains in soil profiles and weathering  
456 processes in the surface environment may play an important role for luminescence sensitization  
457 of quartz. The sensitivities of suspended silt (polymineral aliquots) and riverbed sand (pure  
458 quartz aliquots) show minor variation along transport distances of respectively 2750 km and  
459 250 km in the Solimões-Amazon River main stem (Figure 5). This may suggest that sediment  
460 transport has minimal influence on the luminescence sensitization of sediments in the Amazon  
461 basin.

462         The inverse relationship between denudation rates and quartz OSL sensitivity (Figure  
463 7B) allows us to propose that the exhumation and weathering history of the source rocks have  
464 a significant influence on the OSL sensitization of quartz. In addition to the surface residence  
465 time, other factors such as uplift rate and age of source rocks may also influence quartz  
466 sensitivity. For example, sensitization may be related to the thermal (cooling) history of source  
467 rocks; lower uplift rates imply proportionately longer storage time under higher subsurface  
468 temperatures. The age of source rocks can also contribute to sensitization since older rocks will  
469 promote a higher absorbed radiation dose in quartz crystals before they become sediment  
470 grains. The accumulated dose in quartz crystals provided by Cenozoic or Pre-Cambrian igneous  
471 and metamorphic source rocks can differ in several orders of magnitude. Quartz crystals within  
472 Proterozoic granites (810-580 Ma) from Brazilian shield areas were exposed to radiation doses  
473 about 4.1-5.7 MGy, assuming dose rates of 7 Gy/ka (gamma and beta) calculated through  
474 average concentrations of U, Th and K obtained for granite plutons from southeastern Brazil  
475 (Alves et al., 2016). The combination of higher accumulated dose and longer heating of source  
476 rocks from Pre-Cambrian shield areas under slow uplift rates resembles a natural pre-dose and  
477 accumulated thermal sensitization effects (Bailey, 2001).

478         Finally, the sensitization of quartz may be related to chemical weathering processes.  
479 Our data suggest that more sensitive quartz comes from source rocks exposed to longer duration  
480 of chemical weathering (because of lower denudation rates). According to Sharma et al. (2017)  
481 quartz samples with higher water content have lower OSL sensitivity, implying that water-  
482 related defects increase the efficiency of non-radiative recombination centers and favor

483 luminescence quenching. However, it is not clear how water molecules are driven out of quartz  
484 in areas of low denudation (high weathering) rates. This effect must only be important once the  
485 rock enters the active weathering zone. Another hypothesis to link denudation and weathering  
486 processes and luminescence sensitization is that the fragmentation of larger quartz crystals  
487 during weathering increases the capacity of the alpha and beta radiation in promoting physical  
488 damage in quartz. Considering that quartz has a negligible amount of radionuclides, quartz  
489 crystal fragmentation and production of smaller quartz grains increase the interaction between  
490 grains and the external radionuclide-derived alpha and beta radiation. Additionally, the  
491 interaction between the cosmic radiation and quartz increases when source rocks reach  
492 shallower depths. Thus, the effect of the ionizing radiation on quartz grains possibly produces  
493 defects related to luminescence processes. Further studies are necessary to evaluate if the  
494 sensitization of quartz in the Amazon fluvial system is related to a relative decrease of non-  
495 radiative (K-centers) recombination centers (Bailey, 2001) or to changes in electron traps  
496 associated with the OSL fast component.

497

## 498 **5.2. Sources of suspended and riverbed sediments in the Amazon River basin**

499 Despite the incomplete understanding about natural sensitization of quartz in terms of  
500 its electron-hole trapping system, the luminescence signals related to quartz ( $BOSL_F$ ) and  
501 feldspar ( $IRSL_f$ ) clearly discriminate between Andean and shield sediments (Figure 3, Tables  
502 2 and 3). Thus, luminescence signals of quartz and feldspar are useful proxies to track the  
503 relative contribution of Andean and shield areas to the suspended and riverbed sediments  
504 transported by the Amazon River. The  $BOSL_F/BOSL_T$  ratio (Figure 2) indicates that the  
505 Madeira River is the major source of silt to the Amazon River, which is confirmed by Fe/K  
506 data (Figure 6), in accordance with other studies showing that Andean rivers dominate the  
507 suspended sediment load delivered by the Amazon River to the Atlantic Ocean (Govin et al.,  
508 2014; Zhang et al., 2017). Riverbed sands show a different provenance pattern, with higher  
509 contribution of sands from the Solimões River (Figures 2 and 3). The  $BOSL_F/BOSL_T$  ratio  
510 measured in pure quartz aliquots also indicates significant contribution of cratonic sands to the

511 Madeira River, pointing to a high decoupling between the sources of silt (Andean) and sand  
512 (Andean-Shield). The decoupling between catchment area and sediment supply is also observed  
513 for the Solimões River. The Solimões River sediments have low sensitivity, which is similar to  
514 that presented by the Içá and Japurá Rivers. On the other hand, sediments from lowland  
515 tributaries (Jutaí, Juruá, Tefé, Coari and Purus River) of the Solimões River have higher  
516 sensitivity, suggesting a mixture of sediments from Andean and shield sources (Figures 2 and  
517 3). The  $BOSL_F/BOSL_T$  ratio also differentiates sediments supplied by shield rivers (Negro,  
518 Xingu and Tapajós), which are similar regarding their major elemental composition (Govin et  
519 al., 2014; Zhang et al., 2017).

520 Paleoenvironmental changes in the Amazon River basin have been reconstructed  
521 through proxies based on inorganic geochemistry of suspended sediments deposited offshore  
522 the Amazon River mouth (Govin et al., 2014). However, suspended sediments of the Andean  
523 rivers dominate the inorganic geochemical signals measured in sediments of the Amazon River,  
524 potentially hindering the recognition of paleoenvironmental changes in shield and lowland  
525 areas. Shield rivers have a low suspended load (Sioli, 1985), but represent a significant portion  
526 of the Amazon River basin, including the Negro River, its second largest tributary (Figure 1).  
527 Our study suggests that quartz OSL sensitivity may be useful to reconstruct paleoenvironmental  
528 changes in the Amazon River basin, including these underrepresented areas because the clear  
529 distinction between OSL sensitivity of quartz from shield rivers compared to that from the  
530 Andean rivers. The high abundance and high stability of quartz to weathering and diagenetic  
531 processes combined with fast and easy-to-make luminescence measurements are great  
532 advantages of the luminescence techniques to constrain changes in sediments provenance in  
533 the Amazon River basin and potentially in other large river contexts.

534

## 535 **6. Conclusions**

536 Optically stimulated luminescence signals fingerprint the sources of quartz and  
537 feldspar in suspended and riverbed sediments of Amazon rivers. The intensity of the IRSL  
538 signal indicates the relative concentration of feldspar in sediments. The correlation observed

539 between quartz OSL sensitivity and denudation rate in the source area suggests that near-  
540 surface residence time of quartz grains and possible thermal/irradiation history may play an  
541 important role in sensitization of quartz in nature. This correlation explored for the first time in  
542 this study highlights the use of quartz OSL sensitivity as a complementary proxy for sediment  
543 sources and denudation rate estimates. Additionally, luminescence signals measured in riverbed  
544 sand and suspended silt arise from the same components (quartz and feldspar), avoiding the  
545 inevitable grain size bias in provenance analysis caused by the large difference between the  
546 traction and the suspended load of the Andean rivers and the shield rivers. The qualitatively  
547 and quantitatively relationship between quartz OSL sensitivity and denudation rates in sediment  
548 source areas is promising to develop methods for studying landscape evolution. Finally, we  
549 show that in the context of the Amazon fluvial system both the IRSL intensity (a proxy for  
550 quartz to feldspar ratio) as well as the quartz OSL sensitivity can be tied to the provenance.  
551 Thus, a quantitative change in these proxies measured in the stratigraphic record may be linked  
552 to past provenance scenarios and variation in denudation rates, which can track precipitation  
553 changes and drainage configuration in sediment source areas.

554

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