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Title
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Age of riverine carbon suggests rapid export of terrestrial primary production in tropics

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[1] The balance between the storage of vascular plant carbon in soils, oxidation to carbon dioxide, and export via rivers affects calculations of the strength of terrestrial ecosystems as carbon sinks. The magnitude and timescale of the riverine export pathway are not well constrained. Here we use radiocarbon dating of lignin phenols to show that plant-derived carbon carried by suspended sediment of the Mekong River is very young, having been produced within the last 18 years. Further, this plant-derived carbon remains young during times of the year when bulk carbon varies from modern to over 3000 radiocarbon years old. Our results demonstrate that primary-production derivatives are exported rapidly and suggest that the age of riverine lignin is similar to estimates of the residence time of terrestrial organic carbon in tropical catchments. These results are relevant for modeling predictions of the influence of the terrestrial biosphere on atmospheric carbon dioxide levels. **Citation:** Martin, E. E., A. E. Ingalls, J. E. Richey, R. G. Keil, G. M. Santos, L. T. Truxal, S. R. Alin, and E. R. M. Druffel (2013), Age of riverine carbon suggests rapid export of terrestrial primary production in tropics, *Geophys. Res. Lett.*, *40*, 5687–5691, doi:10.1002/2013GL057450.

1. Introduction

[2] Vascular plants comprise almost all of the 650 petagrams (Pg) of carbon (C) stored in vegetation, and their derivatives comprise a significant fraction of the ≈ 2300 Pg C stored in nonfrozen surface soils [*Sabine et al.*, 2004; *Chapin et al.*, 2011]. Because these pools are much larger than the atmospheric C pool, changes in the rate at which vascular plant-derived C (C_{VP}) cycles within Earth's reservoirs can impact atmospheric carbon dioxide (CO_2) levels. Rivers connect terrestrial and marine reservoirs, and C_{VP} is a significant component of the organic matter they carry [*Mayorga et al.*, 2005; *Hedges et al.*, 1986]. Inland waters, including rivers, contribute significantly to the C cycle, collectively receiving 2.7 Pg C yr^{-1} from upland ecosystems, an amount similar to the terrestrial sink for anthropogenic CO_2 emissions [*Battin et al.* 2009, and references therein]. C_{VP} received by rivers is subject to several biogeochemical fates that operate on different timescales. The most biologically available pool is rapidly consumed and returned to the atmosphere as CO_2 [*Mayorga et al.*, 2005; *Ward et al.*, 2013]. Alternatively, some C_{VP}, along with petrogenic C, is exported to coastal environments, where marine burial of C_{VP} represents a permanent sink for carbon over geologic timescales [*Berner*, 1982]. We currently lack direct estimates of the radiocarbon signature ($\Delta^{14}C$) of C_{VP} exported by tropical rivers, which hinders our ability to derive accurate models of how the size of marine and terrestrial C sinks will respond to climate change. Further, constraining the amount and radiocarbon age of C_{VP} exported by rivers is critical for quantifying the fraction of terrestrial primary productivity exported from catchments [*Hilton et al.*, 2008] and enumerating the residence time (RT) of C_{VP} exported from these catchments.

[3] Tropical ecosystems are anticipated to experience unprecedented temperature increases within the next two decades [*Diffenbaugh and Scherer*, 2011]. However, the effects of climate change on C cycling dynamics in the tropics is uncertain, especially concerning net primary production (NPP) [*Saleska et al.*, 2003; *Hutyra et al.*, 2007] and the fate of C_{VP} in soils. Changes in NPP will affect net storage of C in soils, which primarily reflects the balance between inputs from leaf and root detritus and losses dominated by microbial decomposition [*Davidson and Janssens*, 2006]. If inputs exceed decomposition, soils could exert a negative feedback on atmospheric CO_2 levels, lengthening the RT. In contrast, decomposition in excess of inputs would constitute a positive feedback [*Davidson and Janssens*, 2006]. Given that soil properties vary spatially, it is difficult to extrapolate the RT of a single pool of soil C to the watershed. Alternatively, the organic carbon (OC) transported by rivers is mobilized via events occurring across catchments, making this material integrative of catchment-wide biogeochemical processes [*Galy and Eglinton*, 2011]. Thus, the $\Delta^{14}C$ value of vascular-plant detritus exported by rivers could be highly informative in tracking the net effects of climate change on carbon storage within watersheds.

[4] We lack $\Delta^{14}C$ measurements of C_{VP} exported by tropical rivers. This is largely because riverine OC is derived from a mixture of sources with contrasting $\Delta^{14}C$ values [*Galy and Eglinton*, 2011; *Rosenheim and Galy*, 2012]. Potential sources for riverine OC include petrogenic C that contains no ^{14}C ($\Delta^{14}C = -1000‰$) [*Galy et al.*, 2008], and phytoplankton and vascular plants, both of which reflect the inorganic C source incorporated during photosynthesis [*Mayorga et al.*, 2005]. To determine the RT of C_{VP}, we purified lignin

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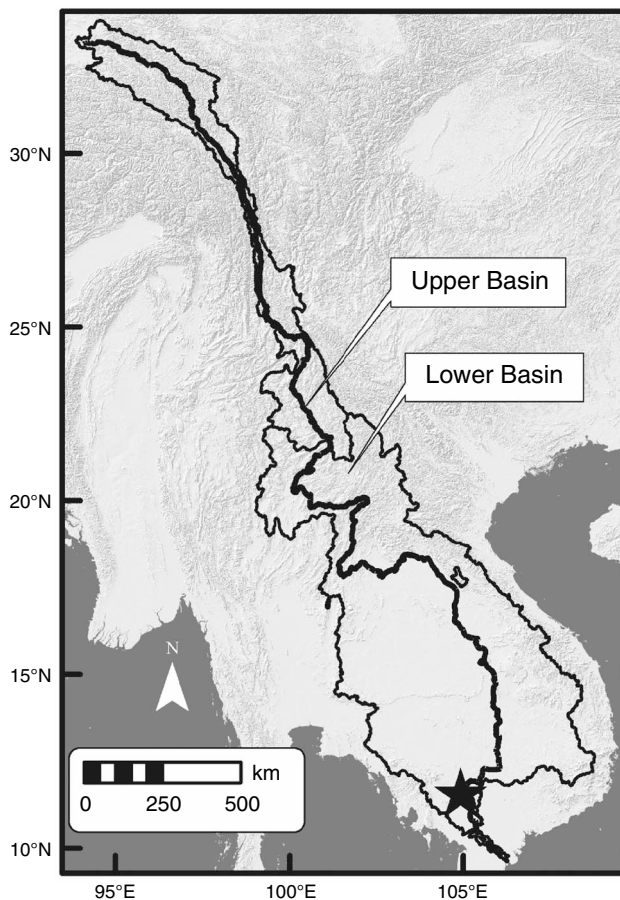
Figure 1. Map of the Mekong basin and the study site. The Upper and Lower Basins denote areas of the catchment above and below China, respectively [*Ellis et al.*, 2012, and references cited within]. The star denotes the sampling location. This figure was modified from *Ellis et al.* [2012].

phenols from fine suspended sediments (FSS) carried by the Mekong River of Southeast Asia (Figure 1) during different seasons and determined $\Delta^{14}C$ values of the individual lignin monomers [*Ingalls et al.*, 2010] and bulk carbon. Lignin is diagnostic for vascular plants and is the second most abundant biomarker on Earth [*Hedges et al.*, 1997]. As the $\Delta^{14}C$ value of each lignin monomer is a composite of that monomer from sources with varying ^{14}C ages, monomers can be used as a proxy for the average RT of C_{VP} exported by rivers within the catchment. This work is complimentary to studies examining the RT of C_{VP} in terrestrial environments using plant wax lipids [*Drenzek*, 2007; *Feng et al.* 2013]. Knowing the RT of lignin in the tropics could improve our understanding of the cycling of C_{VP} given, it is quantitatively more significant relative to lipids [*Hedges et al.*, 1997].

2. Methods

[5] We measured the ^{14}C and the stable isotopic (^{13}C) composition of dissolved OC (DOC) and fine particulate OC (FPOC) between September 2008 and February 2010 just above the mouth of the Mekong River in Cambodia (11.5956°N, 104.9429°E). Samples were collected from mid-depth. Water was filtered through precombusted QM/A quartz filters for ^{14}C analysis of FPOC, after first removing

coarse material [*Ellis et al.*, 2012]. Acidified filters were transferred to 9 mm precombusted quartz tubes along with copper oxide and silver wire, which were then sealed and combusted. All supporting organic parameters ($\delta^{13}C$, weight % OC, C:N ratios, and sediment and carbon concentrations) were measured as in *Ellis et al.* [2012]. Sieved water was also filtered through GF/F filters to yield DOC. For ^{14}C analyses, DOC samples were acidified to pH 2, dried at 60°C under a stream of ultrapure nitrogen gas, double tubed, sealed under vacuum, and combusted. Cryogenically purified CO_2 was converted to graphite, and bulk ^{14}C and ^{13}C analyses were performed at the Keck Carbon Cycle Accelerator Mass Spectrometry/University of California, Irvine facility [*Santos et al.*, 2007].

[6] We determined the ^{14}C content of lignin phenols in FSS collected from up to 1000 L of water during the rising (June and July 2009) and high-water seasons (September 2008). A Sharples T1 continuous flow centrifuge was used to concentrate the sediment from seived water [*Hedges et al.*, 1986]. This sediment was then dried. Seven to twelve grams were subject to oxidative hydrolysis in a microwave to liberate lignin-derived phenols. An aliquot of each sample was quantified by gas chromatography flame ionization detection [*Goni and Montgomery*, 2000]. Lignin phenols were purified using high-performance liquid chromatography, and $\Delta^{14}C$ values were obtained following *Ingalls et al.* [2010]. Between 22–35 and 5–14 injections in reverse and normal phase, respectively, were required to obtain adequate material for ^{14}C-AMS analysis. To account for extraneous carbon contamination, procedural blanks of the entire method were analyzed and used to correct $\Delta^{14}C$ values [*Santos et al.*, 2007]. The amount of modern C contamination (i.e., $\Delta^{14}C = 0\text{‰}$) was 0.5 ± 0.35 µg C, whereas the dead C contamination (i.e., $\Delta^{14}C = -1000\text{‰}$) was 0.8 ± 0.4 µg C.

3. Results and Discussion

[7] The Mekong River ranks among the top 10 rivers in the world with respect to its sediment load [*Milliman and Syvitski*, 1992], and our results demonstrate that the contribution of different OC sources to this sediment varies seasonally. FSS and FPOC concentrations follow the hydrograph (Figure 2a and Table 1) [*Ellis et al.*, 2012]. Although C:N ratios and $\delta^{13}C$ values vary little seasonally (Table S1), the percentage of OC was inversely related to FSS concentrations (Figure 2b) due to the increasing influence of phytoplankton during the low-water period [*Ellis et al.*, 2012]. The $\Delta^{14}C$ values of FPOC indicate that the OC exported during the high-water period is the most contemporary ($\Delta^{14}C = 26\text{‰}$), whereas increasingly aged OC is exported at the end of the low-water/beginning of the rising water period ($\Delta^{14}C = -328\text{‰}$) (Figure 2c). Values of $\Delta^{14}C$ greater than 0‰ indicate that $^{14}CO_2$ was fixed from the atmosphere after the detonations of nuclear weapons during the 1950s and early 1960s, which nearly doubled the concentration of ^{14}C in the atmosphere, with a maximum close to 900‰ in 1964. After the ban on weapons testing, atmospheric ^{14}C concentrations decreased rapidly [*McNichol and Aluwihare*, 2007, and references therein]. Thus, our postbomb $\Delta^{14}C$ values suggest that the dominant source of OC in the youngest FPOC was fixed within the last 50 years. The oldest FPOC predominantly contains OC that exceeds 3000 ^{14}C years in age (Table 1 and Table S1). The $\Delta^{14}C$ value of DOC was
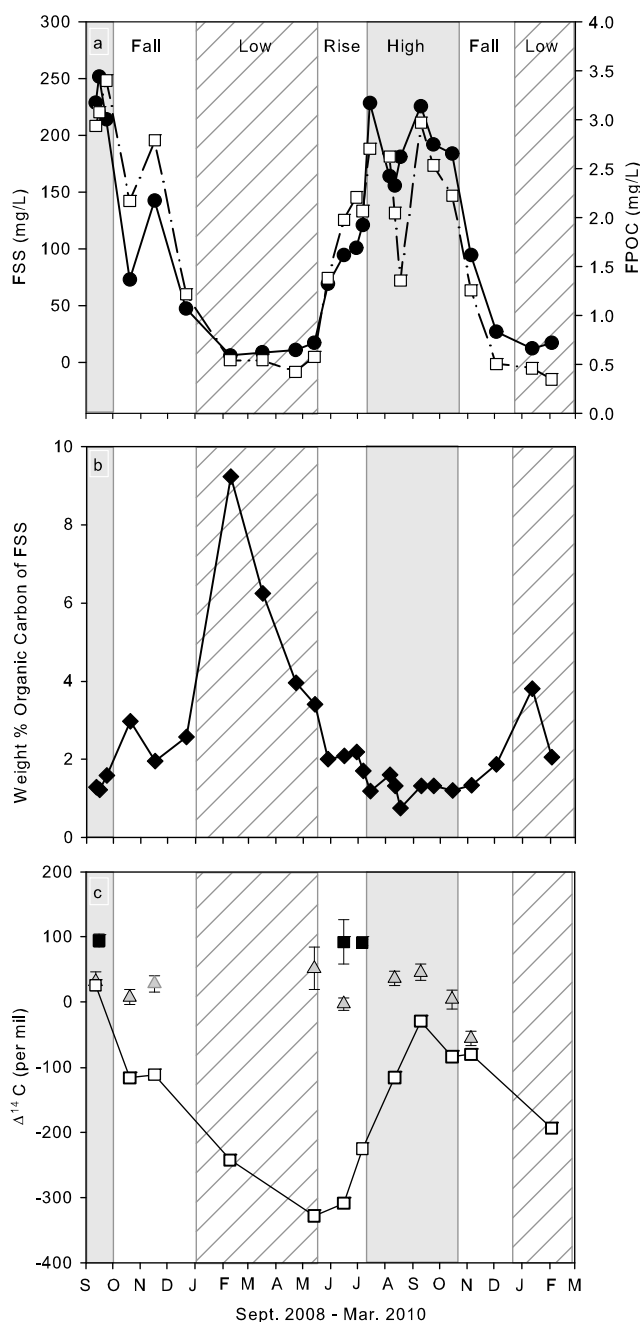
Figure 2. Seasonal variability in the concentration and composition of material transported by the Mekong River. (a) Variability in FSS (circles) and FPOC (squares) concentrations. Gray and diagonal shading indicates high and low-water stages, respectively. (b) Results for variability in the wt % OC content of FSS. (c) Seasonal changes in the average $\Delta^{14}C$ values of FPOC (white squares), DOC (gray triangles), and the concentration-weighted average value of particulate lignin (black squares). Error bars represent one σ_x and are not shown if the symbol is larger than the error bars.

[8] The $\Delta^{14}C$ values of lignin phenols transported during the rising water and high-water periods (Figure 3) enabled us to determine the contribution of C_{VP} to the $\Delta^{14}C$ values of FPOC. Abundance-weighted average values ranged between 92‰ and 95‰ (Figure 2c and Tables S3 and S4), which are more enriched than FPOC and DOC at all times of the year (Table S1 and S2). Atmospheric $\Delta^{14}C$ values were last within this range between 1992 and 1998 (postbomb ages were calibrated using CaliBomb [*Reimer et al.*, 2004; *Hua and Barbetti*, 2004]), suggesting that C_{VP} exported by Mekong FSS has a RT that ranges between 11 and 18 years within the catchment. Although similar atmospheric $\Delta^{14}C$ values were observed during the rapidly rising limb of the bomb curve, the consistency in the ^{14}C values of the individual phenols suggests that lignin is associated with the declining portion of the curve, where depleted ^{14}C values, up to the atmospheric value, mean either more recently fixed carbon or a heterogeneous mixture of different ages. No trends in $\Delta^{14}C$ values were observed across lignin phenol families or functional groups (Figure 3 and Table S3). The $\Delta^{14}C$ value of p-coumaric acid exported during high water was exceptionally low (3 ± 7‰) (below atmospheric values). p-coumaric acid is found in leafy debris, and its ^{14}C value indicates a more heterogeneous mixture of ages relative to the other phenols. Nevertheless, lignin remained high in ^{14}C and unvarying during the rising water and high-water periods (Figure 3), even though the $\Delta^{14}C$ value of FPOC simultaneously varied between -308‰ and 26‰ (Figure 2c). Although we lack estimates of the RT of lignin in tropical soils, the RT of lignin in the Mekong is similar to that of C in tropical surface soils [*Trumbore*, 1993] and C in plant biomass and soils of tropical forests [*Malhi and Grace*, 2000]. Previous work using plant wax lipids in the tropics estimated a RT of 2570 ^{14}C years for the majority of lipids, although there was a small fraction with a decadal RT [*Drenzek*, 2007]. Our results corroborate recent work, suggesting that lignin is a tracer for a significant fraction of terrestrial OC that differs from that which plant waxes trace [*Feng et al.*, 2013].

[9] Factors in addition to the ^{14}C age of C_{VP} control $\Delta^{14}C$ values of bulk FPOC during rising water and high-water periods. Elevated wt % OC values of FSS (Figure 2b) suggest an influence of phytoplankton during rising water [*Ellis et al.*, 2012]. The $\Delta^{14}C$ value of phytoplankton can be estimated from that of dissolved inorganic carbon (DIC), which ranged between -106‰ and -63‰ at this site during 2004 (data not shown). Considering that the $\Delta^{14}C$ values of FPOC are more depleted than this during both the low and rising water seasons, an additional aged C source is indicated. The Upper Basin (above the Chinese border) could be a significant source of aged C exported by the Mekong during the low/early rising water period because a greater proportion of the Mekong's total discharge emanates from this region in the form of snowmelt from the mountains and the Tibetan Plateau [*Kummu and Varis*, 2007]. Petrogenic C has been found within the Himalayas, with rivers from the Tibetan Plateau carrying aged soil OC [*Galy and Eglinton*, 2011; *Galy et al.*, 2008]. Tributaries from the Lower Basin exert more influence on the Mekong's discharge during the high-water period [*Kummu and Varis*, 2007]. The Lower Basin lies entirely within tropical latitudes, where high precipitation and solar radiation leads to high NPP. The young ^{14}C age of high-water FPOC suggests that young soils from the Lower Basin are an important source of C during monsoon

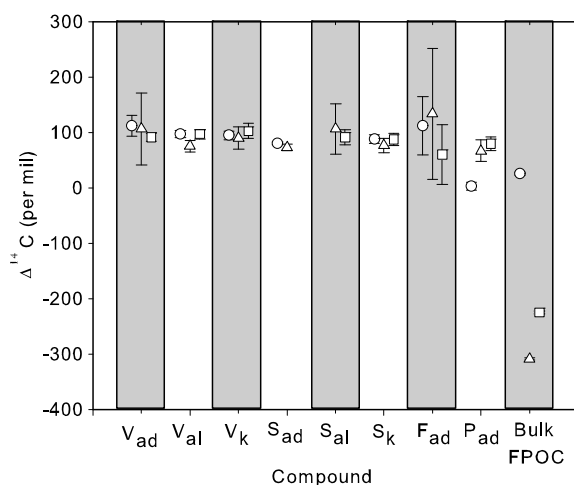consistently higher than FPOC, and varied significantly less (between -56‰ and 52‰), with no detectable seasonal trends (Figure 2c and Table S2). The enriched DOC ^{14}C signatures are likely due to the leaching of young soil organic matter and the short timescales required for DOC export relative to FPOC [*Raymond and Bauer*, 2001; *Mayorga et al.*, 2005].

Table 1. Variability in Particulate and Dissolved OC Composition and Concentration[a]

Stage	Δ^{14}C FPOC	δ^{13}C FPOC	% OC of FSS	[FPOC]	Δ^{14}C DOC	δ^{13}C DOC	[DOC]
Rising	-267 ± 42 (2)	-26.7 ± 0.3 (4)	2.0 ± 0.1	1.9 ± 0.2	-3 (1)	-27.2	3.7 ± 0.2 (4)
High	-51 ± 31 (4)	-26.5 ± 0.2 (10)	1.3 ± 0.1	2.6 ± 0.2	30 ± 9 (4)	-26.7 ± 0.1	3.8 ± 0.6 (9)
Falling	-103 ± 11 (3)	-26.8 ± 0.4 (5)	2.1 ± 0.3	1.6 ± 0.4	-7 ± 25 (3)	-26.2 ± 0.4	3.8 ± 0.7 (3)
Low	-254 ± 39 (3)	-27.2 ± 0.6 (6)	4.8 ± 1.0	0.5 ± 0.0	52 (1)	-	2.5 ± 0.4 (6)

[a]Data are reported in mean $\pm$ s.e. (number of samples). The number of samples for % OC and [FPOC] is the same as for δ^{13}C of FPOC, and the number of samples for δ^{13}C of DOC equals that of Δ^{14}C of DOC. Δ^{14}C and δ^{13}C are in per mil (‰), and units of concentration are mg/L.

rains. The lack of variability in $\Delta^{14}C_{VP}$ values between the rising water and high-water seasons suggests either that the geographical source of C_{VP} is the same between seasons or that the age of exported C_{VP}, regardless of where it is mobilized from, is consistently young. Given that previous work suggests that the lignin composition of Mekong FPOC (Table S5) cycles between vegetation common to the Upper and Lower Basin between the low and high-water periods, respectively [*Ellis et al.*, 2012], it appears that the latter explanation is more likely.

[10] To assess changes in the partitioning of C amongst its sources, we constructed a mixing model and applied it to samples for periods when the ^{14}C content of lignin was determined (supporting information). For the high-water period, we assumed that the ^{14}C value of FPOC was equal to plants ($\Delta^{14}C = 93‰$) and other soil-derived OM (Δ^{14}C is variable). During the rising water period, we added a third component, phytoplankton, and used the Δ^{14}C of DIC as a proxy for its Δ^{14}C value. As the influence of phytoplankton decreased into the flood period, the contribution of vascular plants increased: C_{VP} represented 18–38% of the FPOC during rising water and 38–82% during the high-water stage (Table S6). Further, the range of Δ^{14}C values of the residual pool exported during rising water was lower (older) than that during high water, consistent with the Upper Basin exerting a stronger influence on sediment composition during this time.

4. Conclusions

[11] We provide evidence that tropical rivers rapidly export primary production derivatives from catchments, within 18 years. Further, the ^{14}C age of lignin phenols remains constant during different periods of the rainy season, among bulk ^{14}C values that range from modern to several thousand years old. The young age of lignin suggests that the pre-aged carbon pools being concurrently transported in rivers are also significant. Further, the general agreement between the ^{14}C age of lignin phenols with the RT of C in forest ecosystems [*Trumbore*, 1993; *Malhi and Grace*, 2000] suggests that the Δ^{14}C value of riverine lignin is valuable in tracking changes in the size and age of a significant fraction of terrestrial carbon in tropical catchments.

[12] Tropical biomes will continue to experience elevated temperatures before the end of the century [*Diffenbaugh and Scherer*, 2011], with impacts on the cycling of C_{VP} [*Davidson and Janssens*, 2006]. A variety of approaches are needed to assess impacts on terrestrial C storage. The riverine approach is likely to be highly informative, but it is hindered by a lack of radiocarbon data on lignin phenols and a lack of understanding of how these signatures respond to interannual variation. This work represents a proof of concept from which we can use rivers to assess how climate change will impact terrestrial C cycling.

Figure 3. The Δ^{14}C values of individual lignin monomers. Circles, triangles, and squares denote September 10–17, 2008 (high), June 15–19, 2009 (rising), and July 2–6, 2009 (rising), respectively. Abbreviations are as follows: V_{ad} (vanillic acid), V_{al} (vanillin), V_k (acetovanillone), S_{ad} (syringic acid), S_{al} (syringaldehyde), S_k (acetosyringone), F_{ad} (ferulic acid), and P_{ad} (p-coumaric acid).

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