

# High diurnal variation in dissolved inorganic C, $\delta^{13}\text{C}$ values and surface efflux of $\text{CO}_2$ in a seasonal tropical floodplain

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Received: 5 September 2012 / Accepted: 6 May 2013 / Published online: 12 May 2013  
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**Abstract** Diurnal variations in aquatic systems may be a major factor influencing carbon cycling. However, few studies have examined diurnal variation on floodplains and wetlands, especially in the tropics. Stable isotope analysis of dissolved inorganic carbon ( $\delta^{13}\text{C}_{\text{DIC}}$ ) provides insight into the driving factors behind diurnal physio-chemical variability, but to date, the manual collection of large sample numbers at high temporal frequency has been prohibitive. Here, we report one of the first, high-resolution isotopic studies of  $\delta^{13}\text{C}_{\text{DIC}}$  on a tropical floodplain using acidification-interface cavity ring-down spectrometry. Water samples were analysed for  $\delta^{13}\text{C}_{\text{DIC}}$  and other water quality parameters at 15-min intervals for 24 h. Our results show significant diurnal variation in both DIC concentration and  $\delta^{13}\text{C}_{\text{DIC}}$ . Maximum DIC concentration, recorded overnight, was approximately 100 % greater than during the day. Maximum DIC concentration coincided with minimum  $\delta^{13}\text{C}_{\text{DIC}}$  as a result of shifting autotrophic/heterotrophic balance. Changes were significant over small time scales and showed  $\text{CO}_2$  gas evasion estimates could vary by as much as 50 % based on measurements taken less than 5 h apart. These data show that to accurately evaluate the role of tropical floodplains in global carbon dynamics, a comprehensive understanding of diurnal variation will be essential.

**Keywords** Diurnal hydrochemistry · Floodplain · Tropical · Dissolved carbon · Carbon dioxide evasion · Stable isotope

## Introduction

Wetlands comprise a relatively minor feature of the earth's surface, covering approximately 4–6 % of terrestrial environments (Mitsch and Gosselink 2000; Kuehn et al. 2004). Despite this, they have been shown to be significant conduits of carbon processing, storage and release (Zhou et al. 2009). Estimates of wetland  $\text{CO}_2$  carbon flux vary significantly depending on location and vegetation type, and current regional estimates are also confounded by the majority of studies being conducted in temperate and boreal zones (Kuehn et al. 2004; Miller 2011). In general, temperate and boreal wetlands act as net carbon sinks, with the accumulation of produced organic matter exceeding evasion fluxes of  $\text{CO}_2$  and methane. However, as understanding of tropical wetland processes develops, they have been revealed as significant sources of both  $\text{CO}_2$  and methane to the atmosphere (Richey et al. 2002; Melack et al. 2004; Belger et al. 2011).

Dissolved inorganic carbon (DIC) and dissolved organic carbon (DOC) are the dominant fractions of the aquatic carbon pool. DIC can account for significant lateral flux as well as drive processes leading to  $\text{CO}_2$  ingress from (e.g. Miller 2011) or egression to the atmosphere (e.g. Mayorga et al. 2005). DOC is the most significant pool of reduced carbon in aquatic systems (Volk et al. 1997), being responsible for significant lateral fluxes (Cole et al. 2007; Bass et al. 2011) and is a significant source of energy to aquatic food webs (Maurice and Leff 2002).

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Processes controlling carbon dynamics in aquatic systems operate over varying spatial and temporal scales. Until recently high temporal resolution studies examining rapidly changing processes have been rare, likely due to the expense and inconvenience associated with collecting such data. However, the data that does exist reveal rapidly changing conditions within aquatic carbon pools. For example, stream DIC concentration has been observed to rise overnight and decrease during the day due to respiratory and photosynthetic pathways, respectively (Waldron et al. 2007; Poulson and Sullivan 2010). The magnitude of this change can be anywhere up to 400 % (Parker et al. 2010).

In order to accurately model processes that drive carbon dynamics at small scales, high temporal resolution data is necessary. Progress to date has mainly focussed on the development of continuous  $p\text{CO}_2$  sensors (Dinsmore et al. 2009; Johnson et al. 2010; Dyson et al. 2010). Even more recently, development of instruments capable of measuring total DIC concentration has enabled further refinement to our understanding of the carbon cycle in rapidly changing aquatic systems (Bass et al. 2012).

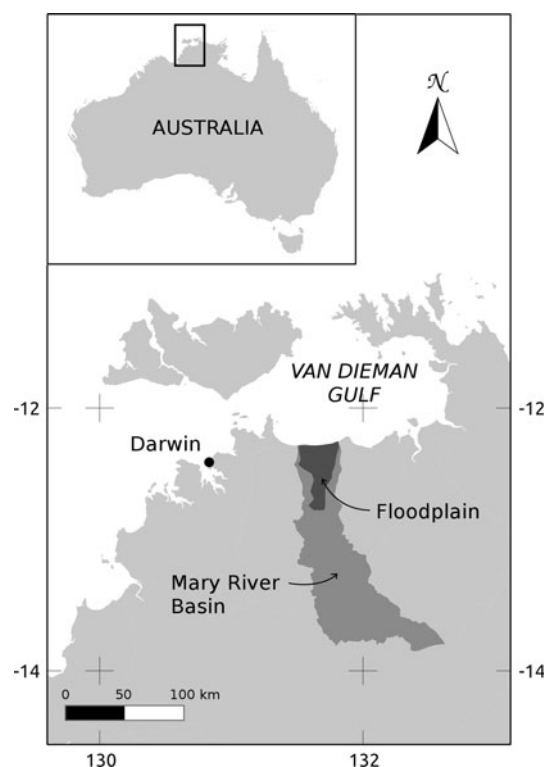
Along with concentration of various carbon species, the stable isotope composition of DIC and DOC can provide further insight into controlling biogeochemical processes. However, the collection of stable isotope data has been logistically challenging to date, especially if capture of rapidly changing processes is the goal. The notable attempts that have been made, using samples collected 4–6 h apart have revealed dynamic controls on  $\delta^{13}\text{C}_{\text{DIC}}$  driven by both physical and biological controls (Ziegler and Fogel 2003; Waldron et al. 2007). Past work has focused primarily on the temperate/boreal zones; we know of no study that has measured carbon isotopic and concentration data at sub-hourly intervals in tropical wetlands.

Here, we present diurnal data from a tropical floodplain detailing changes in carbon dynamics at sub-hourly resolution. This data provide an initial baseline on which we can further our understanding of biogeochemical processes in tropical environments which are under-represented in the scientific literature. Specifically, using state of the art instrumentation, we demonstrate the rapidity and magnitude of changes in tropical floodplain aquatic carbon dynamics.

## Experimental

### Study site

The Mary River basin is situated in the wet–dry tropics of Northern Australia ( $12^\circ 30'\text{S}$ ,  $131^\circ 30'\text{E}$ ). The Mary River and its tributaries drain a catchment of approximately



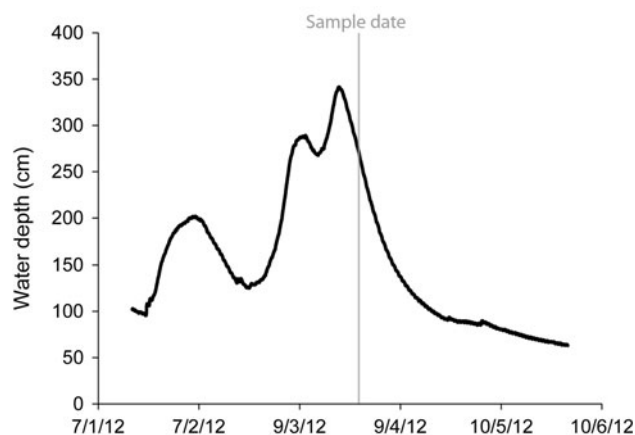
**Fig. 1** Location of the Mary River floodplain in Northern Australia approximately 95 km east of Darwin. Magnified area shows the catchment and floodplain extent

8,090 km<sup>2</sup> (Fig. 1). The local climate is monsoonal, with distinct wet and dry seasons. The wet season, in which the majority of the 915 mm annual average rain falls, starts in November–December and lasts for 3–4 months, though this is variable year to year (Finlayson et al. 1990). The river system then distributes water over a significant clay-based floodplain system (approximately 1,080 km<sup>2</sup>). During flood seasons, relatively shallow silt and clay-based soils have built up through deposition processes. These are underlain by gleyed marine sediments (Wasson 1992).

Vegetation communities are related to water depth during the wet season (Finlayson 2005; Petit et al. 2011). Aquatic grasslands dominate the floodplain though forested, and woodland areas (dominated by *Melaleuca* spp) are significant. The site studied in this experiment was on the floodplain edge, dominated by aquatic grasses.

### Data collection

In March 2012, we monitored environmental parameters for approximately 24 h at the floodplain edge ( $12^\circ 39.462'\text{S}$ ,  $131^\circ 42.661'\text{E}$ ) on the receding limb of a flood peak (Fig. 2). Surface water was continuously pumped approximately 10 metres from the floodplain edge into a polyethylene flume at a rate of approximately 6 L min<sup>-1</sup>. The flume was instrumented with a Hydrolab SONDE water quality probe to



**Fig. 2** Average water depth of the Mary River floodplain. Data recorded continuously at hourly intervals at seven locations. The presented diurnal experiment was conducted on the falling limb of the highest recorded peak in the 2012 wet season

record water temperature ( $\pm 0.1$  °C), pH ( $\pm 0.1$ ), dissolved oxygen (DO) concentration ( $\pm 0.2$  mg L<sup>-1</sup>) and electrical conductivity ( $\pm 0.5$  %). DOC concentration was measured using a S::can Spectrolyser (e.g. Waterloo et al. 2006; Bass et al. 2011). By repeated calibration using known DOC solutions, error in the DOC measurement was estimated at less than 0.15 mg L<sup>-1</sup>. Both the SONDE and Spectrolyser recorded data at 15-min intervals.

DIC concentration and  $\delta^{13}\text{C}_{\text{DIC}}$  were measured using the Isotopic Continuous Dissolved Inorganic Carbon Analyser (ISO-CADICA) (Bass et al. 2012). Briefly, the ISO-CADICA utilises an automated acidification interface to sequentially convert all DIC in a sample to free CO<sub>2</sub>. Acidified water is held in gas-permeable Teflon tubing allowing CO<sub>2</sub> from each acidified aliquot to fill an extraction chamber. The extraction chamber is linked to a Wavelength Scanning Cavity Ring-down Spectrometer (WS-CRDS), capable of measuring  $\delta^{13}\text{C}$  at accuracies comparable to isotope ratio mass spectrometry (IRMS). Each acidification and analysis takes 15 min, after which time the extraction chamber is refreshed with CO<sub>2</sub>-free air and the cycle repeats. The ISO-CADICA records DIC concentration to  $\pm 0.12$  mg L<sup>-1</sup> and  $\delta^{13}\text{C}_{\text{DIC}}$  to  $\pm 0.2$  ‰. Scaling of the  $\delta^{13}\text{C}_{\text{DIC}}$  values to the PDB scale was carried out during the study by concurrent collection and analysis of manual samples using a headspace equilibration technique and IRMS (e.g. Waldron et al. 2007; Bass et al. 2010). Specifically, calibration samples were analysed using a Gas-Bench III headspace analyser coupled to a Delta V Plus mass spectrometer.

Chlorophyll *a* was measured using an AquaFluor™ handheld fluorometer. The *in vivo* fluorescence data were correlated with extracted chlorophyll *a* data from samples measured spectrophotometrically at the Cairns Regional Council laboratory.

## CO<sub>2</sub> flux calculations

Using measured pH and DIC concentration values,  $p\text{CO}_2$  was calculated using CO2SYS software. The flux of CO<sub>2</sub> between the water and atmosphere was then calculated using a one-dimensional stagnant film model (e.g. Raymond et al. 2000) where

$$\text{CO}_2 \text{ flux} = k \times (p\text{CO}_{2\text{w}} - p\text{CO}_{2\text{a}}) \quad (1)$$

where  $p\text{CO}_{2\text{w}}$  is the partial pressure of CO<sub>2</sub> in the water,  $p\text{CO}_{2\text{a}}$  is the partial pressure of CO<sub>2</sub> in the atmosphere and  $k$  is the gas transfer velocity (m day<sup>-1</sup>). During other work on the same system, CO<sub>2</sub> emission rates were measured using a floating chamber methodology based on that described in Rosenqvist et al. (2002). CO<sub>2</sub> build-up in the chamber was measured over a 15 min period using an INNOVA photoacoustic gas analyser. Using these measured fluxes, and re-arranging Eq. 1,  $k$  was calculated for each sample time. The average  $k$  value for the four representative sites ( $1.06 \pm 0.71$ ) was used to estimate the minimum, maximum and average flux rates over the study period.

## Results and discussion

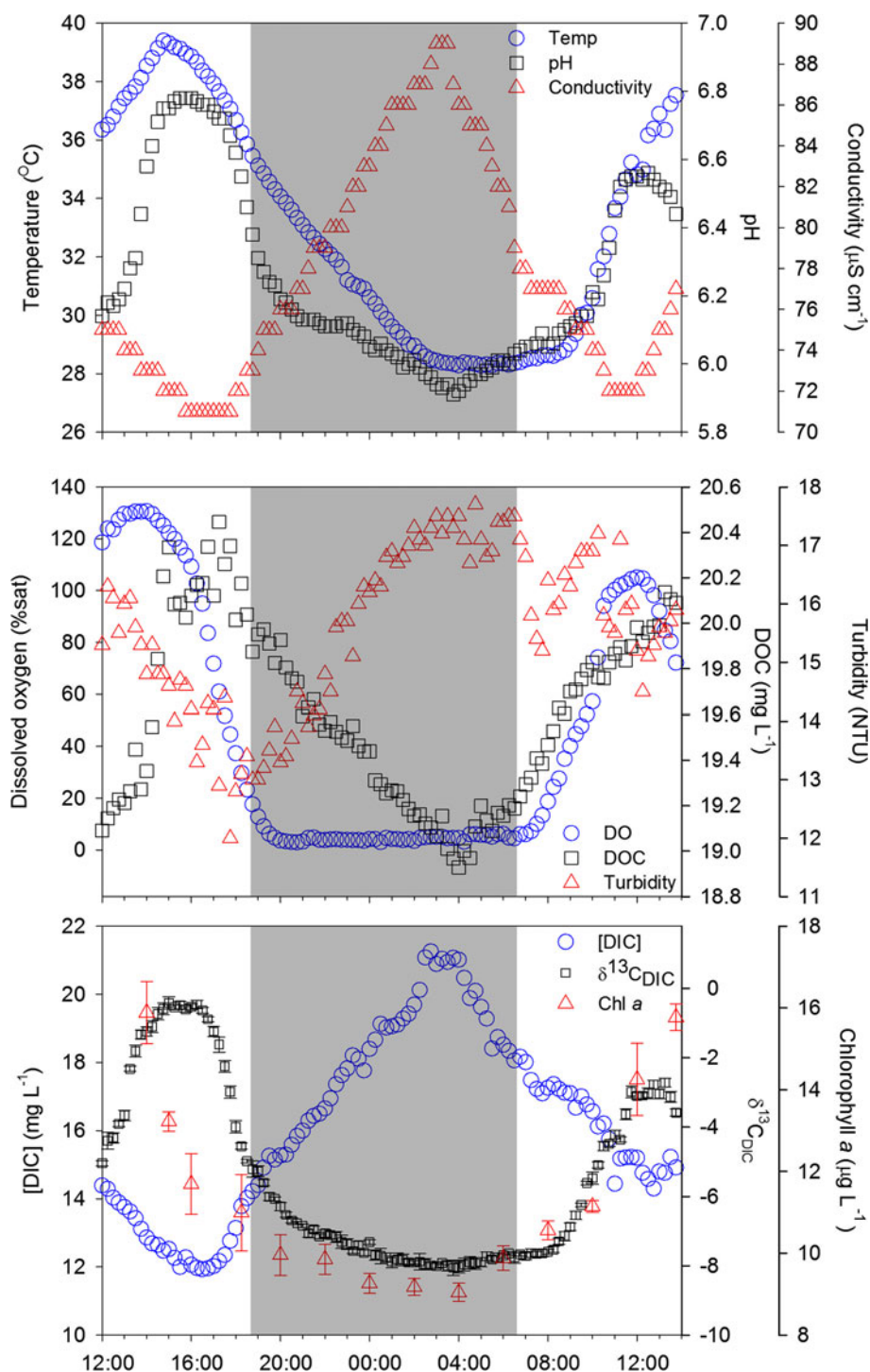
### Diurnal patterns of change in physio-chemical parameters

The data presented in this study are likely subject to two different cycles, the first associated with a receding flood limb and the second from diurnal variations. Sequential peaks and troughs for certain parameters seem to be either increasing (e.g. conductivity) or decreasing (e.g. Temp, pH, DO) (Fig. 3) suggesting longer term change related to flood recession. However, the magnitude of change is significantly smaller than the measured diurnal cycles, and as such, diurnal cycles will be the focus of this discussion.

During the 24 h period measured, water depth decreased from 35 to 9 cm at a rate of approximately 1.1 cm h<sup>-1</sup>. The sampling period corresponded to the falling limb of the largest flood peak recorded during the wet season of 2011–2012 (Fig. 2). Our sampling site was located on the extreme edge of the floodplain, and as such, the location is only intermittently inundated during the wet season. Consequently, this site may be subject to more rapidly changing wet–dry cycles than the more permanently flooded sites.

Significant diurnal variation was observed in all measured parameters. Water temperature decreased from a daytime maximum of 39.4 °C at 1445 hours to a minimum of 28.3 °C at 0500 hours, induced by the change in solar radiation. Water temperature can potentially cause variation

**Fig. 3** Diurnal variation in physio-chemical characteristics over a 24-h period. *Error bars* for  $\delta^{13}\text{C}_{\text{DIC}}$  represent the standard deviation of approximately 30 measurements. *Error bars* for Chlorophyll *a* are the standard deviation of three replicated measurements. *Shaded regions* show the approximate hours of darkness. The opposite responses of dissolved inorganic carbon (DIC) and dissolved oxygen (DO) concentration to the diurnal cycle point to a primarily biological control



in dissolved gas concentrations by affecting the solubility coefficients. While temperature has been observed to cause diurnal variation in both DIC and DO in some streams (Gammons et al. 2005), generally the effect is minor when compared to biologically mediated change (Nimick et al. 2011). Montety et al. (2011), for example, found that temperature could account for only 1 % of the observed changes in dissolved gas concentration. If temperature was driving

dissolved gas concentration, we would expect similar diurnal cycles for both DIC and DO as they both become less soluble at higher temperatures. The fact we observed DIC and DO respond in opposite directions to changing temperature suggests temperature is not the primary driver of concentration, even though the measured temperature change of 11 °C is significantly greater than that reported in diurnal stream studies (Montety et al. 2011; Nimick et al.



2011; Tobias and Böhlke 2011) we calculate it could account for less than 20 % of the measured change in dissolved CO<sub>2</sub> concentration.

Conductivity reached a minimum and pH reached a maximum just before nightfall. Conductivity gradually increased overnight showing a distinct peak before day-break, while pH rapidly declined reaching a relatively stable minimum which lasted several hours. An initial drop of 0.6 pH units in 3 h was followed by a decline of only 0.2 pH units in the following 10 h. The most common driving factor of diurnal pH change is the change in dissolved CO<sub>2</sub> concentration, though various other processes such as temperature, trace element adsorption and desorption, mineral precipitation, chemical speciation, nitrification and microbial growth can all have an effect (Nimick et al. 2011).

The concentration of DIC can be affected by within-system biological processes, particularly the photosynthetic/respiratory balance, and by the dissolution/precipitation of carbonate minerals, as well as the previously mentioned temperature effect. Carbonate mineral input is minimal at our site (background Mg and Ca concentration generally less than 1.0 mg L<sup>-1</sup> (Supervising scientist 2010)), and thus, biological factors are considered the most significant driver. The significant inverse linear correlation between DIC and DO ( $R^2 = 0.57$ ,  $p < 0.005$ ,  $n = 104$ ) suggests biological rather than physical controls. DIC concentration increased by approximately 100 %, from a minimum at 1600 hours to a maximum at 0330 hours. Conversely, the concentration of DO declined overnight, remaining stable, just above anoxia for much of the night. During the day, photosynthesis utilises DIC and produces DO, resulting in the drop and rise of each, respectively, with the opposite occurring overnight as respiratory processes become dominant. The very low DO concentrations are likely a function of enhanced respiratory processes associated with the submerged vegetation.

We measured a significant increase in  $\delta^{13}\text{C}_{\text{DIC}}$  during the day, peaking at approximately -1 ‰ and then declining overnight to a relatively stable -8 ‰. The apparently high baseline value of -8 ‰ may reflect the dominance of C4 grasses in the catchment, which are characterised by higher  $\delta^{13}\text{C}$  values, or significant input of DIC that has been fractionated during photosynthesis by aquatic plants in the rest of the catchment. The diurnal magnitude of change in  $\delta^{13}\text{C}_{\text{DIC}}$  (~ 7 ‰) recorded here significantly exceeds that reported for other aquatic systems such as a karst river (~ 1.3 ‰, Montety et al. 2011), coral reef lagoon (~ 2.4 ‰, Bass et al. 2012) and temperate freshwater marsh (~ 5 ‰, Ziegler and Fogel 2003). The temporal patterns in  $\delta^{13}\text{C}_{\text{DIC}}$  alone cannot delineate between biological and physical control in this case. However, coupled to the significant inverse relationship between DIC and DO concentration, it is probable that

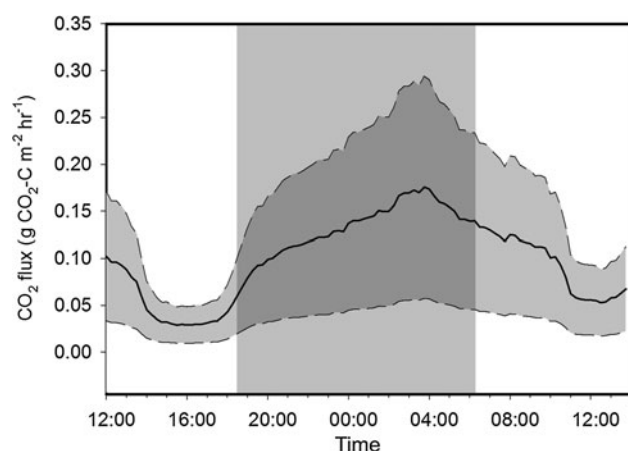
biological control is significant. Subaquatic vegetation and photosynthetic plankton are known to discriminate against  $^{13}\text{C}_{\text{DIC}}$  during photosynthesis (Smith and Walker 1980; Keeley 1990), leading to a build-up of  $^{13}\text{C}$  in the remaining DIC pool, with the opposite being true when respiratory processes dominate. While we suspect biological processing to be a significant driver of  $\delta^{13}\text{C}_{\text{DIC}}$  change, a combination of biological and physical factors (e.g. elevated CO<sub>2</sub> evasion at high temperatures) may account for the high range in  $\delta^{13}\text{C}_{\text{DIC}}$  values observed in this study.

If biological processing dominates chemical processes in an aquatic system, it should be reflected in the DOC pool. During photosynthetic processes, DOC will be produced causing a rise in the water column, with the opposite occurring overnight as respiratory organisms use the DOC as a substrate. While the change in DOC recorded in this study was relatively small compared to DIC (1.4 compared to ~ 10 mg L<sup>-1</sup>), there was a significant rise during the day followed by a decline overnight and was comparable to other studies (Ziegler and Fogel 2003). Diurnal variation in DOC has been observed in some other systems (Kaplan and Bott 1982; Parker et al. 2010), but not in others (Bourg and Bertin 1996; Spencer et al. 2007; Beck et al. 2009), but when diurnal was observed, they have generally been characterised by an increasing concentration during the day, decreasing overnight, as measured in this study. The concentration of Chlorophyll *a* followed a similar pattern to DOC, likely related to the consumption and breakdown of chlorophyll containing material overnight and the day-time production of plant material and further supporting the conclusion of a primarily biological control.

#### CO<sub>2</sub> evasion from surface water

CO<sub>2</sub> was found to be supersaturated in surficial waters over the entire 24 h sampling period ( $p\text{CO}_2 = 5,436\text{--}33,041 \mu\text{atm}$ ), consistent with measurements from other tropical floodplains (Belger et al. 2011). Peak emissions of CO<sub>2</sub> corresponded to periods where anoxia was recorded (Fig. 4), as also observed in central Amazon floodplains (Bartlett et al. 1990; Devol et al. 1994). This pattern is a result of the varying metabolic balance during a diurnal cycle where photosynthetic processes produce oxygen and consume CO<sub>2</sub> during the day, while aerobic respiration leads to production of CO<sub>2</sub> and a concomitant consumption of oxygen overnight.

Wetlands have the potential to act as both carbon sinks and sources. The estimates we are aware of point to tropical floodplains as being net sources of atmospheric CO<sub>2</sub> (Richey et al. 2002; Melack et al. 2004; Belger et al. 2011). However, this study shows the calculated magnitude of any flux estimate may be highly influenced by the temporal sampling regime.



**Fig. 4** Diurnal variation in CO<sub>2</sub> flux from the water surface to the atmosphere. The *shaded region* shows the approximate hours of darkness. The *solid black line* shows efflux based on the average estimate of gas transfer velocity (*k*). The *shaded region* shows the maximum and minimum estimated efflux based on maximum and minimum *k* values. Efflux of CO<sub>2</sub> was approximately six times greater during the hours of darkness than during the day

CO<sub>2</sub> flux from the Mary River floodplain followed a clear diurnal cycle, increasing by a factor of six from a minimum of 0.03 g CO<sub>2</sub>-C m<sup>-2</sup> h<sup>-1</sup> at 1600 hours to a maximum of 0.18 g CO<sub>2</sub>-C m<sup>-2</sup> h<sup>-1</sup> at 0345 hours. The time frame required to observe significant changes in flux is small. The average change in flux over time was 5.7 ± 4.2 % h<sup>-1</sup>, meaning samples taken only 5 h apart could potentially yield flux estimates that diverge by close to 50 %. Extrapolated further, if we estimated floodplain CO<sub>2</sub> flux based on a measurement at 1000 hours, assuming a floodplain of 1,080 km<sup>-2</sup>, we would calculate a flux of 156.0 tonnes C h<sup>-1</sup>, compared to only 86.2 tonnes C h<sup>-1</sup> if measured at 1200 hours. This level of variability has potentially significant implications to up-scaling regional carbon flux estimates from tropical floodplain systems.

## Conclusion

High-resolution data have elucidated the existence of significant and rapid changes to the physio-chemical characteristics of water on a tropical floodplain margin. Data recorded on the Mary River floodplain have revealed that the magnitude of diurnal variability on tropical floodplains can significantly exceed that previously reported in other aquatic environments. We find that the majority of previous studies of aquatic carbon dynamics focus on rivers and streams, generally in northern hemisphere temperate/boreal zones, highlighting a significant paucity of data from tropical floodplain environments. This work adds to the growing body of evidence that tropical wetlands assume significance to global carbon budgets disproportionate to

their area. It also shows that failing to consider the magnitude of diurnal variation when up-scaling to regional estimates may yield significant inaccuracies.

**Acknowledgments** The staff of the Wildman Ranger station is thanked for their assistance and willingness to provide logistical advice. We thank owners and staff of the Wildman Wilderness lodge for providing access to the study site. Comments from two anonymous reviewers substantially improved this manuscript.

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